



Remedial Work Plan

42 York Street

Location:

42 York Street
Rochester, New York 14614
NYSDEC Spill No. 2206496

Prepared on Behalf of:

City of Rochester
30 Church Street
Rochester, New York 14614

Prepared By:

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CERTIFICATION

"I, Ann Barber, certify that I am currently a NYS registered professional engineer and that this Remedial Work Plan was prepared in accordance with all applicable statutes and regulations and in substantial conformance with the DER Technical Guidance for Site Investigation and Remediation (DER-10)."



NYS Prof. Engineer 100521

August 19, 2024
Date

Ann Barber

Signature



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COMMON ACRONYMS / ABBREVIATIONS

<i>ASP</i>	– Analytical Services Protocol
<i>bgs</i>	– below ground surface
<i>BOA</i>	– Brownfield Opportunity Area
<i>CAMP</i>	– Community Air Monitoring Plan
<i>CAP</i>	– Corrective Action Plan
<i>COC</i>	– Constituents of Concern
<i>DEC</i>	– (New York State) Department of Environmental Conservation
<i>DER</i>	– Department of Environmental Remediation
<i>DER-10</i>	– DER Technical Guidance for Site Investigation and Remediation
<i>ELAP</i>	– Environmental Laboratory Approval Program
<i>HASP</i>	– Health and Safety Plan
<i>IRM</i>	– Interim Remedial Measure
<i>NYSDEC</i>	– New York State Department of Environmental Conservation
<i>NYSDOH</i>	– New York State Department of Health
<i>PAH</i>	– Polycyclic Aromatic Hydrocarbon
<i>ppb</i>	– parts per billion (equal to micrograms per liter - ug/L)
<i>PPE</i>	– Personal Protective Equipment
<i>QAPP</i>	– Quality Assurance Project Plan
<i>QC</i>	– Quality Control
<i>(RP)SCO</i>	– (Remedial Program) Soil Cleanup Objective
<i>SCGs</i>	– Standards, Criteria, and Guidelines
<i>SF</i>	– Square feet
<i>SVOC</i>	– Semi-volatile Organic Compound
<i>USEPA</i>	– United States Environmental Protection Agency
<i>VOC</i>	– Volatile Organic Compound

1.0 INTRODUCTION

LaBella Associates, D.P.C. (“LaBella”) was retained by the City of Rochester to prepare a Remedial Work Plan (RWP) for the property located at 42 York Street, in the City of Rochester, Monroe County, New York, hereinafter referred to as the “Site” (see Figures 1 and 2). This RWP is part of the USEPA Brownfields Multipurpose Grant Program awarded to the City of Rochester in 2021.

The scope and conditions of this RWP were in accordance with Task 5C of LaBella’s Proposal dated August 3, 2022. A list of key project contacts has been included as Appendix 1.

The Site is included in a Stipulation Agreement for NYSDEC Spill No. 2206496 (dated December 30, 2022) between the City of Rochester (City) and the New York State Department of Environmental Conservation (NYSDEC). With respect to the Stipulation Agreement, this RWP should also be considered the Final Corrective Action Plan (CAP). Per the Stipulation Agreement made with the NYSDEC, the cleanup and removal of discharges of petroleum-related compounds at the Site is required to be initiated no later than October 1, 2024 (with the Construction Completion Report prepared by April 30, 2025).

1.1 Project Objective/Background

The Site is located within the Bull’s Head BOA and associated with the City of Rochester’s Bull’s Head Revitalization Project. Remedial efforts targeting the removal of fill material and the associated SVOCs and metals impacts are necessary to provide a clean site that promotes redevelopment, in accordance with the Bull’s Head Revitalization Plan.

As part of the Comprehensive Professional Environmental Investigation and Remediation services for the property located at 42 York Street, LaBella was retained by the City of Rochester to develop a Remedial Work Plan (RWP) to meet NYCRR Part 375 Restricted-Residential Use SCOs for the future mixed commercial and residential use of the property.

2.0 SITE BACKGROUND

2.1 Site Description and Surrounding Properties

The Site is comprised of one approximate 0.48-acre parcel (SBL #120.42-2-72.001) located at 42 York Street, in the City of Rochester, Monroe County, New York. Refer to Figure 1 for the approximate Site location (map) and Figure 2 for a local site plan. The Site is within the Bull’s Head redevelopment area and is currently an unused paved parking lot. The Site is located in an urban setting.

Surrounding Properties

The Site is presently bordered by the following properties:

Direction	Address	Owner	Current Land Use
North	50 York Street	City of Rochester	Vacant Lot / Undeveloped
East	866 West Main Street	City of Rochester	Vacant Lot / Gravel Parking
South	(Beyond Ruby Place ROW) (890-920 West Main Street)	City of Rochester	Vacant Lot / Undeveloped
West	Multiple (24-32 York Street)	City of Rochester	Vacant Lot / Undeveloped
	(Beyond York St ROW) Multiple (21-55 York Street)	City of Rochester and Other Private Individuals	Single Family Residential

2.2 Site History

The Site appeared to be first developed with several residential dwellings and sheds/barns on portions of the parcel from 1892 to at least 1935.

On aerial photographs dated 1988, 1993, and 2003, approximately 15 vehicles are parked on the Site. In addition, apparent dark staining and miscellaneous items (which may be indicative of debris) appear to be located throughout the Site. The staining and debris on the Site may also be indicative of current or former industrial/manufacturing use of the property or effects from surrounding properties. Potential concerns associated with an industrial/manufacturing use of a property include the contamination of soil and/or groundwater if leaks/spills and/or improper handling/disposal of hazardous materials, petroleum products, and/or hazardous wastes has occurred.

3.0 PREVIOUS ENVIRONMENTAL INVESTIGATIONS & REPORTS

The following historical environmental reports exist for the Site:

- *Environmental Screen, Bulls Head Project Area (103 Contiguous Parcels of Land), Rochester, New York*, prepared by Day Environmental, Inc. and dated September 2009.
- *Phase I Environmental Site Assessment*, prepared by Day Environmental, Inc. and dated August 16, 2016.
- *Pre-Development Phase II Environmental Site Assessment and Geotechnical Study Report*, prepared by Day Environmental, Inc. dated July 2019.
- *Phase II Environmental Site Assessment 42 York Street*, prepared by LaBella and dated October 9, 2023 (included as Appendix 2 of this RWP).

3.1 Environmental Screen, Bulls Head Project Area (103 Contiguous Parcels of Land), Rochester, New York – September 2009

Day Environmental Inc. completed an Environmental Screen titled “Bulls Head Project Area (103 Contiguous Parcels of Land) Rochester, New York” in September 2009. 42 York Street was classified as “Property #93” within the document and the following RECs pertaining to the Site, were stated in the Environmental Screen:

“In the 1988, 1993, and 2003 aerial photographs, approximately 15 vehicles are parked on this property. In addition, apparent dark staining, and miscellaneous items, which may be indicative of debris appear to be located throughout this property. The staining and debris on this property may be indicative of current or former industrial/manufacturing use of the property or effects from surrounding properties. Potential concerns associated with an industrial/manufacturing use of a property include the contamination of soil and/or groundwater if leaks/spills and/or improper handling/disposal of hazardous materials, petroleum products, and/or hazardous wastes has occurred.”

3.2 Phase I Environmental Site Assessment – August 2016

Day Environmental Inc. completed a Phase I Environmental Site Assessment dated August 16, 2016 for the properties addressed 894-898 West Main Street, and 42 York Street, Rochester, New York. Based on this assessment, Day Environmental called out the same RECs at 42 York Street identified within the Environmental Screen dated September 2009. Based on the identified RECs, Day Environmental recommended additional investigation to identify any potential environmental impacts at the Site.

3.3 Pre-Development Phase II Environmental Site Assessment and Geotechnical Study Report – July 2019

Day Environmental Inc. (“Day”) completed a Phase II Environmental Site Assessment and Geotechnical Study Report dated July 2019. In 2018, Day advanced two (2) test pits (TP-13 & TP-14), six (6) soil borings (TB-

04, TB-05, TB-06, TB-18, TB-24, and TB-27), and installed one (1) groundwater monitoring well (MW-01) on the 42 York Street Site.

Day identified fill material consisting of re-worked soil with lesser amounts of topsoil, ash, cinders, coal, asphalt, brick, concrete, organics, wood, metal, and/or plastics. Day identified approximately 20% to 90% larger debris in some locations containing concrete, brick, concrete block, rock, metal, and wood. Additionally, Day identified fill material containing 50% ash and/or cinders (>0.5 feet thick) in the center of the Site spanning from York Street to Kensington Street.

Below is a list of the following soil and groundwater samples collected from the Site in 2018:

- TP-13(7.0) – Metals exceeded CSCOs (arsenic, cadmium, chromium, and lead)
- TP-14(7.0) – VOCs exceeding UUSCOs (1,2,4-trimethylbenzene and m,p-xylene); SVOC exceeding RSCOs (2-methylnaphthalene)
- TP-14(3.5) – Metals exceeding RRSCOs (lead and mercury)
- TB-04(2.5) – Metal exceeded UUSCOs (lead)
- TP-13(1.0-2.0) and TP-14(3.5) sampled for TCLP analysis – did not contain TCLP metals exceeding TCLP regulatory levels for the toxicity characteristic.
- MW-01 - No VOCs were detected above laboratory MDLs from the groundwater sample collected on March 9, 2018. VOCs were detected at concentrations exceeding laboratory MDLs in groundwater samples collected on April 16, 2018; however, the concentrations of VOCs did not exceed their respective NYSDEC TOGS 1.1.1 guidance values.

3.4 Phase II Environmental Site Assessment 42 York Street – October 2023

LaBella completed a Phase II ESA report titled “Phase II Environmental Site Assessment 42 York Street”, dated October 9, 2023. The Phase II ESA included the advancement of ten (10) test pits at the Site, the installation of one (1) groundwater monitoring well, and the redevelopment of one (1) existing groundwater monitoring well. Six (6) soil samples for further investigation/delineation of contaminants were collected and analyzed for VOCs, SVOCs, and metals concentrations. Six (6) soil samples for waste characterization purposes were analyzed for TCLP VOCs, TCLP SVOCs, TCLP Metals, PCBs, Reactivity, Corrosivity, and Ignitability. Two (2) groundwater samples (one from the newly installed monitoring well and one from an existing monitoring well) were analyzed for VOCs, SVOCs, and metals concentrations. Activities were conducted in accordance with the USEPA-approved QAPP (including collection of QA/QC samples). The Phase II ESA concluded the following:

- LaBella performed a Pre-Characterization Study of the subsurface materials planned for off-site disposal during the remedial excavation. Six (6) soil samples for waste characterization purposes were submitted to Alpha Analytical, a NYSDOH ELAP-certified laboratory for analysis of the following:
 - Toxicity Characteristics Leachate Procedure (TCLP) VOCs using USEPA Method 8260/1311;
 - TCLP SVOCs using USEPA Method 8270/1311;
 - TCLP Metals using USEPA Method 6010/7471/1311;
 - Polychlorinated Biphenyls (PCBs) using USEPA Method 8082;
 - Reactivity using USEPA Method 7.3;
 - Ignitability using USEPA Method 1030;
 - pH using USEPA Method 9045
- The Pre-Characterization Study determined that the urban fill/soil to be removed during the remedial excavation is considered non-hazardous material.
- Test pits were backfilled to grade using the excavated material on a first-out, last-in basis. The material was bucket tamped. Any asphalt present at the surface was segregated from soils and placed back on top of each test pit following backfill.

- The finding of urban soil/fill containing ash and cinders is consistent with historic investigation of the Site and surrounding area performed in 2018 (by others).
- Lead was detected in exceedance of applicable SCG in one (1) soil/fill sample collected during this assessment, expanding the footprint of previously identified extents where soil/fill in exceedance of applicable SCG is present on the Site. Various other metals (including cadmium, copper, mercury, nickel, and zinc) were detected in exceedance of Unrestricted Use SCOs in one or more samples of the urban soil/fill collected from the Site during this investigation.
- Groundwater is present at an approximate depth of five to six (5-6) feet below existing ground surface at the Site. Groundwater is estimated to flow to the northwest across the Site, based on areal data collected during this investigation.
- Although not detected in exceedance of applicable SCG, TCE was detected in groundwater at one sample location on the Site.
- Apparent bedrock is generally present at an approximate depth of 7 to 9.5 feet below existing ground surface at the Site.
- Fill/soil exceeding the standards for hazardous waste via TCLP analysis (waste characterization sampling) has not been identified at the Site.

The complete October 2023 Phase II ESA has been included as Appendix 2 of this RWP.

4.0 SITE GEOLOGY AND HYDROLOGY

This section summarizes the Site geology and hydrology based on historical environmental investigations and reports:

Geology

The following geology description is based on the Phase II ESA by LaBella (LaBella Project No. 2230119, dated October 9, 2023). Most of the Site is covered by an approximately 0.25-ft thick layer of broken asphalt. An approximately 0.5-ft thick layer of angular subbase gravel exists beneath the asphalt layer. Noticeable soil-fill material (including apparent ash, brick, various forms of metal, slag, glass, wood, and apparent dolostone building footer blocks) was observed below the asphalt and asphalt subbase (gravel) layers, apart from test pit TP-02 (where urban fill was not observed). Soil beneath the urban fill/ash layer consisted generally of light tan to brown fine sand, some silt, little sub-rounded gravel including limestone, red sandstones, dolostones, trace amounts of chert, trace plastic clay, and trace cobbles. Apparent bedrock was encountered at depths ranging from 4.5 to 9.75 ft bgs. No oxidation-transition zone was identified in any of the ten (10) test pits completed. Dolomite bedrock of the Eramosa (Lockport) Formation underlays the overburden soil and fill material.

Hydrology

Based on the LaBella Phase II ESA dated October 2023, groundwater surrounding the Site is generally encountered between 5.05 and 9.73 ft bgs. Static water levels obtained in August 2023 from the on-site monitoring wells MW-01 and YS-MW-2023-01 were 5.05 and 5.54 ft bgs respectively. Based on static water levels obtained in August 2023 from wells surrounding the site and within the Site on, the general groundwater flow direction is towards the northwest.

5.0 APPLICABLE REGULATIONS AND CLEANUP STANDARDS

5.1 Remedial Oversight Responsibility

The cleanup will be performed by LaBella Associates, D.P.C. and LaBella Environmental, LLC, who are appropriately licensed, insured, and experienced to perform the activities described herein. The work shall

also be in coordination with the NYSDEC and the Stipulation Agreement made between the NYSDEC and the City of Rochester for NYSDEC Spill No. 2206496 (dated December 30, 2022).

5.2 *Standards, Criteria, and Guidance (SCG)*

The Site is located in a mixed residential/commercial area, with future proposed use being the same (mixed residential/commercial). As such, the following NYSDEC Standards, Criteria and Guidance (SCGs) appropriate for such proposed development apply:

Soil

- NYSDEC Part 375 Unrestricted Use Soil Cleanup Objectives (SCOs)
- NYSDEC Part 375 Restricted Residential Use SCOs
- NYSDEC Part 375 Commercial Use SCOs
- NYSDEC CP-51 Soil Cleanup Levels
- Maximum Concentration of Contaminants for Toxicity Characteristic (RCRA Hazardous Waste “D-List” – CFR Part 261)

Groundwater

- NYSDEC Division of Water Technical and Operational Guidance Series (1.1.1), Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations

5.3 *Remedial Action Objectives (RAOs)*

RAOs are medium-specific objectives for the protection of human health and the environment. RAOs for this project are as follows:

Soil

RAOs for Public Health Protection

- Prevent ingestion/direct contact with contaminated soil.
- Prevent inhalation of, or exposure from, contaminants volatilizing from contaminants in soil.

RAOs for Environmental Protection

- Prevent migration of contaminants that would result in groundwater or surface water contamination.

5.4 *Laws & Regulations Applicable to the Remedy*

The NYSDEC is the agency responsible for the cleanup and removal of discharges of petroleum pursuant to Article 12 of the Navigation Law and Article 17 of the Environmental Conservation Law. In accordance with the Stipulation Agreement made between the NYSDEC and the City of Rochester, the City has agreed to clean up and remove a discharge of petroleum at the Site which was reported to the NYSDEC on October 28, 2022 (NYSDEC Spill No. 2206496).

In accordance with 6 NYCRR Part 360.13, fill materials containing ash and cinders may be managed and placed into similar filled areas within the same site under appropriate cover. Alternatively, these materials can be disposed off-site in a New York State Part 360 permitted landfill.

All local laws, permits, and notification requirements (i.e., *UDig NY* notification, soil/fill transport/disposal manifest procedures, etc.) shall be obtained/completed and followed during remedial activities.

6.0 REMEDIAL ACTION IMPLEMENTATION

This section summarizes the remedial actions planned to address the soil-fill material. All work will be completed in accordance with LaBella's Health and Safety Plan (HASP) (refer to Appendix 3), Community Air Monitoring Plan (CAMP) (refer to Appendix 4), and Quality Assurance Project Plan (QAPP) (refer to Appendix 5). All laboratory analyses will be completed by a New York State Department of Health (NYSDOH) Environmental Laboratory Accreditation Program (ELAP)-certified laboratory. ASP Category B deliverables and Data Usability Summary Reports (DUSRs) will be prepared for confirmatory/documentation samples.

The remedy will consist of excavation and off-Site disposal of the soil-fill material.

6.1 Site Preparation

Prior to initiating remedial activities at the Site, the wooden bollards currently installed at the Site will be removed as needed and stored for use after project completion. A 6-foot-high temporary chain-link fence will be installed around the perimeter of the Site. The fence will be completed with one locked gate which will be utilized for the construction entrance. Additionally, a USEPA/NYSDEC sign will be created and adhered to the chain-linked fence with appropriate grant information and agency contact information. LaBella will provide the USEPA/NYSDEC sign and appropriately affix the sign to the Site fencing. The temporary fence will remain at the Site until remedial activities are complete and the Site has been restored to its pre-remedial grade.

Utility Stakeout / Locating

Prior to remedial excavation, a *UDig NY* stakeout will be requested to locate subsurface utilities where they enter the Site. LaBella assumes any relevant utility drawings and/or other information regarding underground utilities will be provided by the City prior to implementation of subsurface work at the Site.

Decommissioning of Existing Wells

Prior to remedial excavation, the following two (2) groundwater monitoring wells shall be decommissioned. The terminal depth of each well is also included and has been obtained from the Phase II ESA dated October 9, 2023.

- YS-MW-2023-01 (10.60-ft bgs)
- MW-01 (10.39-ft bgs)

The total length of wells to be decommissioned is approximately 20.99 feet.

Wells will be decommissioned per NYSDEC Commissioner's Policy (CP)-43. LaBella will remove and dispose of the surface completion (curb box, etc.) and cut the well material roughly 1 foot below the existing grade. The wells will be grouted in-place. For safety reasons, limited surface restoration shall be performed, but will not include repaving, etc.

LaBella shall provide field oversight of well decommissioning activities and document the work completed in accordance with NYSDEC CP-43, including the completion of well decommissioning logs.

This RWP assumes the CAMP shall not be implemented during well decommissioning activities.

Asphalt

Asphalt at the Site is broken-up, weathered, and in many areas comingled with underlying soil-fill material. It is anticipated that asphalt shall be transported and disposed alongside the soil-fill material. If asphalt is able to be segregated / cleaned of any fill/soil material, it will be recycled off-site as asphalt millings.

6.2 Excavation and Removal of Soil/Fill Material Exceeding Site SCGs

LaBella will mobilize to the Site to conduct the remedial excavation and construction oversight. The NYSDOH Generic CAMP included as Appendix 4 will be implemented during all ground intrusive activities.

Excavation

The excavation will extend to depths of presumably native material and/or bedrock which ranges from approximately 2.0-9.75-ft bgs, as shown on Figures 3 and 4.

Since the remedial excavation consists of a large footprint, the excavation may be completed in sections or “cells” to limit sidewall collapse and dewatering as well as to facilitate more efficient materials management. Each excavation cell will be advanced to the full depths required for remediation, and partially backfilled to a nominal depth of 1-2-ft above the water table. A vertical sheet of poly will be placed between the backfill area and adjacent unexcavated cell to prevent cross contamination of impacts into clean backfill. The poly sheeting will be removed when the next cell is excavated. Confirmatory/ documentation samples will be collected from the excavation sidewalls and/or bottom prior to backfilling as discussed in Section 6.5.

Bedrock

Based on previous assessment, it is not anticipated that gross petroleum impacted soil and/or bedrock will be encountered on Site. If petroleum contaminated bedrock is encountered during the remedial excavation, it is anticipated that up to the top 2-ft of rock in certain areas of the excavation will be pulverized using an excavator with a hoe ram breaker attachment and broken bedrock removed for off-Site disposal. Since petroleum contaminated bedrock has not been identified to-date, any such material would need to be properly characterized for waste disposal during the remedial activities.

Transportation and Disposal

All Disposal facilities and waste transporters must provide evidence of applicable NYSDEC permits prior to handling, transporting, and/or receiving impacted media. All operators responsible for the removal and disposal of contaminated media shall comply with the applicable Federal, State, and local laws, regulations, and policies. The contractor shall provide the City with documentation that the receiving facility is permitted to receive the accepted waste and the waste transporter is permitted to haul such waste. Documentation of proper disposal, including copies of all waste disposal manifests and disposal facility receipts shall be provided to the City within the weekly reporting period.

Soil and fill material have been tested during the Phase II ESA dated October 9, 2023 to determine it was non-hazardous material. Six (6) waste characteristic samples were collected and submitted for the following parameters described in the table below:

Sample Location	Sample Depth (ft bgs)	Sample ID	Material	Analysis
TP-01	0.25 – 5.0	WC-01-0.25-5 FT	Urban Fill & Ash	-TCLP VOCs -TCLP SVOCs -TCLP Metals -Total PCBs -Reactivity -Corrosivity (pH) -Ignitability
TP-06	0.4 – 3.0	WC-08-0.4-3 FT	Urban Fill & Ash	
TP-08	1.0 – 6.0	WC-03-1-6 FT	Urban Fill & Ash	
TP-09	0.75 – 2.0	WC-02-0.75-2 FT	Urban Fill & Ash	
TP-09	4.0 – 5.0	WC-05-4-5 FT	Soil (No Fill or Ash)	
TP-10	3.5 – 6.5	WC-04-3.5-6.5 FT	Urban Fill	

Analytical data from the above sample analysis will be provided to an approved New York State Part 360 permitted landfill for disposal.

Trucks will enter the Site from York Street directly onto 24, 32 or 42 York Street. Trucks or other construction equipment will not occupy other properties other than these 3 City-owned parcels.

Based on previous investigations, it is anticipated that the following materials and quantities will be encountered during this remedial excavation:

- **Soil-Fill Material** – Approximately 2,689 cubic yards (CY) of fill material, equal to approximately 4,302 tons (when using a 1.6 multiplier). All soil-fill material will be transported off-site to a NYSDEC Part 360 permitted landfill.
- **Petroleum Contaminated Soil and/or Bedrock** – Although it is not anticipated that gross petroleum contaminated soil is to be encountered during the remedial excavation, it is notable that the adjacent property addressed 24 & 32 York Street had petroleum impacts that may be present on 42 York Street. If petroleum contaminated soil and/or groundwater is encountered at 42 York Street during the remedial excavation, they will be removed.

6.3 Soil Screening/Management Methods

All field screening of soil and fill materials will be performed by a LaBella scientist, engineer, or geologist possessing the required qualifications and certifications for work on contaminated sites (i.e., 40-hour OSHA HAZWOPER certification, etc.). All excavated subsurface soils will be continuously assessed in the field for visible impairment, olfactory indications of impairment, and total volatile organic compounds (VOCs) using a photoionization detector (PID). Based on PID readings observed during previous investigations by others, it is not anticipated that gross petroleum contaminated soil will be encountered during the remedial excavation of the Site. However, if soil exhibits PID readings greater than 10 ppm, and/or evidence of petroleum odors and/or staining are observed, then it will be considered petroleum contaminated soil and handled appropriately.

To the extent practical, material to be transported and disposed off-site will be live-loaded into dump trucks to avoid double-handling material. When staging and stockpiling is required, all stockpiled materials will be staged on and covered with minimum 6-mil poly sheeting until transported off-Site for disposal. Poly sheeting will be secured to prevent erosion.

Reuse

Based on previous investigations, fill material is present beneath the asphalt layer at 42 York Street; as such, there is no uncontaminated soil that will be used as backfill. All material from the surface to the bottom of fill will be excavated and disposed of at a NYCRR Part 360 permitted landfill.

6.4 Groundwater Infiltration Management

According to previously prepared reports for the Site, the groundwater table has been encountered at approximately 5.0-6.0-ft bgs. Since the water table will be encountered at depths shallower than the terminal depth of the remedial excavation (6.5-7.0-ft bgs.) and because rain events may cause stormwater to accumulate in the excavations, dewatering appears to be warranted during excavation. As such, a temporary water storage container will be staged at the Site or at an adjacent City owned parcel. Water from the excavation, if encountered, will be pumped into the water storage container as needed to facilitate further excavation.

At the conclusion of the project or when the water storage container approaches maximum capacity, the contents will be sampled and discharged to public combined sewer under a Specialty Short Term Discharge, pending permit approval from Monroe County Pure Waters (MCPW). It is anticipated one (1) sample will be

collected from the water storage tank for analytical parameters consistent with current Monroe County permit requirements as follows:

- PPL Metals & mercury (EPA 200.7/245.1)
- PPL acids/ base/ neutrals, including PAHs (USEPA 625)

LaBella will discuss additional or subsequent sampling requirements with MCPW (including the potential need for VOCs analysis based on areal (off-site) petroleum impacts that may be encountered) based on field observations, the results of the initial sampling event, and volume of groundwater generated during the remedial event (i.e., the MCPW discharge permit may require additional samples be collected after certain volume thresholds are reached).

6.5 *Confirmatory/Documentation Sampling*

Prior to backfilling the excavation, confirmatory/documentation samples will be collected from the sidewalls and areas of the bottom of the excavation where soil is present (i.e., where excavation does not reach bedrock). The definition of each type of sample is as follows:

- **Documentation samples** will refer to samples collected from the perimeter of the excavation that may not represent final endpoint samples and may not meet SCOs/SCLs (i.e., additional soil/fill removal to meet SCOs/SCLs may be necessary in the future on adjacent properties, right-of-ways, or utility corridors or these sample locations may be managed in place via a Soil and Groundwater Management Plan during future redevelopment).
- **Confirmatory samples** will refer to endpoint samples that are collected from the bottom or perimeter of the excavation where the sampling results demonstrate that soil conditions meet applicable SCOs/SCLs. Confirmatory/ documentation samples will not be collected from the bottom of the excavation in areas of exposed bedrock.

The confirmatory/documentation samples will be collected in accordance with DER-10; one (1) sidewall confirmatory/documentation sample will be collected for every 40 linear feet of excavation perimeter, and one (1) bottom confirmatory/documentation sample will be collected for every 1,600-sq.ft. of excavation bottom area. While it is currently anticipated that some of the excavation bottom will consist of bedrock, the bottom confirmatory/ documentation sample quantities will be calculated based on the area of exposed soils at the excavation bottom. If minimal soil remains at the bottom of the excavation (less than 6 inches), it will be removed to bedrock.

Since the perimeter of the remedial excavation is currently anticipated to measure approximately 700-ft, up to eighteen (18) sidewall confirmatory/documentation soil samples will be collected. While the exact area of exposed soil at the bottom of the excavation is unknown, it is estimated that thirteen (13) bottom confirmatory/documentation soil samples will be collected. Quantities of confirmatory/documentation samples are subject to change based on actual excavation perimeter/ area. Each confirmatory/documentation soil sample will be submitted for laboratory analysis of the following:

- NYCRR Part 375 and CP-51 List VOCs using USPA Method 8260
- NYCRR Part 375 and CP-51 List SVOCs using USEPA Method 8270
- TAL Metals using USEPA Method 6010/7470

A blind duplicate and matrix spike/matrix spike duplicate sample will be collected from the confirmatory/documentation samples at a rate of one (1) per twenty (20) samples, respectively.

Samples will be sent under standard Chain of Custody procedures to a NYSDOH ELAP-certified laboratory. To reduce the amount of time the excavation will remain open without backfill, all confirmatory samples will be submitted with a rush turnaround time of approximately 3 to 5 business days.

Upon receipt of the analytical results, LaBella will share the data with the City in real time so the City can evaluate the effectiveness of the remedy on achieving the cleanup objectives for the Site. If soil sample results do not meet 6 NYCRR Part 375 Restricted Residential Use SCOs, Commercial Use SCOs, and NYSDEC CP-51 SCLs, the excavation may be expanded if feasible and confirmatory soil samples will be recollected following further excavation. If expansion of the excavation is not feasible due to proximity to infrastructure such as sidewalks, etc., LaBella will discuss it with the City and NYSDEC. Confirmatory/documentation sample locations and elevations will be recorded utilizing a global positioning system (GPS) or tape measured from site features. It should be noted that because the City is serviced by public water supply and groundwater at the Site is not used as a potable water source, Protection of Groundwater SCOs are not applicable for determining when the excavation is complete.

ASP Category B data deliverables will be provided by the laboratory. DUSRs will be completed by a third party for confirmatory/documentation soil samples. All sampling and analysis will be completed in accordance with the Quality Assurance Project Plan (QAPP) that has been prepared for the project and is included in Appendix 5.

6.6 *In-Situ Treatment Methods*

Based on the test pit study performed during the Phase II ESA dated October 9, 2023, it is not anticipated that petroleum related contamination will not be encountered on Site during remedial excavations. However, if petroleum contamination is encountered during the remedial excavation and cannot be removed properly during remediation, the City and NYSDEC will be notified, and *in-situ* treatment methods will be explored.

One treatment option is to first remove all petroleum contaminated soil and bedrock, and then apply the oxygen release compound (ORC) by Regenesis, ORC-Advanced®. ORC-Advanced® could be added to the remedial excavation to facilitate aerobic bioremediation of any residual petroleum contamination at the Site.

6.7 *Site Restoration*

Based on the conceptual development plans for this Site, redevelopment will likely entail the construction of a building and/or parking lot. As such, the excavation will be backfilled with crushed stone (CR2). It is assumed crushed stone will be exempt from analytical testing per DER-10, physical characteristics of the CR2 to be used will be documented to prove suitability. Backfill materials will be compacted to 95% of their maximum dry density to facilitate future development more readily.

6.8 *Monitoring Well Installation (If Applicable)*

Based historical analytical groundwater and soil/fill data from the Site, the installation of monitoring wells after the remedial excavation is not necessary. However, if petroleum impacted material is identified in the remedial excavation that cannot be removed, such as at the extent of the Site parcel near Ruby Street, then groundwater monitoring well installation may occur.

If petroleum impacted material is identified during the remedial excavation that cannot be removed, the need for post-remedial excavation groundwater monitoring will be evaluated and the following performed as part of the Post-Remedial Groundwater Monitoring program.

Any newly installed groundwater monitoring well(s) would be sampled quarterly for the first year after completion of the project. The need for further monitoring (and the frequency of such) would be determined based on the results of the first year of monitoring,

Each groundwater sample would be collected using low-flow methodology as follows:

1. Wells will be checked for NAPL immediately prior to groundwater sampling and static water levels will be collected.
2. Groundwater will be purged from each well using a bladder pump. The top of pump will be placed approximately in the center of the screened interval for each well.
3. Water quality parameters including turbidity, pH, temperature, specific conductivity, dissolved oxygen, and depth to water will be recorded at five (5) minute intervals during sampling until the parameters have stabilized for two (2) consecutive intervals within the specified ranges below, or after 60 minutes (whichever occurs first), at which time the samples will be collected:
 - o Water level drawdown (<0.3')
 - o Turbidity (+/- 10%)
 - o pH (+/-0.1)
 - o Temperature (+/- 3%)
 - o Specific conductivity (+/- 3%)
 - o Dissolved Oxygen (+/- 10%)
 - o Oxidation reduction potential (+/- 10 millivolts)

Samples will be submitted to a NYSDOH ELAP laboratory for analysis of the following:

- CP-51 list VOCs using USEPA Method 8260;
- CP-51 list SVOCs using USEPA Method 8270;
- TAL Metals using USEPA Method 6020/7471.

If SVOCs are non-detect during the first round of groundwater sampling for any well, that well will not be analyzed for SVOCs during subsequent groundwater monitoring events.

One (1) blind duplicate and MS/MSD will be collected during each groundwater sampling event. ASP Category B data deliverables will be provided by the laboratory. DUSRs will be completed for groundwater data. All sampling and analysis will be completed in accordance with the QAPP.

At the conclusion of each sampling event, a brief letter report will be prepared. The letter reports will include a description of the work performed, detailed summary of groundwater data, and a groundwater potentiometric contour map.

7.0 SOIL & GROUNDWATER MANAGEMENT PLAN DEVELOPMENT

The remedial action is expected to address all contamination at the Site. As such, future use of the Site may occur without environmental restriction and without a need for a Soil and Groundwater Management Plan (SGMP).

In the event that the remedial action is unsuccessful in achieving the cleanup goals (i.e., based on documentation sampling results and/or gross petroleum impacts encountered at the perimeter of the Site), a SGMP will be prepared for the Site in accordance with the NYSDEC Region 8 Spills Unit criteria to address any residual contamination.

8.0 SCHEDULE AND DELIVERABLES

Remedial activities are planned to begin in the Fall of 2024 and are expected to last one to three months.

During remedy implementation, LaBella will provide a weekly email summary to the City and NYSDEC summarizing the work completed, any samples collected, significant observations, deviations to the RWP (if any), and planned work for the following week.

8.1 Reporting

At the conclusion of the project, a Remedial Construction/Closure Report (RCCR) will be prepared for the Site. This report will document all remedial actions implemented, and include the following (at minimum):

1. Project background and pertinent history;
2. Remedial objectives;
3. Summary of all remedial work performed;
4. Field documentation in a field notebook and daily summaries;
5. Scaled drawings showing the Site location and layout, previous testing locations, confirmatory soil sample locations, and actual limits of excavation(s);
6. All quantities (tonnage) of all media disposed of;
7. Tabulated data for analytical results comparing to applicable cleanup criteria (i.e., Restricted Residential Use SCOs);
8. Laboratory analytical reports in ASP Category B format;
9. DUSRs;
10. CAMP data;
11. NYSDEC approvals of work plans, requests to import/reuse material, etc.;
12. Imported material documentation including weight tickets, laboratory data etc.;
13. Disposal documentation including weight tickets, landfill approval, laboratory data, etc.;
- and,
14. Photographs of the work performed with summary and date of each photograph.

Throughout the project, LaBella will keep a record of all remedial excavation limits, depths, and soil sample locations utilizing a GPS unit capable of recording locations on the US State Plane 1983 (New York Western Zone) coordinate system. The RCCR will be submitted as draft to the City for review prior to submittal to NYSDEC. LaBella will address any comments from the City and NYSDEC.

9.0 HEALTH AND SAFETY PLAN (HASP)

LaBella's HASP included in Appendix 3 will be implemented by all LaBella personnel. This HASP reflects LaBella's policy only and other contractors working on the Site will follow their own HASP.

10.0 QUALITY ASSURANCE PROJECT PLAN (QAPP)

The QAPP included in Appendix 5 will be implemented during this RWP/CAP. The QAPP was developed in accordance with *EPA QA/R-5 EPA Requirements for Quality Assurance Project Plans* as required per the EPA grant.

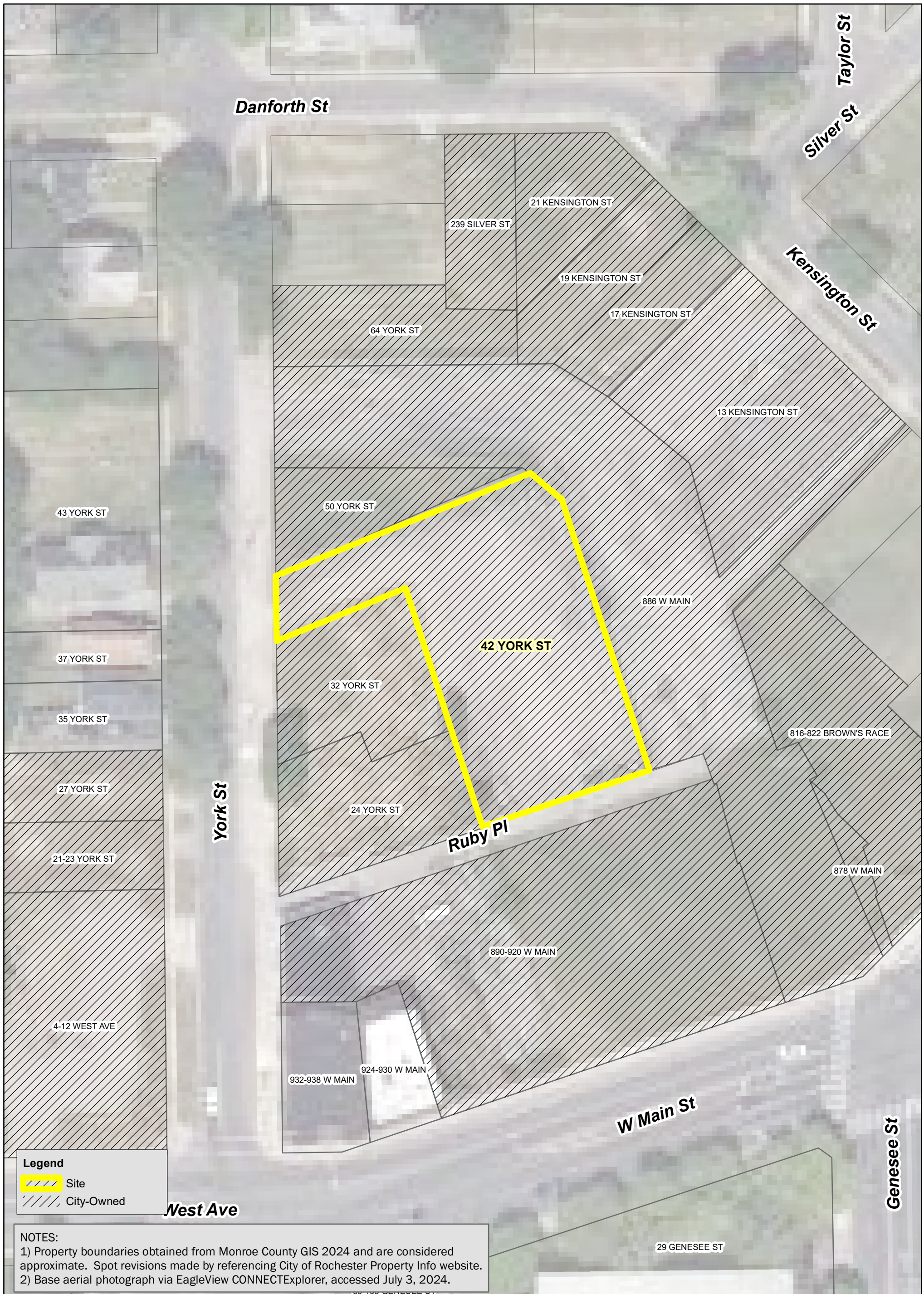
11.0 COMMUNITY AIR MONITORING PLAN (CAMP)

The NYSDOH Generic CAMP included as Appendix 4 will be implemented during all ground intrusive activities to monitor for dust and VOCs. The CAMP will include one upwind and one downwind station, and each station will include an airborne particulate monitor to measure aerosolized particulates and a PID to measure VOCs. Data will be continuously recorded at 15 minute intervals. If CAMP action levels are exceeded, measures will be implemented to reduce dust and/or VOCs as warranted. Dust suppression may include, but is not limited to, the use of potable water during excavation, working in discrete areas/stages to limit the area of exposed, unsealed soils vulnerable to dust production, or providing gravel roadways. VOC suppression may include the use of BioSolve®, a product with has been effective in quickly suppressing vapors and odors in other similar petroleum source removal projects. Any exceedances of the CAMP and subsequent corrective actions will be document and detailed in the final report.

\\PROJECTS2\PROJECTSNZ-2\ROCHESTER, CITY\2230119 - 42 YORK ST & 835-855 W MAIN ST\11_REPORTS\42 YORK - RWP\SPILL.2206496.2024-08-19.42_YORK_REMEDIAL_WORK_PLAN.DOCX



FIGURES

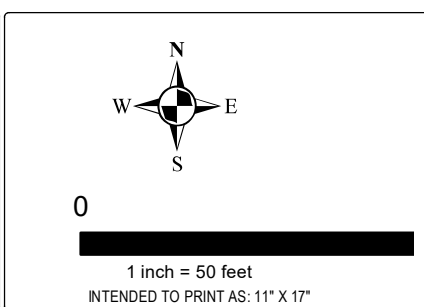


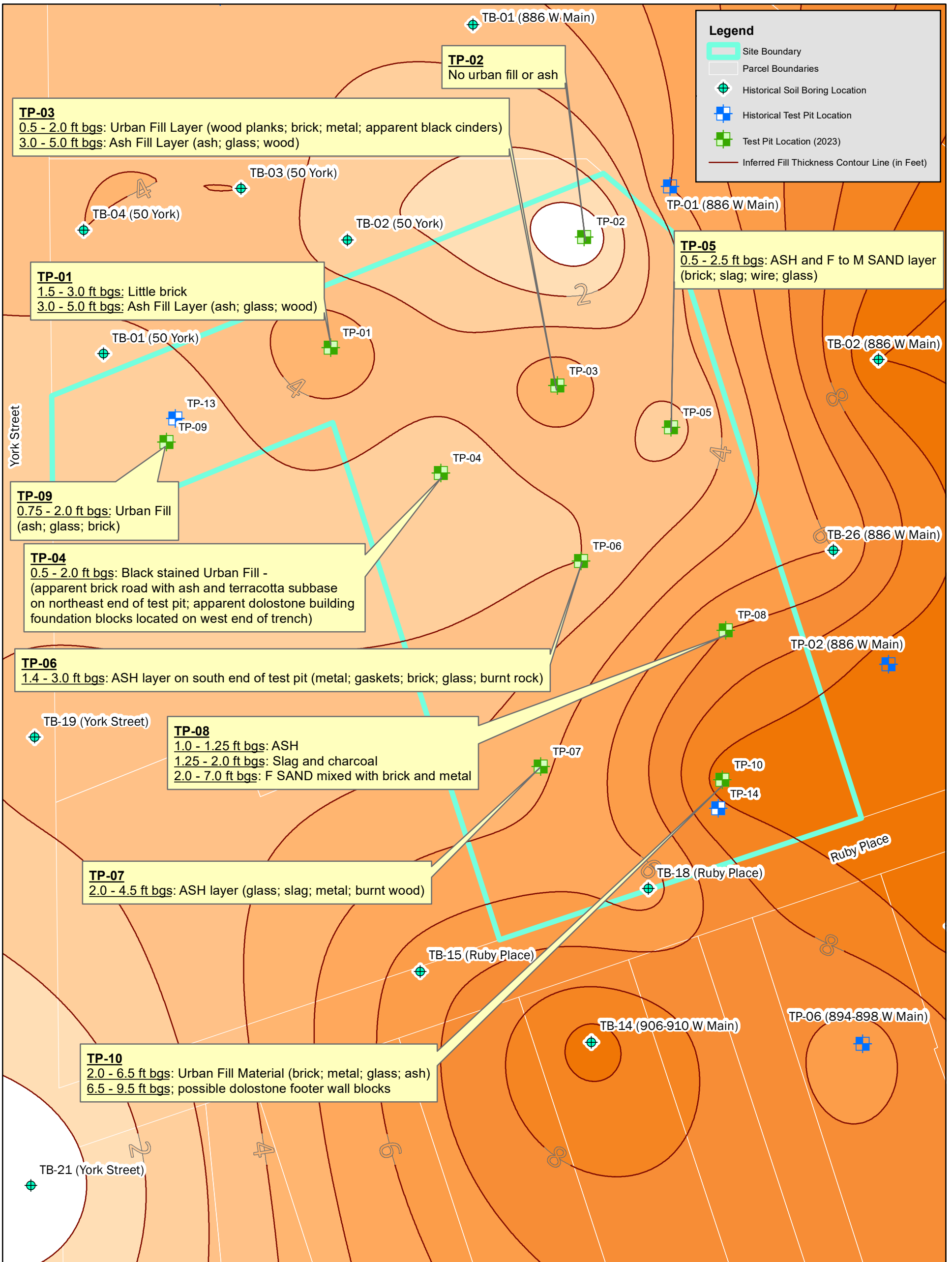
PROJECT/DRAWING NUMBER:
 [2230119]
 [FIGURE 2]

PROJECT:
**REMEDIAL WORK PLAN
 42 YORK STREET
 ROCHESTER, NEW YORK**

DRAWING NAME:
SITE PLAN MAP

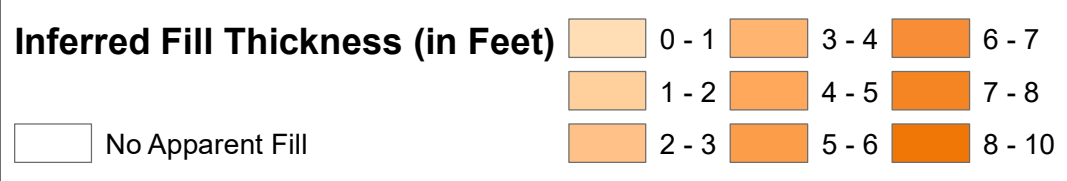
CLIENT:
CITY OF ROCHESTER





NOTES:

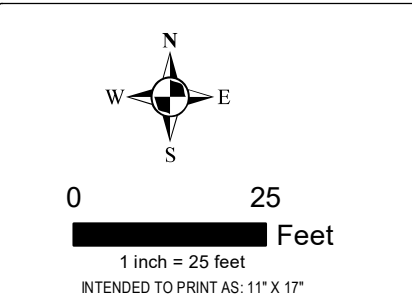
- 1) Property boundaries obtained from Monroe County GIS 2019 and considered approximate.
- 2) Fill depth contours were created in Surfer 23.2.176 via the Kriging method from depths recorded at the bottom of the urban fill layer measured in the test pits and surrounding borings. These contours are shown to illustrate general urban fill depth patterns in the context of this report. The contour lines are approximate and actual contours may vary from the locations shown. This data should be considered accurate to the degree implied by the method used.
- 3) Refer to test pit and soil boring logs for additional information.



PROJECT/DRAWING NUMBER:
[2230119]
[FIGURE 3]

PROJECT:
**REMEDIAL WORK PLAN
42 YORK STREET
ROCHESTER, NEW YORK**
DRAWING NAME:
**FILL LOCATION
AND DEPTHS**

CLIENT:
CITY OF ROCHESTER





APPENDIX 1

Project Contact List

Project Contact List
Remedial Work Plan
42 York Street, Rochester, New York 14614
NYSDEC Spill No. 2206496



Project Personnel				
Name	Title	Telephone Number	Organizational Affiliation	Responsibilities
Drew Brantner	Project Manager	585-287-9089	LaBella Associates, D.P.C.	Coordinate planning, sampling, reporting tasks, client liaison, project oversight, coordinate sampling and reporting
Ann Barber	Assistant Project Manager and Engineer	585-295-6289	LaBella Associates, D.P.C.	Client liaison and assist with planning and reporting.
Dan Noll	LaBella Quality Assurance Officer (QAO)	585-295-6611	LaBella Associates, D.P.C.	Provide input on quality of technical work completed
Alex daSilva	Environmental Geologist	585-295-6268	LaBella Associates, D.P.C.	Perform soil and groundwater sampling in accordance with QAPP, project reporting
Steve Rinker	Environmental Excavation / Construction Subcontractor	585-303-9403	LaBella Environmental, LLC	Coordinate / Implement the remedial program (excavation, transportation, disposal, etc.)
Rick Rynski	Grant Recipient	315-338-0393	City of Rochester	Manage grant budget and schedule, coordinate consultants
Harold Thurston	Environmental Specialist	585-428-6721	City of Rochester - Division of Environmental Quality	Project representative for grant recipient and all environmental aspects of project
Yocasta DeJesus	EPA Brownfields Project Officer (BPO)	212-637-4340	EPA Region 2	Provide grant administration and technical assistance as needed
Adly Michael	EPA Brownfields Quality Assurance Officer (QAO)	732-906-6161	EPA Region 2	Provide input on quality of technical work completed
Michael Zamiarski	Regional Spill Engineer	585-226-5438	NYSDEC Region 8	Oversee remedial activities, review reports/documents, etc.
Melissa Deyo	Analytical Laboratory	716-427-5229	Alpha Analytical	Analyze environmental samples collected during field portion of the projects
Stella Cuenco et. al.	Third Party Data Validator	760-827-1100	Laboratory Data Consultants, Inc.	Assess the validity of analytical data generated by the laboratory



APPENDIX 2

Phase II Environmental Site Assessment (October 9, 2023)

Phase II Environmental Site Assessment 42 York Street

Location:

42 York Street
Rochester, New York 14614

Prepared for:

City of Rochester
30 Church Street
Rochester, New York 14614

LaBella Project No. 2230119

October 9, 2023



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COMMON / FREQUENT ACRONYMS & ABBREVIATIONS

bgs – Below Ground Surface

CP-51 – (NYSDEC) Commissioner’s Policy #51 (for Soil Cleanup Guidance)

DEC – Department of Environmental Conservation

DUSR – Data Usability Summary Report

ELAP – Environmental Laboratory Approval Program

ESA – Environmental Site Assessment

eV – electron volt

ft – feet

IDW – Investigation Derived Waste

in - inch

NYCRR – New York Codes, Rules and Regulations

NYSDEC – New York State Department of Environmental Conservation

NYSDOH – New York State Department of Health

PAH – polycyclic aromatic hydrocarbon

PID – Photoionization Detector

ppb – Parts Per Billion

ppm – Parts Per Million

RCRA – Resource Conservation and Recovery Act

SCO – Soil Cleanup Objective

SSDS – Sub-Slab Depressurization System

SVOC – Semi-Volatile Organic Compound

TCL – Target Compound List

TCLP – Toxicity Characteristic Leachate Procedure

TOGS – Technical & Operational Guidance Series 1.1.1

TB-## - Test Boring (Number)

TCE – Trichloroethene

TP-## - Test Pit (Number)

USEPA – United States Environmental Protection Agency

VOC – Volatile Organic Compound



EXECUTIVE SUMMARY

Background and Objective

LaBella was retained by the City of Rochester to conduct a Phase II ESA of the property located at 42 York Street, in the City of Rochester, Monroe County, New York (“Site”). The Site is comprised of one approximate 0.48-acre parcel located within the Bull’s Head redevelopment area and is currently an unused paved parking lot located in an urban setting. This Phase II ESA is part of the USEPA Brownfields Multipurpose Grant Program awarded to the City of Rochester in 2021.

An environmental investigation was previously completed for the Site and surrounding properties (by others) in 2018. The previous investigation identified the presence of SVOCs and metals in soil/fill material on the Site, including select areas where heavy metals and SVOCs were present at concentrations exceeding NYSDEC Restricted Residential Use SCOs. The purpose of this Phase II ESA was to further define the nature and extent of contamination identified by the previous investigation. The primary scope of this Phase II ESA included the excavation of test pits, collection of soil samples, installation of a groundwater monitoring well, and collection of groundwater samples.

Scope

Ten (10) test pits were advanced at the Site and one (1) groundwater monitoring well was installed. Six (6) soil samples for further investigation/delineation of contaminants were collected and analyzed for VOCs, SVOCs, and metals concentrations. Six (6) soil samples for waste characterization purposes were analyzed for TCLP VOCs, TCLP SVOCs, TCLP Metals, PCBs, Reactivity, Corrosivity, and Ignitability. Two (2) groundwater samples (one from the newly installed monitoring well and one from an existing monitoring well) were analyzed for VOCs, SVOCs, and metals concentrations. Activities were conducted in accordance with the USEPA-approved QAPP (including collection of QA/QC samples).

Findings / Conclusions

Shallow soil at the Site consists of a layer of material typical of urban soil and fill. The fill layer is generally present across the Site, with varying thickness. The fill is generally thickest (approximately seven (7) feet) on the southern portion of the Site and thinner (approximately two (2) feet) on the northern portion of the Site. The fill contains one or more of the following constituents at each location investigated during this assessment:

- Ash;
- cinders;
- slag;
- brick;
- wood;
- metal;
- glass; and/or,
- stone/concrete fragments.

The finding of urban soil/fill containing ash and cinders is consistent with historic investigation of the Site and surrounding area performed in 2018 (by others).

Lead was detected in exceedance of applicable SCG in one (1) soil/fill sample collected during this assessment, expanding the footprint of previously identified extents where soil/fill in exceedance of applicable SCG is present on the Site. Various other metals (including cadmium, copper, mercury, nickel, and zinc) were detected in exceedance of Unrestricted Use SCOs in one or more samples of the urban soil/fill collected from the Site during this investigation.



Groundwater is present at an approximate depth of five to six (5-6) feet below existing ground surface at the Site. Groundwater is estimated to flow to the northwest across the Site, based on areal data collected during this investigation.

Although not detected in exceedance of applicable SCG, TCE was detected in groundwater at one sample location on the Site.

Apparent bedrock is generally present at an approximate depth of 7 to 9.5 feet below existing ground surface at the Site.

Fill/soil exceeding the standards for hazardous waste via TCLP analysis (waste characterization sampling) has not been identified at the Site.

Recommendation

Urban soil/fill material present at the Site includes (but is not limited to) ash and cinders. These fill materials are considered a solid waste by the NYSDEC that cannot be treated as construction and demolition (C&D) solid waste, due to the nature of its origin as a solid waste derived from an industrial source. In accordance with 6 NYCRR Part 360.13(c), fill materials containing ash and cinders may be managed and placed into similar filled areas within the same site under appropriate cover. Alternatively, these materials can be disposed off-site in a New York State Part 360 permitted landfill. Based on the proposed future development of the Site, the presence of benzo(a)pyrene, arsenic, and lead in exceedance of applicable SCGs (Restricted Residential Use and Commercial Use SCOs) in the urban soil/fill layer in discrete areas, and additional metals in exceedance of Unrestricted Use SCOs in additional areas, it is recommended that any such urban soil/fill be appropriately handled, transported, and disposed at a NYS Part 360 permitted landfill, rather than re-used on-site, as the presence of this fill material places a hindrance on the redevelopment of the property from both an environmental and geotechnical consideration.

In summary, remedial efforts targeting the removal of fill material and the associated SVOCs and metals impacts are recommended in order to provide a clean site that promotes redevelopment.



1.0 INTRODUCTION

LaBella Associates, D.P.C. (“LaBella”) was retained by the City of Rochester to conduct a Phase II Environmental Site Assessment (ESA) of the property located at 42 York Street, in the City of Rochester, Monroe County, New York, hereinafter referred to as the “Site” (see Figures 1 and 2). This Phase II ESA is part of the USEPA Brownfields Multipurpose Grant Program awarded to the City of Rochester in 2021.

The scope and conditions of this ESA were in accordance with Task 3 of LaBella’s Proposal dated August 3, 2022, and the Quality Assurance Project Plan (QAPP) prepared for the assessment (Revision Number 3, dated June 19, 2023).

1.1 Limitations & Exceptions

Work associated with this Assessment was performed in accordance with generally accepted environmental engineering and environmental contracting practices for this region. LaBella Associates, D.P.C., makes no other warranty or representation, either expressed or implied, nor is one intended to be included as part of its services, proposals, contracts, or reports.

In addition, LaBella cannot provide guarantees, certifications, or warranties that the property is or is not free of environmental impairment or other regulated solid wastes. The Client shall be aware that the data and representative samples from any given soil or groundwater sampling point may represent conditions that apply only at that particular location, and such conditions may not necessarily apply to the general Site as a whole and may change with time.

2.0 BACKGROUND

2.1 Site Location and Description

The Site is comprised of one approximate 0.48-acre parcel (SBL #120.42-2-72.001) located at 42 York Street, in the City of Rochester, Monroe County, New York. Refer to Figure 1 for the approximate Site location (map) and Figure 2 for a local site plan. The Site is within the Bull’s Head redevelopment area and is currently an unused paved parking lot. The Site is located in an urban setting.

2.2 Adjacent Property Use

The Site is presently bordered by the following properties:

Direction	Address	Current Land Use
North	50 York Street	Vacant Lot / Undeveloped
East	866 West Main Street	Vacant Lot / Gravel Parking
South	(Beyond Ruby Place ROW) Multiple (888-910 West Main Street)	Vacant Lot / Undeveloped
West	Multiple (24-32 York Street)	Vacant Lot / Undeveloped
	(Beyond York St ROW) Multiple (21-55 York Street)	Single Family Residential



2.3 Site History & Land Use

The Site appeared to be first developed with several residential dwellings and sheds/barns on portions of the parcel from 1892 to at least 1935.

On aerial photographs dated 1988, 1993, and 2003, approximately 15 vehicles are parked on the Site. In addition, apparent dark staining and miscellaneous items (which may be indicative of debris) appear to be located throughout the Site. The staining and debris on the Site may also be indicative of current or former industrial/manufacturing use of the property or affects from surrounding properties. Potential concerns associated with an industrial/manufacturing use of a property include the contamination of soil and/or groundwater if leaks/spills and/or improper handling/disposal of hazardous materials, petroleum products, and/or hazardous wastes has occurred.

2.4 Summary of Previous Studies

A previous environmental investigation was completed for the Site and surrounding properties by Day Environmental in 2018. The previous investigation identified the presence of semi-volatile organic compounds (SVOCs) and metals in soil/fill material on the Site. The prior investigation identified select areas where heavy metals and SVOCs were present at concentrations above Restricted Residential Use Soil Cleanup Objectives (SCOs). More specifically, these impacts were identified in samples collected from test pits TP-13 (sample depth 1-2-ft bgs) and TP-14 (sample depth 3.5-ft bgs), both located on the subject Site (42 York Street).

Further reference to the environmental investigation performed in 2018 is provided throughout this report and the included Figures. On Figures 2 and 5, the 2018 investigation locations are identified as “Historic Soil Boring” and “Historic Test Pit Location.”

3.0 OBJECTIVE

The purpose of this Phase II ESA was to further define the nature and extent of contamination identified by previous investigation of the Site, which identified the presence SVOCs and metals in fill material on the Site.

In addition to field screening and observation, up to six (6) soil samples for laboratory analysis of the following parameters was scoped. These six (6) soil samples were for further investigation and delineation purposes.

- Target Compound List (TCL) and NYSDEC Commissioner Policy (CP)-51 list Volatile Organic Compounds (VOCs) using USEPA Method 8260D;
- TCL and NYSDEC CP-51 list Semi-volatile Organic Compounds (SVOCs) using USEPA method 8270E; and,
- Target Analyte List (TAL) Metals using USEPA 6010D/7471B.

In addition to the six (6) investigation (aka delineation) samples (above), four (4) soil samples for waste characterization purposes were scoped for the following parameters:

- Toxicity Characteristics Leachate Procedure (TCLP) VOCs using USEPA Method 8260D/1311;
- TCLP SVOCs using USEPA Method 8270E/1311;
- TCLP Metals using USEPA Method 6010D/7471B/1311;
- Polychlorinated Biphenyls (PCBs) using USEPA Method 8082A;
- Reactivity using USEPA Methods 9010C and Ch. 7 of SW-846;
- Ignitability using USEPA Method 1030; and,
- Corrosivity (pH) using USEPA Method 9045D.



Finally, to assess groundwater conditions on the Site, two (2) groundwater samples for laboratory analysis of the following parameters was scoped:

- TCL and CP-51 list VOCs using USEPA Method 8260D;
- TCL and CP-51 list SVOCs using USEPA method 8270E; and,
- TAL Metals using USEPA method 6020B/7470A.

4.0 FIELD INVESTIGATION - METHODOLOGY

Field activities associated with this ESA occurred in July-August 2023.

4.1 Public Utility Stakeout and Private Mark-Out

Prior to the initiation of subsurface work, an underground utility stake-out, via *UDig NY*, was completed at the Site to locate public utilities at the perimeter of the Site and along easements.

To supplement the public utility stake-out and further verify that proposed test pit locations would not disrupt subsurface utilities, a private utility mark-out for the investigation areas was completed by On The Mark Utility Locating Services, Inc. on July 5, 2023.

4.2 Test Pitting Study and Soil Sample Analysis

LaBella personnel oversaw the advancement of ten (10) test pits (TP-01 through TP-10) at the Site on July 11-12, 2023. Test pit locations were determined by the results of the public and private utility stakeout, historical records, and limited to approved/accessible areas of the Site. Test pits were excavated to depths ranging from approximately 4.5 to 9.75 feet (ft) below ground surface (bgs). Test pit locations are depicted on Figure 3.

Test pits were advanced by LaBella ENV, LLC, using a Kubota KX080-4 excavator. Soil samples were collected from various depths in each test pit and were visually and physically examined by LaBella personnel. Observations were made of the general lithology, visible layering, evidence of non-native fill/historic fill materials, indications of chemical or other staining, odors, and other distinctive features. Portions of the soil from borings were field screened for the presence of VOCs using a PID equipped with a 10.6 electronvolt (eV) lamp. Positive indications from any of these screening methods are collectively referred to as “evidence of impairment”. PID data and observations from each test pit are included on the test pit logs presented in Appendix 2.

Samples were selected from the test pits for laboratory analysis based on field evidence for the presence of fill materials, field screening, and PID readings to address the environmental concerns identified at the Site. A summary of the soil samples submitted for laboratory analysis are summarized in Section 4.6 (Laboratory Analytical Program).

Upon completion of test pit activities, the excavated materials were returned to the test pits from which they originated from on a first-out, last-in basis. The excavator bucket and tracks were used to compact the backfilled material. No additional compaction / testing was performed. Asphalt present at the surface was segregated from soils and placed back on top of each test pit following backfill.

4.3 Groundwater Monitoring Well Installation & Sampling

4.3.1 Groundwater Monitoring Well Installation

LaBella personnel oversaw the installation of one (1) overburden / bedrock interface groundwater monitoring well (YS-MW-2023-01) on July 17, 2023. The groundwater monitoring well location was determined by the results of the public and private utility stakeout, historical records, and limited to



the accessible area of the Site. The groundwater monitoring well was advanced to a terminal depth of 11.0 ft bgs. The groundwater monitoring well location is depicted on Figure 3.

The groundwater monitoring well was installed by LaBella ENV, LLC, using a Geoprobe Model 7822DT rig. Hollow stem augers having a 4.25-inch interior diameter and an 8-inch exterior diameter, were advanced into the upper 2-feet of bedrock, which was identified at an approximate depth of 7-feet 10-inches bgs. LaBella personnel logged and screened the soil using a PID during well installation. The monitoring well was constructed with 8-ft of 0.010-slot well screen of 2-inch inner diameter Schedule 40 polyvinyl chloride (PVC) screen attached to solid riser piping of the same material to complete the well. The well was installed as an overburden/bedrock interface well that was intended to intersect the top of the overburden groundwater table. The annulus was sand packed with quartz sand to a nominal depth of 1-ft above the screen section and a 0.8-ft bentonite seal was placed above the sand pack. Monitoring well YS-MW-2023-01 was completed with a flush mount well cover grouted into place. The groundwater monitoring well construction log is presented in Appendix 2.

4.3.2 Groundwater Monitoring Well Development

Lu Engineers developed the newly installed groundwater monitoring well (YS-MW-2023-01) on July 19, 2023 (approximately 48 hours after installation). Development occurred by the use of a submersible pump until the well was dry (after approximately 5 gallons (equal to approximately 6.4 well volumes) was removed from the well).

Turbidity noticeably improved during the well development process, based on visual observation.

The groundwater well development log is presented in Appendix 2.

4.3.3 Groundwater Sampling

Lu Engineers collected groundwater samples from monitoring wells YS-MW-2023-01 and MW-01 on July 26, 2023 (one (1) week after development of YS-MW-2023-01). Groundwater samples were collected using low-flow methodologies (bladder pump).

Groundwater purge/flow rates were minimized in an effort to limit drawdown of groundwater in the wells (i.e., to ensure the water table remained within proximity to the initial static water level depth). During purging activities, groundwater passed through a flow through cell equipped with a YSI Pro DDS water quality meter that measured certain groundwater quality parameters. After passing through the cell, the groundwater was discharged and temporarily contained in 5-gallon buckets (before ultimate placement in a 55-gallon drum, labeled, and left on-site pending proper disposal). The following water quality parameters were measured and recorded at five (5) minute intervals during the groundwater purge and sampling process. Groundwater samples for laboratory analysis were collected once the parameters stabilized according to the provided allowances:

- Temperature (+/- 3%)
- pH (+/- 0.1 unit)
- Dissolved oxygen (+/- 10%)
- Specific conductance (+/- 3%)
- Oxidation reduction potential (+/- 10 millivolts)
- Turbidity (+/- 10% or <50 NTU for metals is possible)

Groundwater samples submitted for laboratory analysis are summarized in Section 4.6 (Laboratory Analytical Program). Groundwater sampling logs are included in Appendix 2.

4.4 Investigation Location and Elevation Survey

Costich Engineering, D.P.C., a professional land surveyor (PLS), collected investigation location and elevation information on August 3, 2023, under the supervision and direction of LaBella personnel.



The survey included collection of data from beyond the limits of the Site, where historic investigation has occurred (i.e., historic/existing groundwater monitoring wells in the vicinity of the Site) in support of preparing an areal groundwater elevation contour map.

The information collected during the survey has been used throughout this report, including all Figures herein. The complete survey has been included as Appendix 1.

4.5 Community Air Monitoring Program

Air monitoring was completed in accordance with the NYSDOH Generic Community Air Monitoring Plan (CAMP) during intrusive subsurface activities. The CAMP includes the use of PIDs and particulate monitoring equipment (i.e., 'DustTrak') to document VOCs and airborne dust/particulate in real-time. Data from upwind (background) and downwind of the work area is compared over fifteen (15) minute intervals.

Air monitoring instruments were calibrated and maintained in accordance with the manufacturer's specifications.

No exceedances of applicable air quality standards were observed during field investigation activities where active air monitoring (CAMP implementation) occurred.

Refer to Appendix 4 for CAMP data and equipment calibration certificates.

4.6 Laboratory Analytical Program

Soil and groundwater samples collected for laboratory analysis were placed directly into laboratory-supplied containers, preserved as appropriate in a cooler, and submitted under standard chain-of-custody protocol to the local office of Alpha Analytical (in Rochester, NY) for courier service to Alpha Analytical's laboratory in Westborough, Massachusetts. Alpha Analytical is a New York State Department of Health (NYSDOH) Environmental Laboratory Approval Program (ELAP) certified laboratory.

The following samples were submitted for laboratory analysis:

4.6.1 Soil Samples for Laboratory Analysis

Sample ID	Exploration Location	Sample Depth (ft bgs)	Laboratory Analysis
TP-02-3-4 FT	TP-02	3.0 - 4.0	<ul style="list-style-type: none">• CP-51 & TCL VOCs and TICs• CP-51 & TCL SVOCs and TICs• TAL Metals
TP-03-0.5-2 FT	TP-03	0.5 - 2.0	
TP-04-0.5-2.5 FT	TP-04	0.5 - 2.5	
TP-05-0.5-2.5 FT	TP-05	0.5 - 2.5	
TP-06-0.4-3 FT	TP-06	0.4 - 3.0	
TP-07-2.0-4.5 FT	TP-07	2.0 - 4.5	
BD-01-3-4 FT (TP-02-3-4 FT)	TP-02	3.0 - 4.0	

(table continues on next page)



Sample ID	Exploration Location	Sample Depth (ft bgs)	Laboratory Analysis
WC-01-0.25-5 FT	TP-01	0.25 - 5.0	<ul style="list-style-type: none"> • TCLP VOCs • TCLP SVOCs • TCLP Metals • PCBs • Reactivity • Ignitability • Corrosivity (pH)
WC-02-0.75-2 FT	TP-09	0.75 - 2.0	
WC-03-1-6 FT	TP-08	1.0 - 6.0	
WC-04-3.5-6.5 FT	TP-10	3.5 - 6.5	
WC-08-0.4-3 FT	TP-06	0.4 - 3.0	

Table Notes:

1. USEPA TCL and NYSDEC Commissioner Policy (CP-51) list volatile organic compound (VOC) analysis performed via USEPA Method 8260D.
2. USEPA TCL and NYSDEC CP-51 lists of semi-volatile organic compounds (SVOCs) analysis performed via USEPA Method 8270E.
3. Target Analyte List (TAL) metals analysis performed via USEPA Methods 6010D.
4. Tentatively Identified Compounds (TICs) are an additional tool the USEPA uses to characterize potentially hazardous site. The USEPA refers to chemicals observed in the analysis, but not on the "Target Compound List" (TCL), as unknown compounds.
5. Toxicity Characteristic Leaching Procedure (TCLP) analysis performed via USEPA 1311.
6. Polychlorinated Biphenyls (PCBs) analysis performed via USEPA method 8082A.
7. Ignitability analysis performed via USEPA 1030.
8. Reactive Cyanide and Sulfide analysis performed via USEPA 9010C and Ch. 7.
9. Corrosivity (pH) analysis performed via USEPA method 9045D.

4.6.2 Groundwater Samples for Laboratory Analysis

Sample ID	Exploration Location	Screened Interval (ft bgs)	Laboratory Analysis
YS-MW-2023-01	YS-MW-2023-01	3.0 - 11.0	<ul style="list-style-type: none"> • CP-51 & TCL VOCs • CP-51 & TCL SVOCs • TAL Metals
MW-01	MW-01	3.5 - 10.5	
YS-MW-BD-072623 (YS-MW-2023-01)	YS-MW-2023-01	3.0 - 11.0	

Table Notes:

1. USEPA TCL and NYSDEC CP-51 list VOCs analysis performed via USEPA Method 8260D.
2. USEPA TCL and NYSDEC CP-51 list SVOCs analysis performed via USEPA Method 8270E.
3. TAL metals analysis performed via USEPA Method 6020B/7470A.

4.7 Investigation Derived Waste

Two (2) fifty-five (55) gallon steel drums of investigation derived waste (IDW) were generated during this investigation:

- One (1) drum of excess soil cuttings generated by the installation of groundwater monitoring well YS-MW-2023-01; and,
- One (1) drum of groundwater generated by the development of groundwater monitoring well YS-MW-2023-01 and the pre-sampling purging of groundwater monitoring wells YS-MW-2023-01 and MW-01 (historic well).

Both drums are only partially filled and have been appropriately labeled and staged on-site. Additional groundwater generated by potential future sampling/monitoring events may be added to the



groundwater drum. Each drum is pending appropriate future disposal.

4.8 Deviations to Objective

Based on field observations and a re-evaluation of the estimated quantity of fill material present on the Site, two (2) additional waste characterization samples (for a total of six (6) waste characterization samples) were collected during this assessment. The additional samples are necessary for obtaining approval to dispose of the additional material expected to require off-site transport/disposal, based on fill layer thickness observations made during the field investigation.

5.0 FINDINGS

5.1 Utility Locating

LaBella notified *UDig NY* of the pending subsurface investigation planned for the Site so that public buried utilities would be field-marked prior to initiating any excavation/test pits or groundwater monitoring well installation activities. Via the *UDig NY* stakeout process, no public utilities were identified in an area of concern relative to the proposed investigation.

On July 5, 2023, a private utility locator (On The Mark Locating) used GPR and electro-magnetic sensing equipment to clear an approximately fifteen (15) foot radius around each investigation area to re-confirm that no buried utilities or other subsurface anomalies were present.

No buried utilities or other subsurface anomalies were identified by the private utility locating process.

5.2 Test Pit Evaluation - Localized Geology and Hydrology

5.2.1 Geology (Soil)

Ten (10) test pits were advanced at the Site on July 12-13, 2023, designated TP-01 through TP-10.

Most of the Site is covered by an approximately 0.25-ft thick layer of broken asphalt. An approximately 0.5-ft thick layer of angular subbase gravel exists beneath the asphalt layer. Noticeable urban fill (including apparent ash, brick, various forms of metal, slag, glass, wood, and apparent dolostone building footer blocks) was observed below the asphalt and asphalt subbase (gravel) layers, with the exception of test pit TP-02 (where urban fill was not observed). Soil beneath the urban fill/ash layer consisted generally of light tan to brown fine sand, some silt, little sub-rounded gravel including limestone, red sandstones, dolostones, trace amounts of chert, trace plastic clay, and trace cobbles. Apparent bedrock was encountered at depths ranging from 4.5 to 9.75 ft bgs. No oxidation-transition zone was identified in any of the ten (10) test pits completed.

Below is a table summarizing the fill identified in each of the ten (10) test pits advanced during this investigation:

Location / Sample ID	Depth Range of Identified Fill (ft bgs)	Identified Fill Material Type
TP-01	1.5 - 3.0	Little brick
	3.0 - 5.0	Ash layer including glass and wood
TP-02	<i>No identified fill material</i>	
TP-03	0.5 - 2.0	Wood planks; brick; metal; apparent black cinders
	3.0 - 5.0	Ash fill layer including glass and wood



Location / Sample ID	Depth Range of Identified Fill (ft bgs)	Identified Fill Material Type
TP-04	0.5 - 2.0	Black stained fill material – apparent brick road with ash and terracotta subbase on the northeast end of test pit; apparent dolostone building foundation blocks on the west end of the test pit.
TP-05	0.5 - 2.5	Ash layer including brick, slag, wire, and glass
TP-06	1.4 - 3.0	Ash layer on the south end of test pit including metal; gaskets; brick; glass; apparent burnt rock
TP-07	2.0 - 4.5	Ash layer including glass; slag; metal; burnt wood
TP-08	1.0 - 1.25	Ash
	1.25 - 2.0	Slag; charcoal
	2.0 - 7.0	Fine sand intermixed with brick and metal
TP-09	0.75 - 2.0	Ash; glass; brick
TP-10	2.0 - 6.5	Brick; metal; glass; ash
	6.5 - 9.5	Possible dolostone footer wall blocks

Refer to Figure 6 for a depiction of fill locations and depths.

5.2.2 Hydrology (Groundwater)

Groundwater was encountered in test pits TP-06 and TP-07 at depths of approximately 6.0 and 5.4 ft bgs, respectively.

The limited number of groundwater monitoring wells on the Site limits the ability to infer groundwater flow direction solely based on Site data. In lieu of such, depth to groundwater data collected from existent groundwater monitoring wells in the vicinity of the Site (on adjacent and nearby City-owned parcels) was used to generate an areal groundwater contour map. Figure 4 displays approximate groundwater elevation contours and flow direction using surveyed elevations and depth to groundwater measurements collected on August 3, 2023. Based on the measurements and Figure 4, groundwater appears to flow to the northwest in the vicinity of the Site, generally consistent with the assumed regional flow toward Lake Ontario, north of the Site.

5.3 Field Screening Results

The table below summarizes PID readings obtained at various depth intervals from the Phase II investigation. As shown in the table, no detectable PID response was observed during the investigation.

Summary of PID Readings

Location ID	Approximate Sample Interval (ft bgs)				
	0-2	2-4	4-6	6-8	8-10
TP-01	0.0	0.0*	0.0*	0.0	0.0
TP-02	0.0	0.0*	0.0	0.0	0.0
TP-03	0.0*	0.0	0.0	0.0	0.0
TP-04	0.0*	0.0	0.0	0.0	0.0
TP-05	0.0*	0.0*	0.0	--	--
TP-06	0.0*	0.0*	0.0	0.0	--
TP-07	0.0	0.0*	0.0*	0.0	--



Location ID	Approximate Sample Interval (ft bgs)				
	0-2	2-4	4-6	6-8	8-10
TP-08	0.0*	0.0	0.0	0.0	--
TP-09	0.0	0.0	0.0	0.0	0.0
TP-10	0.0	0.0	0.0	0.0	0.0

Notes:

1. All PID readings were collected utilizing a Minirae 3000 photoionization detector and are expressed in parts per million.
2. The PID screening is performed as a method of determining general presence of VOCs in soil, and to provide a basis for selecting samples for laboratory analysis. The readings obtained provide only an indication of the relative levels of VOC presence in the soil and is not considered to be a direct quantization of actual soil VOC concentration.
3. "--" denotes boring not completed to above-listed depth or insufficient recovery occurred at specified depth.
4. "*" denotes a soil sample was submitted for laboratory analysis from this interval.

5.4 Laboratory Analytical Results

The purpose/objective of the Phase II ESA is to evaluate the nature and extent of contamination at the Site to determine the remediation necessary to redevelop the Site. The Site is located in a mixed residential/commercial area; therefore, the data generated during the course of the project is compared to the following NYSDEC Standards, Criteria and Guidance (SCGs) most appropriate for such proposed redevelopment.

Soil

- NYSDEC Part 375 Restricted Residential Use Soil Cleanup Objectives (SCOs) (12/14/2006)
- NYSDEC Part 375 Commercial Use SCOs (12/14/2006)
- NYSDEC CP-51 Soil Cleanup Levels (10/21/2010)
- Maximum Concentration of Contaminants for Toxicity Characteristic (RCRA Hazardous Waste "D-List" – CFR Part 261)

Groundwater

- NYSDEC Division of Water Technical and Operational Guidance Series (1.1.1), Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations (6/1998 and subsequent updates).

5.4.1 Soil Sample Results

VOCs:

Soil samples from six (6) locations were analyzed for VOCs. VOCs were not detected in any of the samples. As such, no exceedances of applicable VOC SCGs were identified.

SVOCs:

Soil samples from six (6) locations were analyzed for SVOCs. SVOCs were detected in two (2) of the samples. However, no exceedances of applicable SVOC SCGs were identified.

Metals:

Soil samples from six (6) locations were analyzed for metals. Metals were detected in each of the samples (Note: many metals are naturally occurring in the environment and are routinely detected at 'background' concentrations in soil. Only when metals concentrations exceed Unrestricted Use SCOs are they of potential concern).

At least one metal was detected at a concentration exceeding its Unrestricted Use SCO in five (5) of



the samples. Exceedances of Unrestricted Use SCO for metals included at least one of the following:

- cadmium;
- copper;
- lead;
- mercury;
- nickel; and/or,
- zinc.

Lead was detected at a concentration exceeding its Restricted Residential Use SCO and Commercial Use SCO in one (1) sample/location (TP-04).

For a complete summary of analyzed and detected compounds in soil, refer to Tables 3A, 3B, and 3C. Refer also to Figure 5 (Summary of Soil Conditions).

5.4.2 Groundwater Sample Results

VOCs:

Groundwater samples from two (2) locations were analyzed for VOCs.

The VOCs trichloroethene (TCE) and acetone were detected at one location; however, the detected concentrations do not exceed their applicable SCGs.

No other VOCs were detected in the groundwater samples collected.

SVOCs:

Groundwater samples from two (2) locations were analyzed for SVOCs.

The SVOC pentachlorophenol (PCP) was detected at both locations; however, the detected concentrations do not exceed the applicable SCG.

Trace concentrations of various polyaromatic hydrocarbons (PAHs – a subset of SVOCs) were detected at one location. However, the detected concentrations were below the laboratory method reporting limit and are therefore qualified as estimates, and the results were not reproduced in the field duplicate collected from the same location.

Metals:

Groundwater samples from two (2) locations were analyzed for metals.

Metals were detected in each of the samples (*Note: many metals are naturally occurring in the environment and are routinely detected at 'background' concentrations in soil and groundwater. Only when metals concentrations exceed SCGs are they of potential concern*).

Iron was detected at a concentration exceeding its SCG in both locations. The detected concentration was not reproduced in the field duplicate collected from the same location (one field duplicate).

For a complete summary of analyzed and detected compounds in groundwater, refer to Tables 4A, 4B, and 4C.

5.4.3 Waste Characterization Sample Results

Six (6) soil samples were collected and analyzed for waste characterization purposes, including via Toxicity Characteristic Leachate Procedure (TCLP) procedures, for total polychlorinated biphenyls



(PCBs), and hazardous waste characteristics (reactivity, ignitability, and corrosivity).

TCLP VOCs:

No targeted VOCs were detected by TCLP analysis of the soil/fill.

TCLP SVOCs:

No targeted SVOCs were detected by TCLP analysis of the soil/fill.

TCLP Metals:

Trace concentrations of barium, cadmium, and lead were detected by TCLP analysis of the soil/fill; however, the detected concentrations do not exceed the applicable TCLP standard.

Reactivity:

Reactive cyanide and sulfide were not detected within the soil/fill.

Corrosivity:

The soil/fill is relatively neutral (non-corrosive), with a pH ranging from 7.07 to 8.32.

Ignitability:

The soil/fill is not ignitable (i.e., flash point greater than 140° F).

5.4.4 Quality Assurance / Quality Control

Soil QA/QC Samples

One Matrix Spike/Matrix Spike Duplicate (MS/MSD) for VOCs, SVOCs, and metals in soil was collected.

One blind field duplicate for VOCs, SVOCs, and metals in soil was collected. The blind field duplicate results are reported alongside their respective parent sample in the attached Summary Tables. There was general agreement between the duplicate and parent sample results for all parameters.

Groundwater QA/QC Samples

One MS/MSD for VOCs, SVOCs, and metals in groundwater was collected.

One blind field duplicate for VOCs, SVOCs, and metals in groundwater was collected. The blind field duplicate results are reported alongside their respective parent sample in the attached Summary Tables. There was general agreement between the duplicate and parent sample results for VOCs. Trace concentrations of SVOCs in the parent sample were not reproduced in the duplicate sample for SVOCs. For metals analysis, the SCG exceedance of iron identified in the parent sample was not reproduced in the duplicate sample.

It is possible that turbidity varied between the parent and duplicate groundwater samples (i.e., that the parent sample was more turbid than the duplicate sample), which can result in differing results for SVOCs and metals. A more turbid sample may contain concentrations of SVOCs and metals as a result of the presence of suspended sediment.

A Trip Blank accompanied the groundwater samples collected for VOCs analysis. No VOCs were detected in the Trip Blank sample, indicating no concerns with cross-contamination of the groundwater samples by VOCs during sample collection/handling and transport.

Data Usability Summary Report

Data Usability Summary Reports (DUSR) and third-party data validation was provided by Laboratory Data Consultants, Inc. A separate DUSR was prepared for the soil and groundwater data obtained during this assessment, since the samples were collected on different days and submitted under



separate chain of custody (i.e., separate laboratory reports).

As stated in the DUSR, all results are usable as reported or usable with minor qualification due to laboratory quality control outliers and/or sample matrix. The complete DUSR are included in Appendix 6 for reference.

Qualifiers added by the data validation process have been included in the Summary Tables, where applicable.

6.0 CONCLUSIONS

Based on the findings of this Phase II ESA, the following conclusions have been drawn:

- Shallow soil at the Site consists of a layer of material typical of urban soil and fill. The fill layer is generally present across the Site, with varying thickness. The fill is generally thickest (approximately seven (7) feet) on the southern portion of the Site and thinner (approximately two (2) feet) on the northern portion of the Site. The fill contains one or more of the following constituents at each location investigated during this assessment:
 - Ash;
 - cinders;
 - slag;
 - brick;
 - wood;
 - metal;
 - glass; and/or,
 - stone/concrete fragments.

Urban fill materials (including those containing ash and cinders) are considered a solid waste by the NYSDEC (see Section 7.0 – Recommendations, for further information).

The finding of urban soil/fill containing ash and cinders is consistent with historic investigation of the Site and surrounding area performed in 2018 (by others).

- Lead was detected in exceedance of applicable SCG in the soil/fill sample collected from TP-04 (0.5-2.5 ft bgs) (material unsuitable to remain where Restricted Residential or Commercial Use is planned). This finding expands the footprint of previously identified extents where soil/fill in exceedance of applicable SCG is present on the Site (i.e., TP-13 1-2 ft bgs and TP-14 3.5 ft bgs).
- Various other metals (including cadmium, copper, mercury, nickel, and zinc) were detected in exceedance of Unrestricted Use SCOs in one or more samples of the urban soil/fill collected from the Site during this investigation.
- Groundwater is present at an approximate depth of five to six (5-6) feet below existing ground surface at the Site. Groundwater is estimated to flow to the northwest across the Site, based on areal data collected during this investigation.
- Iron was detected in exceedance of applicable SCG in the two (2) groundwater samples collected from the Site.
- Although not detected in exceedance of applicable SCG, TCE was detected in groundwater at one sample location on the Site.



- Apparent bedrock is generally present at an approximate depth of 7 to 9.5 feet below existing ground surface at the Site.
- Fill/soil exceeding the standards for hazardous waste via TCLP analysis has not been identified at the Site, based on field observations and analytical data.

7.0 RECOMMENDATION

Urban soil/fill material present at the Site includes (but is not limited to) ash and cinders. These fill materials are considered a solid waste by the NYSDEC that cannot be treated as construction and demolition (C&D) solid waste, due to the nature of its origin as a solid waste derived from an industrial source. In accordance with 6 NYCRR Part 360.13(c), fill materials containing ash and cinders may be managed and placed into similar filled areas within the same site under appropriate cover. Alternatively, these materials can be disposed off-site in a New York State Part 360 permitted landfill. Based on the proposed future development of the Site, the presence of benzo(a)pyrene, arsenic, and lead in exceedance of applicable SCGs (Restricted Residential Use and Commercial Use SCOs) in the urban soil/fill layer in discrete areas, and additional metals in exceedance of Unrestricted Use SCOs in additional areas, it is recommended that any such urban soil/fill be appropriately handled, transported, and disposed at a NYS Part 360 permitted landfill, rather than re-used on-site, as the presence of this fill material places a hindrance on the redevelopment of the property from both an environmental and geotechnical consideration. In summary, remedial efforts targeting the removal of fill material and the associated SVOCs and metals impacts are recommended in order to provide a clean site that promotes redevelopment.

8.0 CLOSING AND SIGNATURE OF ENVIRONMENTAL PROFESSIONAL

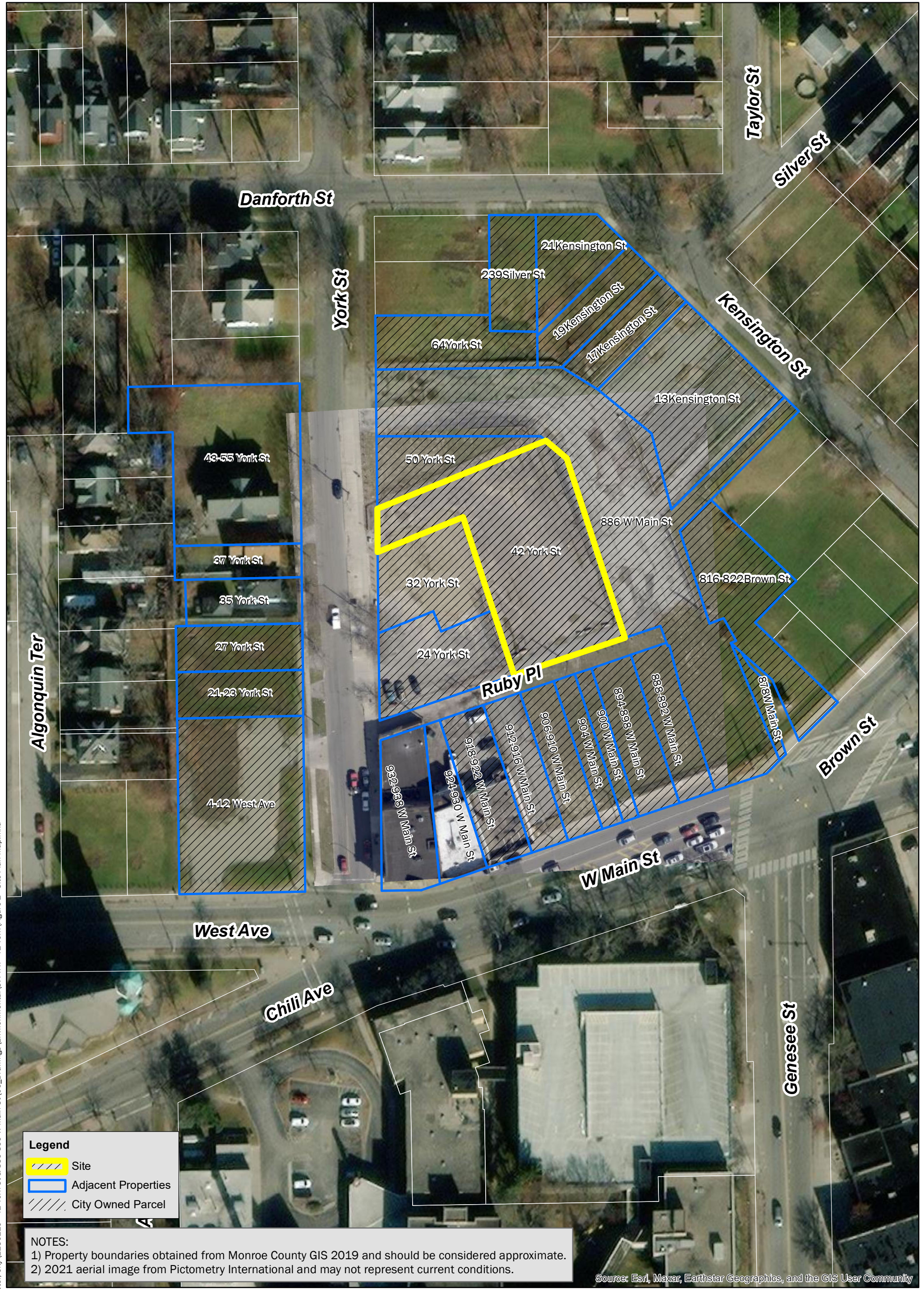
Thank you for the opportunity to provide our professional environmental engineering and consulting services for this project. If you have any questions pertaining to this report, please feel free to reach out to me directly at 585-287-9089 or at dbrantner@labellapc.com.

Alex daSilva
Staff Geologist

Drew Brantner
Project Manager/Qualified Environmental
Professional



FIGURES

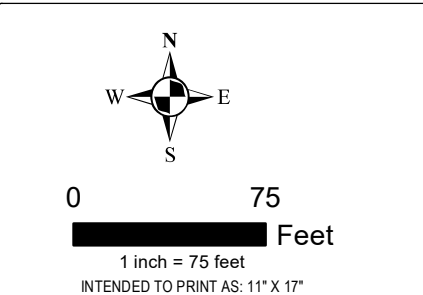


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 [FIGURE 2]

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 42 YORK STREET
 ROCHESTER, NEW YORK**

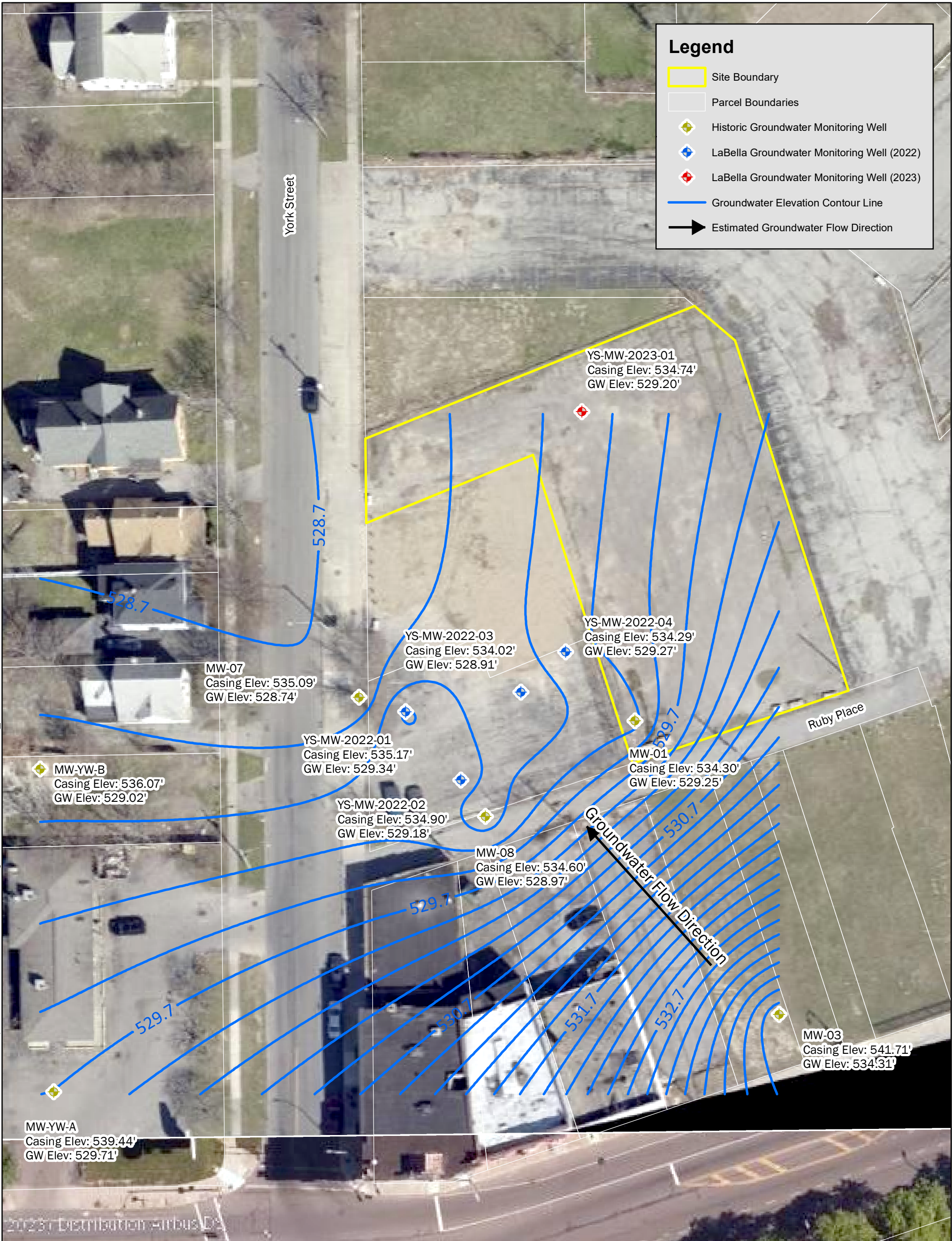
DRAWING NAME:
SITE PLAN MAP

CLIENT:
CITY OF ROCHESTER



Legend

- Site Boundary
- Parcel Boundaries
- ◆ Historic Groundwater Monitoring Well
- ◆ LaBella Groundwater Monitoring Well (2022)
- ◆ LaBella Groundwater Monitoring Well (2023)
- Groundwater Elevation Contour Line
- ➔ Estimated Groundwater Flow Direction



NOTES:

- 1) Property boundaries obtained from Monroe County GIS 2019 and considered approximate.
- 2) 2021 aerial image from Pictometry International and may not represent current conditions.
- 3) All groundwater monitoring well locations were surveyed by Costich Engineering, DPC, on August 3, 2023.
- 4) Groundwater contours were created in Surfer 23.2.176 via the Kriging method from static water levels measured on August 3, 2023. These contours are shown to illustrate general groundwater patterns in the context of this report. The contour lines are approximate and actual contours may vary from the locations shown. This data should be considered accurate to the degree implied by the method used. Monitoring wells utilized to generate contours are noted with elevation data.

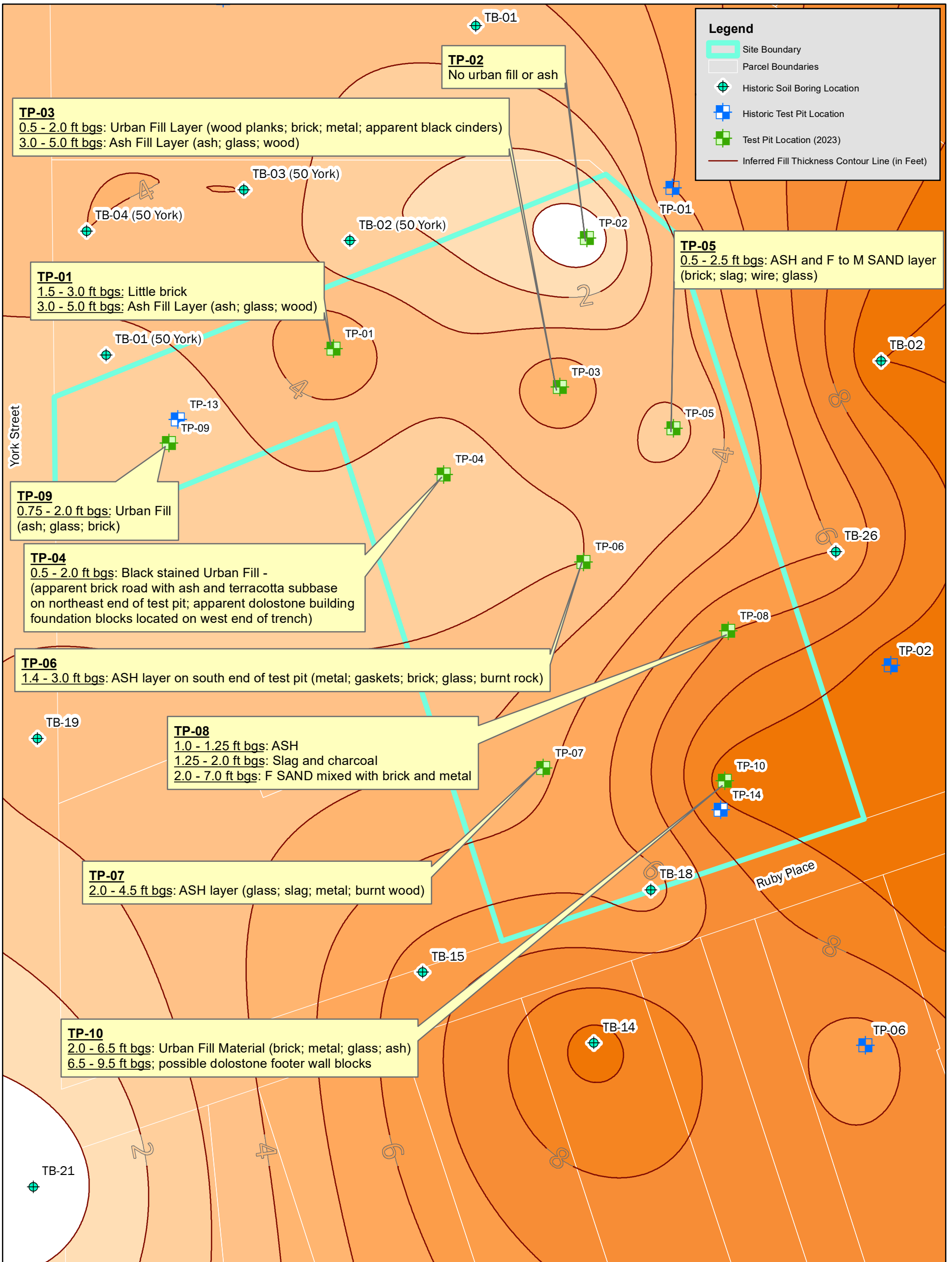
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2230119
FIGURE 4

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 ROCHESTER, NEW YORK**
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 ELEVATION MAP**

CLIENT:
CITY OF ROCHESTER

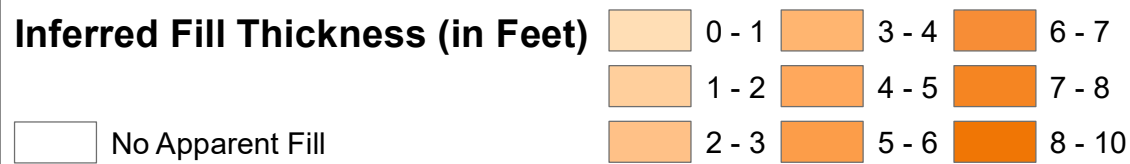
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NOTES:

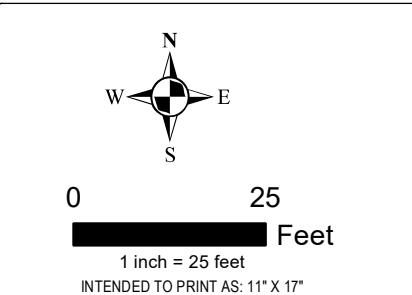
- 1) Property boundaries obtained from Monroe County GIS 2019 and considered approximate.
- 2) Fill depth contours were created in Surfer 23.2.176 via the Kriging method from depths recorded at the bottom of the urban fill layer measured in the test pits and surrounding borings. These contours are shown to illustrate general urban fill depth patterns in the context of this report. The contour lines are approximate and actual contours may vary from the locations shown. This data should be considered accurate to the degree implied by the method used.
- 3) Refer to soil boring logs for additional information.



PROJECT/DRAWING NUMBER:
[2230119]
[FIGURE 6]

PROJECT:
**PHASE II ESA
42 YORK STREET
ROCHESTER, NEW YORK**
DRAWING NAME:
**FILL LOCATION
AND DEPTHS**

CLIENT:
CITY OF ROCHESTER





TABLES

Table 1
Phase II Environmental Site Assessment
42 York Street, Rochester, New York 14614
Sample Log
LaBella Project # 2230119



Sample Location	Sample Depth (ft)	Sample Purpose	Date Sampled	PID Reading (ppm)	Staining / Odors	Material Description / Sample Observation	Lab Report Number	Laboratory Analysis																
								TCL & CP-51 VOCs	TCL & CP-51 SVOCs	TAL Metals	TCLP VOCs	TCLP SVOCs	TCLP Metals	PCBs	Reactivity	Corrosivity (pH)	Ignitability							
Test Pit Study																								
TP-02	3-4 ft	Investigation	7/11/2023	0	No	Light tan-brown F SAND ; some silt ; little SR gravel ; massive ; moist	L2339907	X	X	X														
TP-03	0.5-2 ft	Investigation	7/11/2023	0	No	Dark brown F SAND ; some silt ; little SR gravel ; little urban fill (wood planks ; brick chunks ; metal chunks and wire ; black apparent cinders) ; moist		X	X	X														
TP-04	0.5-2 ft	Investigation	7/12/2023	0	Yes	Black stained URBAN FILL MATERIAL (apparent brick road with ash and terracotta subbase on northeast end of test pit beneath asphalt subbase gravel; apparent dolostone building foundation blocks located on west end of trench)		X	X	X														
TP-05	0.5-2.5 ft	Investigation	7/12/2023	0	No	URBAN FILL - ASH intermixed with dark brown F to M SAND, AND SILT ; little brick ; little metal slag and wire ; little SA to SR gravel ; trace glass ; moist		X	X	X														
TP-06	0.4-3 ft	Investigation	7/12/2023	0	No	ASH layer located on south end consisting of ash, metal, engine gaskets, slag (copper, unknown material, and bluish metal), glass, burnt rock, and brick chunks		X	X	X														
TP-07	2.0-4.5 ft	Investigation	7/12/2023	0	No	ASH FILL including glass, slag, metal, burnt wood debris		X	X	X														
TP-02 (Blind Dup)	3-4 ft	Investigation	7/11/2023	0	No	Light tan-brown F SAND ; some silt ; little SR gravel ; massive ; moist		X	X	X														
TP-01/WC-01	0.25-5 ft	Waste Characterization	7/11/2023	0	No	Brown reworked F to M SAND ; little brick ; little cobble ; little silt ; moist to wet ASH FILL LAYER - ash ; glass ; wood ; moist	L2339895				X	X	X	X	X	X	X	X	X					
TP-09/WC-02	0.75-2 ft	Waste Characterization	7/11/2023	0	No	URBAN FILL MATERIAL (ASH, glass, F to M brick chunks) ; some C sand ; moist to dry Brown F SAND ; some silt ; little SA gravel ; trace C sand ; moist ; Not native					X	X	X	X	X	X	X	X	X					
TP-08/WC-03	1-6 ft	Waste Characterization	7/12/2023	0	No	ASH AND F brown SAND ; some silt ; dry SAND ; some silt ; some metal slag and charcoal ; dry					X	X	X	X	X	X	X	X	X					
TP-10/WC-04	3.5-6.5 ft	Waste Characterization	7/12/2023	0	No	Fill material - Dark brown F to M SAND ; little silt ; little fill material (brick chunks, metal, glass, and ash) ; moist to dry reworked brown F SAND ; some silt ; some dolostone footer wall blocks ; little SR gravel ; moist					X	X	X	X	X	X	X	X	X					
TP-02/WC-05	4-5 ft	Waste Characterization	7/11/2023	0	No	Brown F to M SAND ; little C SA sand ; little SR gravel ; little cobble ; trace silt ; moist to wet ; massive					X	X	X	X	X	X	X	X	X					
TP-06/WC-08	0.4-3 ft	Waste Characterization	7/12/2023	0	No	Dark and light brown F to M possible bedding SAND ; little silt ; little SR gravel ; moist ASH layer located on south end consisting of ash, metal, engine gaskets, slag (copper, unknown material, and bluish metal), glass, burnt rock, and brick chunks					X	X	X	X	X	X	X	X	X					
Groundwater Sampling Event																								
YS-MW-2023-01	7 ft	GW Sample	7/26/2023	-	-	Orange Discoloration	L2343170	X	X	X														
MW-01-072623	7 ft	GW Sample	7/26/2023	-	-	Clear water		X	X	X														
YS-MW-BD-072623	7 ft	GW Sample	7/26/2023	-	-	Parent Well: YS-MW-2023-01		X	X	X														

- Notes
1. TCL & CP-51 VOCs analyzed using USEPA Method 8260D
 2. TCL & CP-51 SVOCs analyzed using USEPA Method 8270E
 3. TAL Metals analyzed using USEPA Method 6010D/7471B
 4. Toxicity Characteristics Leachate Procedure (TCLP) VOCs analyzed using USEPA Method 8260D/1311
 5. TCLP SVOCs analyzed using USEPA Method 8270E/1311
 6. TCLP Metals analyzed using USEPA Method 6010D/7470A/1311
 7. Polychlorinate Biphenyls (PCBs) analyzed using USEPA Method 8082A
 8. Reactive Cyanide analyzed using USEPA Method 9010C
 9. Reactive Sulfide analyzed using Chp. 7 SW-846
 10. Ignitability analyzed using USEPA Method 1030
 11. Corrosivity (pH) analyzed using USEPA Method 9045D

Table 2
Phase II Environmental Site Assessment
42 York Street, Rochester New York 14614
Groundwater Elevation Data for August 3, 2023
LaBella Project # 2230119



Well ID	Parcel Location	Ground Elevation (ft)	Elevation of PVC Well Casing (ft)	SWL ³	Groundwater Elevation (ft)	Depth of Well ³
YS-MW-2023-01	42 York St	535.09	534.74	5.54	529.20	10.60
MW-01	42 York St	534.70	534.30	5.05	529.25	10.39
YS-MW-2022-01	24 York St	535.46	535.17	5.83	529.34	9.10
YS-MW-2022-02	24 York St	535.21	534.90	5.72	529.18	9.13
YS-MW-2022-03	24 York St	534.45	534.02	5.11	528.91	9.18
YS-MW-2022-04	24 York St	534.86	534.29	5.02	529.27	10.60
MW-03	906-910 West Main St	542.07	541.71	7.40	534.31	13.94
MW-07	ROW 24 York St	535.71	535.09	6.35	528.74	10.52
MW-08	Ruby Pl	534.82	534.60	5.63	528.97	15.26
MWYW-A	4-12 West Ave	539.69	539.44	9.73	529.71	16.02
MWYW-B	4-12 West Ave	536.28	536.07	7.05	529.02	28.40

Notes

1. Survey completed by Costich Engineering, DPC, on August 3, 2023
2. Datum used during survey was: NAVD88
3. Static groundwater levels were collected from marked location at top of casing. In the absence of any mark, data collected from highest point of casing.

Table 3A (1 of 2)
Phase II Environmental Site Assessment
42 York Street, Rochester, New York 14614
Summary of Volatile Organic Compounds in Soil
LaBella Project # 2230119



Sample Location	Sample ID	NYCRR Part 375 Unrestricted Use SCOs	NYCRR Part 375 Restricted Residential Use SCOs	NYCRR Part 375 Commercial Use SCOs	TP-02		TP-03	TP-04
					TP-02-3-4FT	BD-01-3-4 FT	TP-03-0.5-2 FT	TP-04-0.5-2.5 FT
Laboratory ID					L2339907-02	L2339907-09	L2339907-03	L2339907-04
Sample Depth (ft bgs)					3-4	3-4	0.5-2	0.5-2.5
Sample Date					7/11/2023	7/11/2023	7/11/2023	7/12/2023
VOCs								
1,1,1-Trichloroethane	0.68	100	500	<0.00016	<0.00014	<0.00014	<0.00018	<0.00026
1,1,2,2-Tetrachloroethane	NL	NL	NL	<0.00016	<0.00014	<0.00014	<0.00017	<0.00026
1,1,2-Trichloroethane	NL	NL	NL	<0.00025	<0.00022	<0.00022	<0.00028	<0.00042
1,1-Dichloroethane	0.27	26	240	<0.00014	<0.00012	<0.00012	<0.00015	<0.00023
1,1-Dichloroethene	0.33	100	500	<0.00023	<0.00023	<0.00023	<0.00025	<0.00038
1,2,4-Trichlorobenzene	NL	NL	NL	<0.00026	<0.00022	<0.00022	<0.00028	<0.00043
1,2,4-Trimethylbenzene	3.6	52	190	<0.00032	<0.00028	<0.00028	<0.00035	<0.00053
1,2-Dibromo-3-chloropropane	NL	NL	NL	<0.00095 UJ	<0.00082 UJ	<0.00082 UJ	<0.001 UJ	<0.0016 UJ
1,2-Dibromoethane	NL	NL	NL	<0.00026	<0.00023	<0.00023	<0.00029	<0.00044
1,2-Dichlorobenzene	1.1	100	500	<0.00014	<0.00012	<0.00012	<0.00015	<0.00023
1,2-Dichloroethane	0.02	3.1	30	<0.00024	<0.00021	<0.00021	<0.00027	<0.00041
1,2-Dichloropropane	NL	NL	NL	<0.00012	<0.0001	<0.0001	<0.00013	<0.0002
1,3,5-Trimethylbenzene	8.4	52	190	<0.00018	<0.00016	<0.00016	<0.0002	<0.00031
1,3-Dichlorobenzene	2.4	49	280	<0.00014	<0.00012	<0.00012	<0.00016	<0.00023
1,4-Dichlorobenzene	1.8	13	130	<0.00016	<0.00014	<0.00014	<0.00018	<0.00027
2-Butanone	0.12	100	500	<0.0021 UJ	<0.0018 UJ	<0.0018 UJ	<0.0023 UJ	<0.0035 UJ
2-Hexanone	NL	NL	NL	<0.0011	<0.00097	<0.00097	<0.0012	<0.0019
4-Methyl-2-pentanone	NL	NL	NL	<0.0012 UJ	<0.001 UJ	<0.001 UJ	<0.0013 UJ	<0.002 UJ
Acetone	0.05	100	500	<0.0046	<0.004	<0.004	<0.005	<0.0076
Benzene	0.06	4.8	44	<0.00016	<0.00014	<0.00014	<0.00017	<0.00026
Bromodichloromethane	NL	NL	NL	<0.0001	<0.00009	<0.00009	<0.00011	<0.00017
Bromoform	NL	NL	NL	<0.00023	<0.0002	<0.0002	<0.00026	<0.00039
Bromomethane	NL	NL	NL	<0.00055 UJ	<0.00048 UJ	<0.00048 UJ	<0.00061 UJ	<0.00092 UJ
Carbon disulfide	NL	NL	NL	<0.0043	<0.0038	<0.0038	<0.0048	<0.0072
Carbon tetrachloride	0.76	2.4	22	<0.00022	<0.00019	<0.00019	<0.00024	<0.00036
Chlorobenzene	1.1	100	500	<0.00012	<0.0001	<0.0001	<0.00013	<0.0002
Chloroethane	NL	NL	NL	<0.00043	<0.00037	<0.00037	<0.00047	<0.00072
Chloroform	0.37	49	350	<0.00013	<0.00012	<0.00012	<0.00015	<0.00022
Chloromethane	NL	NL	NL	<0.00089 UJ	<0.00077 UJ	<0.00077 UJ	<0.00098 UJ	<0.0015 UJ
cis-1,2-Dichloroethene	0.25	100	500	<0.00017	<0.00014	<0.00014	<0.00018	<0.00028
cis-1,3-Dichloropropene	NL	NL	NL	<0.00015	<0.00013	<0.00013	<0.00016	<0.00025
Cyclohexane	NL	NL	NL	<0.00052	<0.00045	<0.00045	<0.00057	<0.00086
Dibromochloromethane	NL	NL	NL	<0.00013	<0.00012	<0.00012	<0.00015	<0.00022
Dichlorodifluoromethane	NL	NL	NL	<0.00087 UJ	<0.00075 UJ	<0.00075 UJ	<0.00096 UJ	<0.0014 UJ
Ethylbenzene	1	41	390	<0.00013	<0.00012	<0.00012	<0.00015	<0.00022
Freon-113	NL	NL	NL	<0.00066	<0.00057	<0.00057	<0.00073	<0.0011
Isopropylbenzene	NL	NL	NL	<0.0001	<0.00009	<0.00009	<0.00011	<0.00017
Methyl Acetate	NL	NL	NL	<0.0009	<0.00078	<0.00078	<0.001	<0.0015
Methyl cyclohexane	NL	NL	NL	<0.00057	<0.0005	<0.0005	<0.00063	<0.00096
Methyl tert butyl ether	0.93	100	500	<0.00019	<0.00016	<0.00016	<0.00021	<0.00032
Methylene chloride	0.05	100	500	<0.0022	<0.0019	<0.0019	<0.0024	<0.0036
Naphthalene	12	100	500	<0.00062	<0.00054	<0.00054	<0.00068	<0.001
n-Butylbenzene	12	100	500	<0.00016	<0.00014	<0.00014	<0.00018	<0.00026
n-Propylbenzene	3.9	100	500	<0.00016	<0.00014	<0.00014	<0.00018	<0.00027
o-Xylene	NL	NL	NL	<0.00028	<0.00024	<0.00024	<0.0003	<0.00046
p/m-Xylene	NL	NL	NL	<0.00053	<0.00046	<0.00046	<0.00059	<0.00089
p-Isopropyltoluene	NL	NL	NL	<0.0001	<0.00009	<0.00009	<0.00011	<0.00017
sec-Butylbenzene	11	100	500	<0.00014	<0.00012	<0.00012	<0.00015	<0.00023
Styrene	NL	NL	NL	<0.00019	<0.00016	<0.00016	<0.0002	<0.00031
tert-Butylbenzene	5.9	100	500	<0.00011	<0.0001	<0.0001	<0.00012	<0.00019
Tetrachloroethene	1.3	19	150	<0.00019	<0.00016	<0.00016	<0.0002	<0.00031
Toluene	0.7	100	500	<0.00052	<0.00045	<0.00045	<0.00057	<0.00086
trans-1,2-Dichloroethene	0.19	100	500	<0.00013	<0.00011	<0.00011	<0.00014	<0.00022
trans-1,3-Dichloropropene	NL	NL	NL	<0.00026	<0.00022	<0.00022	<0.00029	<0.00043
Trichloroethene	0.47	21	200	<0.00013	<0.00011	<0.00011	<0.00014	<0.00022
Trichlorofluoromethane	NL	NL	NL	<0.00066	<0.00057	<0.00057	<0.00073	<0.0011
Vinyl chloride	0.02	0.9	13	<0.00032	<0.00028	<0.00028	<0.00035	<0.00053
Total VOCs				-	-	-	-	-
Total TIC Compounds	NL	NL	NL	0.00496 J	0.0033 J	0.0276 J		

NOTES:

All values displayed in milligrams per kilograms (mg/kg), equal to parts per million (ppm)
* < - Indicates compound was not detected above the indicated laboratory method detection limit (MDL).
Bold font indicates the concentration exceeds the MDL.

VOCs analyzed by USEPA Method 8260D
NL indicates Not Listed
J indicates an estimated value (above the MDL but below the laboratory method reportable limit (RL))
TICs - Tentatively Identified Compounds
Data has been validated
Blue font represents a change made in the DUSR
UJ indicates nondetect with estimated quantitation limits

Table 3A (2 of 2)
Phase II Environmental Site Assessment
42 York Street, Rochester, New York 14614
Summary of Volatile Organic Compounds in Soil
LaBella Project # 2230119



Sample Location	Sample ID	NYCRR Part 375 Unrestricted Use SCOs	NYCRR Part 375 Restricted Residential Use SCOs	NYCRR Part 375 Commercial Use SCOs	TP-05	TP-06	TP-07
					TP-05-0.5-2.5 FT	TP-06-0.4-3 FT	TP-07-2.0-4.5 FT
Laboratory ID					L2339907-05	L2339907-06	L2339907-07
Sample Depth (ft bgs)					0.5-2.5	0.4-3	2-4.5
Sample Date					7/12/2023	7/12/2023	7/11/2023
VOCs							
1,1,1-Trichloroethane	0.68	100	500	<0.0002	<0.00023	<0.00025	
1,1,2,2-Tetrachloroethane	NL	NL	NL	<0.0002	<0.00023	<0.00025	
1,1,2-Trichloroethane	NL	NL	NL	<0.00032	<0.00036	<0.0004	
1,1-Dichloroethane	0.27	26	240	<0.00017	<0.0002	<0.00022	
1,1-Dichloroethene	0.33	100	500	<0.00028	<0.00032	<0.00035	
1,2,4-Trichlorobenzene	NL	NL	NL	<0.00033	<0.00037	<0.0004	
1,2,4-Trimethylbenzene	3.6	52	190	<0.0004	<0.00045	<0.0005	
1,2-Dibromo-3-chloropropane	NL	NL	NL	<0.0012 UJ	<0.0014 UJ	<0.0015 UJ	
1,2-Dibromoethane	NL	NL	NL	<0.00033	<0.00038	<0.00042	
1,2-Dichlorobenzene	1.1	100	500	<0.00017	<0.0002	<0.00021	
1,2-Dichloroethane	0.02	3.1	30	<0.00031	<0.00035	<0.00038	
1,2-Dichloropropane	NL	NL	NL	<0.00015	<0.00017	<0.00019	
1,3,5-Trimethylbenzene	8.4	52	190	<0.00023	<0.00026	<0.00029	
1,3-Dichlorobenzene	2.4	49	280	<0.00018	<0.0002	<0.00022	
1,4-Dichlorobenzene	1.8	13	130	<0.0002	<0.00023	<0.00025	
2-Butanone	0.12	100	500	<0.0027 UJ	<0.003 UJ	<0.0033 UJ	
2-Hexanone	NL	NL	NL	<0.0014	<0.0016	<0.0018	
4-Methyl-2-pentanone	NL	NL	NL	<0.0015 UJ	<0.0017 UJ	<0.0019 UJ	
Acetone	0.05	100	500	<0.0058	<0.0066	<0.0072	
Benzene	0.06	4.8	44	<0.0002	<0.00023	<0.00025	
Bromodichloromethane	NL	NL	NL	<0.00013	<0.00015	<0.00016	
Bromoform	NL	NL	NL	<0.00029	<0.00034	<0.00037	
Bromomethane	NL	NL	NL	<0.0007 UJ	<0.00079 UJ	<0.00086 UJ	
Carbon disulfide	NL	NL	NL	<0.0054	<0.0062	<0.0068	
Carbon tetrachloride	0.76	2.4	22	<0.00028	<0.00031	<0.00034	
Chlorobenzene	1.1	100	500	<0.00015	<0.00017	<0.00019	
Chloroethane	NL	NL	NL	<0.00054	<0.00062	<0.00067	
Chloroform	0.37	49	350	<0.00017	<0.00019	<0.00021	
Chloromethane	NL	NL	NL	<0.0011 UJ	<0.0013 UJ	<0.0014 UJ	
cis-1,2-Dichloroethene	0.25	100	500	<0.00021	<0.00024	<0.00026	
cis-1,3-Dichloropropene	NL	NL	NL	<0.00019	<0.00022	<0.00024	
Cyclohexane	NL	NL	NL	<0.00065	<0.00074	<0.00081	
Dibromochloromethane	NL	NL	NL	<0.00017	<0.00019	<0.00021	
Dichlorodifluoromethane	NL	NL	NL	<0.0011 UJ	<0.0012 UJ	<0.0014 UJ	
Ethylbenzene	1	41	390	<0.00017	<0.00019	<0.00021	
Freon-113	NL	NL	NL	<0.00083	<0.00094	<0.001	
Isopropylbenzene	NL	NL	NL	<0.00013	<0.00015	<0.00016	
Methyl Acetate	NL	NL	NL	<0.0011	<0.0013	<0.0014	
Methyl cyclohexane	NL	NL	NL	<0.00072	<0.00082	<0.0009	
Methyl tert butyl ether	0.93	100	500	<0.00024	<0.00027	<0.0003	
Methylene chloride	0.05	100	500	<0.0027	<0.0031	<0.0034	
Naphthalene	12	100	500	<0.00078	<0.00088	<0.00097	
n-Butylbenzene	12	100	500	<0.0002	<0.00023	<0.00025	
n-Propylbenzene	3.9	100	500	<0.0002	<0.00023	<0.00025	
o-Xylene	NL	NL	NL	<0.00035	<0.0004	<0.00043	
p/m-Xylene	NL	NL	NL	<0.00067	<0.00076	<0.00083	
p-Isopropyltoluene	NL	NL	NL	<0.00013	<0.00015	<0.00016	
sec-Butylbenzene	11	100	500	<0.00018	<0.0002	<0.00022	
Styrene	NL	NL	NL	<0.00023	<0.00027	<0.00029	
tert-Butylbenzene	5.9	100	500	<0.00014	<0.00016	<0.00018	
Tetrachloroethene	1.3	19	150	<0.00023	<0.00027	<0.00029	
Toluene	0.7	100	500	<0.00065	<0.00074	<0.00081	
trans-1,2-Dichloroethene	0.19	100	500	<0.00016	<0.00019	<0.0002	
trans-1,3-Dichloropropene	NL	NL	NL	<0.00033	<0.00037	<0.00041	
Trichloroethene	0.47	21	200	<0.00016	<0.00019	<0.0002	
Trichlorofluoromethane	NL	NL	NL	<0.00083	<0.00095	<0.001	
Vinyl chloride	0.02	0.9	13	<0.0004	<0.00046	<0.0005	
Total VOCs				-	-	-	
Total TIC Compounds	NL	NL	NL	0.00499 J	0.0064 J	-	

NOTES:

- All values displayed in milligrams per kilograms (mg/kg), equal to parts per million (ppm)
- *< - Indicates compound was not detected above the indicated laboratory method detection limit (MDL).
- Bold font indicates the concentration exceeds the MDL.**
- VOCs analyzed by USEPA Method 8260D
- NL indicates Not Listed
- J indicates an estimated value (above the MDL but below the laboratory method reportable limit (RL))
- TICs - Tentatively Identified Compounds
- Data has been validated
- Blue font represents a change made in the DUSR
- UJ indicates nondetect with estimated quantitation limits

Table 3B (1 of 1)
Phase II Environmental Site Assessment
42 York Street, Rochester, New York 14614
Summary of Semi-Volatile Organic Compounds in Soil
LaBella Project # 2230119



Sample Location	NYCRR Part 375 Unrestricted Use SCOs	NYCRR Part 375 Restricted Residential Use SCOs	NYCRR Part 375 Commercial Use SCOs	TP-02		TP-03	TP-04	TP-05	TP-06	TP-07
				TP-02-3-4FT	BD-01-3-4 FT	TP-03-0.5-2 FT	TP-04-0.5-2.5 FT	TP-05-0.5-2.5 FT	TP-06-0.4-3 FT	TP-07-2.0-4.5 FT
				L2339907-02	L2339907-09	L2339907-03	L2339907-04	L2339907-05	L2339907-06	L2339907-07
				3-4	3-4	0.5-2	0.5-2.5	0.5-2.5	0.4-3	2-4.5
Laboratory ID										
Sample Depth (ft bgs)										
Sample Date				7/11/2023	7/11/2023	7/11/2023	7/12/2023	7/12/2023	7/12/2023	7/11/2023
SVOCs										
1,2,4,5-Tetrachlorobenzene	NL	NL	NL	<0.019	<0.019	<0.021	<0.021	<0.021	<0.083	<0.06
2,3,4,6-Tetrachlorophenol	NL	NL	NL	<0.037	<0.037	<0.04	<0.041	<0.041	<0.16	<0.12
2,4,5-Trichlorophenol	NL	NL	NL	<0.035	<0.035	<0.038	<0.039	<0.039	<0.15	<0.11
2,4,6-Trichlorophenol	NL	NL	NL	<0.035	<0.035	<0.038	<0.039	<0.038	<0.15	<0.11
2,4-Dichlorophenol	NL	NL	NL	<0.03	<0.029	<0.032	<0.033	<0.032	<0.13	<0.092
2,4-Dimethylphenol	NL	NL	NL	<0.061	<0.06	<0.066	<0.068	<0.067	<0.26	<0.19
2,4-Dinitrophenol	NL	NL	NL	<0.086	<0.085	<0.093	<0.096	<0.094	<0.37	<0.27
2,4-Dinitrotoluene	NL	NL	NL	<0.037	<0.037	<0.04	<0.041	<0.04	<0.16	<0.11
2,6-Dinitrotoluene	NL	NL	NL	<0.032	<0.031	<0.034	<0.035	<0.035	<0.14	<0.098
2-Chloronaphthalene	NL	NL	NL	<0.018	<0.018	<0.02	<0.02	<0.02	<0.079	<0.057
2-Chlorophenol	NL	NL	NL	<0.022	<0.022	<0.024	<0.024	<0.024	<0.094	<0.068
2-Methylnaphthalene	NL	NL	NL	<0.022	<0.022	<0.024	<0.025	0.043	<0.096	<0.069
2-Methylphenol	0.33	100	500	<0.028	<0.028	<0.031	<0.032	<0.031	<0.12	<0.089
2-Nitroaniline	NL	NL	NL	<0.036	<0.035	<0.038	<0.04	<0.039	<0.15	<0.11
2-Nitrophenol	NL	NL	NL	<0.069	<0.069	<0.075	<0.077	<0.076	<0.3	<0.22
3,3'-Dichlorobenzidine	NL	NL	NL	<0.049	<0.049	<0.053	<0.055	<0.054	<0.21	<0.15
3-Methylphenol/4-Methylphenol	0.33	100	500	<0.029	<0.029	<0.031	<0.032	<0.032	<0.12	<0.09
3-Nitroaniline	NL	NL	NL	<0.035	<0.034	<0.038	<0.039	<0.038	<0.15	<0.11
4,6-Dinitro-p-cresol	NL	NL	NL	<0.088	<0.088	<0.096	<0.098	<0.097	<0.38	<0.27
4-Bromophenyl phenyl ether	NL	NL	NL	<0.028	<0.028	<0.03	<0.031	<0.031	<0.12	<0.087
4-Chloroaniline	NL	NL	NL	<0.034	<0.033	<0.036	<0.037	<0.037	<0.14	<0.1
4-Chlorophenyl phenyl ether	NL	NL	NL	<0.02	<0.02	<0.022	<0.022	<0.022	<0.085	<0.061
4-Nitroaniline	NL	NL	NL	<0.076	<0.076	<0.082	<0.085	<0.084	<0.33	<0.24
4-Nitrophenol	NL	NL	NL	<0.075	<0.075	<0.081	<0.084	<0.083	<0.32	<0.23
Acenaphthene	20	100	500	<0.019	<0.019	<0.021	<0.021	<0.021	<0.082	<0.059
Acenaphthylene	100	100	500	<0.028	<0.028	<0.031	<0.032	<0.031	<0.12	<0.088
Acetophenone	NL	NL	NL	<0.023	<0.023	<0.025	<0.025	<0.025	<0.098	<0.071
Anthracene	100	100	500	<0.036	<0.036	<0.039	<0.04	0.09	<0.15	<0.11
Atrazine	NL	NL	NL	<0.064	<0.064	<0.07	<0.072	<0.071	<0.28	<0.2
Benzaldehyde	NL	NL	NL	<0.05	<0.049	<0.054	<0.055	<0.055	<0.21	<0.15
Benzo(a)anthracene	1	1	5.6	<0.021	<0.021	0.16	<0.023	0.52	<0.089	<0.064
Benzo(a)pyrene	1	1	1	<0.045	<0.045	0.24	<0.05	0.59	<0.19	<0.14
Benzo(b)fluoranthene	1	1	5.6	<0.031	<0.031	0.31	<0.034	0.67	<0.13	<0.096
Benzo(g,h)perylene	100	100	500	<0.022	<0.022	0.16	<0.024	0.36	<0.093	<0.067
Benzo(k)fluoranthene	0.8	3.9	56	<0.029	<0.029	0.095	<0.033	0.21	<0.13	<0.092
Biphenyl	NL	NL	NL	<0.024	<0.024	<0.026	<0.027	<0.026	<0.1	<0.074
Bis(2-chloroethoxy)methane	NL	NL	NL	<0.018	<0.018	<0.02	<0.02	<0.02	<0.079	<0.057
Bis(2-chloroethoxy)ether	NL	NL	NL	<0.025	<0.025	<0.027	<0.028	<0.027	<0.11	<0.078
Bis(2-chloroisopropyl)ether	NL	NL	NL	<0.031	<0.031	<0.034	<0.035	<0.034	<0.14	<0.098
Bis(2-ethylhexyl)phthalate	NL	NL	NL	<0.064	<0.063	<0.069	<0.071	<0.07	<0.27	<0.2
Butyl benzy phthalate	NL	NL	NL	<0.046	<0.046	<0.05	<0.052	<0.051	<0.2	<0.14
Caprolactam	NL	NL	NL	<0.056	<0.056	<0.06	<0.062	<0.062	<0.24	<0.17
Carbazole	NL	NL	NL	<0.018	<0.018	<0.019	<0.02	<0.02	<0.077	<0.056
Chrysene	1	3.9	56	<0.019	<0.019	0.18	<0.021	0.48	<0.082	<0.06
Dibenzo(a,h)anthracene	0.33	0.33	0.56	<0.021	<0.021	0.046	<0.024	0.073	<0.092	<0.066
Dibenzofuran	7	59	350	<0.017	<0.017	<0.019	<0.019	0.03	<0.075	<0.054
Diethyl phthalate	NL	NL	NL	<0.017	<0.017	<0.018	<0.019	<0.019	<0.073	<0.053
Dimethyl phthalate	NL	NL	NL	<0.039	<0.038	<0.042	<0.043	<0.042	<0.17	<0.12
Di-n-butylphthalate	NL	NL	NL	<0.035	<0.035	<0.038	<0.039	<0.038	<0.15	<0.11
Di-n-octylphthalate	NL	NL	NL	<0.063	<0.062	<0.068	<0.07	<0.069	<0.27	<0.19
Fluoranthene	100	100	500	<0.021	<0.021	0.19	<0.024	1	<0.091	<0.066
Fluorene	30	100	500	<0.018	<0.018	<0.019	<0.02	<0.02	<0.077	<0.056
Hexachlorobenzene	0.33	1.2	6	<0.021	<0.02	<0.022	<0.023	<0.023	<0.089	<0.064
Hexachlorobutadiene	NL	NL	NL	<0.027	<0.027	<0.029	<0.03	<0.03	<0.12	<0.084
Hexachlorocyclopentadiene	NL	NL	NL	<0.17	<0.16	<0.18	<0.18	<0.18	<0.72	<0.52
Hexachloroethane	NL	NL	NL	<0.03	<0.03	<0.032	<0.033	<0.033	<0.13	<0.093
Indeno(1,2,3-cd)pyrene	0.5	0.5	5.6	<0.026	<0.026	0.19	<0.029	0.44	<0.11	<0.08
Isophorone	NL	NL	NL	<0.024	<0.024	<0.026	<0.027	<0.026	<0.1	<0.074
Naphthalene	12	100	500	<0.022	<0.022	0.027	<0.025	0.059	<0.097	<0.07
NDPA/DPA	NL	NL	NL	<0.021	<0.021	<0.023	<0.023	<0.023	<0.09	<0.065
Nitrobenzene	NL	NL	NL	<0.027	<0.027	<0.029	<0.03	<0.03	<0.12	<0.085
n-Nitrosodi-n-propylamine	NL	NL	NL	<0.028	<0.028	<0.031	<0.032	<0.031	<0.12	<0.088
p-Chloro-m-cresol	NL	NL	NL	<0.027	<0.027	<0.03	<0.03	<0.03	<0.12	<0.085
Pentachlorophenol	0.8	6.7	6.7	<0.04	<0.04	<0.044	<0.045	<0.044	<0.17	<0.12
Phenanthrene	100	100	500	<0.022	<0.022	0.066	<0.025	0.3	<0.096	<0.07
Phenol	0.33	100	500	<0.028	<0.028	<0.03	<0.031	<0.03	<0.12	<0.086
Pyrene	100	100	500	<0.018	<0.018	0.18	<0.02	0.91	<0.079	<0.057
Total SVOCs				-	-	1,844	-	5,775	-	-
Total TIC Compounds	NL	NL	NL	-	-	-	-	0.863	J	-

NOTES:
All values displayed in milligrams per kilograms (mg/kg), equal to parts per million (ppm)
*- Indicates compound was not detected above the indicated laboratory method detection limit (MDL).
Bold font indicates the concentration exceeds the method detection limit (MDL).
SVOCs analyzed by USEPA Method 8270E
TICs stands for Tentatively Identified Compounds
NL indicates Not Listed
J indicates an estimated value (above the MDL but below the laboratory method reportable limit (RL))
Data has been validated
Blue font represents a change made in the DUSR
UJ indicates nondetect with estimated quantitation limits

Table 3C (1 of 1)
Phase II Environmental Site Assessment
42 York Street, Rochester, New York 14614
Summary of Metals in Soil
LaBella Project # 2230119



Sample Location	Sample ID	NYCRR Part 375 Unrestricted Use SCOs	NYCRR Part 375 Restricted Residential Use SCOs	NYCRR Part 375 Commercial Use SCOs	TP-02		TP-03	TP-04	TP-05	TP-06	TP-07
					TP-02-3-4 FT	BD-01-3-4 FT	TP-03-0.5-2 FT	TP-04-0.5-2.5 FT	TP-05-0.5-2.5 FT	TP-06-0.4-3 FT	TP-07-2-4.5 FT
Laboratory ID					L2339907-02	L2339907-09	L2339907-03	L2339907-04	L2339907-05	L2339907-06	L2339907-07
Sample Depth (ft bgs)					3-4	3-4	0.5-2	0.5-2.5	0.5-2.5	0.4-3	2-4.5
Sample Date					7/11/2023	7/11/2023	7/11/2023	7/12/2023	7/12/2023	7/12/2023	7/11/2023
Metals											
Aluminum, Total	NL	NL	NL	4380	4710	6410	5350	5590	5690	4530	
Antimony, Total	NL	NL	NL	0.611 J	0.484 J	1.42 J	0.698 J	1.2 J	0.682 J	<0.347	
Arsenic, Total	13	16	16	2.9	2.07	6.53	7.92	9.3	6.45	2.82	
Barium, Total	350	400	400	30.5	35.3	128	117	103	69.1	75.4	
Beryllium, Total	7.2	72	590	0.336 J	0.341 J	0.506	0.549	0.543	1.18	0.492	
Cadmium, Total	2.5	4.3	9.3	<0.085	<0.084	1.91	0.53 J	0.35 J	2.8	<0.09	
Calcium, Total	NL	NL	NL	2280	2260	15000	10000	16200	4190	10900	
Chromium, Total	1 / 30	110 / 180	400 / 1500	6.76	6.95	10.7	9.55	8.78	7.74	5.52	
Cobalt, Total	NL	NL	NL	3.41	3.7	4.54	4.52	6.15	5.68	8.32	
Copper, Total	50	270	270	7.86	6.59	94.5	84.5	67.8	241	24.3	
Iron, Total	NL	NL	NL	12000	10900	16400	9950	15100	9290	4080	
Lead, Total	63	400	1,000	17.2	4.34	253	1080	98.4	52.7	324	
Magnesium, Total	NL	NL	NL	1660	1820	5780	3300	7200	1520	3560	
Manganese, Total	1,600	2,000	10,000	359	458	423	265	321	206	134	
Mercury, Total	0.18	0.81	2.8	<0.052	<0.05	0.484	0.358	0.59	<0.084	0.118	
Nickel, Total	30	310	310	7.58	9.16	17	31.5	14.2	51.8	13.7	
Potassium, Total	NL	NL	NL	379	364	548	438	585	408	416	
Selenium, Total	3.9	180	1,500	<0.225	<0.222	0.291 J	0.436 J	<0.243	0.846 J	<0.236	
Silver, Total	2	180	1,500	<0.246	<0.243	<0.263	<0.275	<0.266	<0.358	<0.258	
Sodium, Total	NL	NL	NL	76.9 J	68.4 J	92.3 J	132 J	184 J	174 J	199	
Thallium, Total	NL	NL	NL	<0.274	<0.271	<0.293	<0.306	<0.296	<0.399	<0.288	
Vanadium, Total	NL	NL	NL	13.3	12.4	15.7	16.3	20.7	19.5	16.4	
Zinc, Total	109	10,000	10,000	47	30	1050	248	342	635	52.4	

NOTES:

All values displayed in milligrams per kilograms (mg/kg), equal to parts per million (ppm)

"<" - Indicates compound was not detected above the indicated laboratory method detection limit (MDL).

Bold font indicates the concentration exceeds the method detection limit (MDL).

Underlined font indicates that the compound was detected at a concentration above its respective NYCRR Part 375-6.8(a) Unrestricted Use Soil Cleanup Objective (SCO)

Red font indicates that the compound was detected at a concentration above its respective NYCRR Part 375-6.8(b) Restricted Residential Use SCO

Yellow highlight indicates that the compound was detected at a concentration above its respective NYCRR Part 375-6.8(b) Commercial Use SCO

Metals analyzed by USEPA Method 6010D/7471B

NL indicates Not Listed

J indicates an estimated value (above the MDL but below the laboratory method reportable limit (RL))

Table 4A
Phase II Environmental Site Assessment
42 York Street, Rochester, New York 14614
Summary of Volatile Organic Compounds in Groundwater
LaBella Project # 2230119



Well Location: Sample ID: Laboratory ID	NY-TOGS-GA	MW-01	YS-MW-2023-01		TRIP BLANK
		MW-01 072623 L2343170-02	YS-MW-2023-01 072623 L2343170-01	YS-MW-2023-BD-072623 L2343170-03	
Screen Depth (ft bgs):		3.5 - 10.5	3.0 - 11.0		-
Sample Date:		7/26/2023	7/26/2023	7/26/2023	7/26/2023
VOCs					
1,1,1-Trichloroethane	5	<0.7	<0.7	<0.7	<0.7
1,1,2,2-Tetrachloroethane	5	<0.17	<0.17	<0.17	<0.17
1,1,2-Trichloroethane	1	<0.5	<0.5	<0.5	<0.5
1,1-Dichloroethane	5	<0.7	<0.7	<0.7	<0.7
1,1-Dichloroethene	5	<0.17	<0.17	<0.17	<0.17
1,2,4-Trichlorobenzene	5	<0.7	<0.7	<0.7	<0.7
1,2,4-Trimethylbenzene	5	<0.7	<0.7	<0.7	<0.7
1,2-Dibromo-3-chloropropane	0.04	<0.7 UJ	<0.7 UJ	<0.7 UJ	<0.7 UJ
1,2-Dibromoethane	0.0006	<0.65	<0.65	<0.65	<0.65
1,2-Dichlorobenzene	3	<0.7	<0.7	<0.7	<0.7
1,2-Dichloroethane	0.6	<0.13	<0.13	<0.13	<0.13
1,2-Dichloropropane	1	<0.14	<0.14	<0.14	<0.14
1,3,5-Trimethylbenzene	5	<0.7	<0.7	<0.7	<0.7
1,3-Dichlorobenzene	3	<0.7	<0.7	<0.7	<0.7
1,4-Dichlorobenzene	3	<0.7	<0.7	<0.7	<0.7
2-Butanone	50	<1.9	<1.9	<1.9	<1.9
2-Hexanone	50	<1 UJ	<1 UJ	<1 UJ	<1 UJ
4-Methyl-2-pentanone	NL	<1 UJ	<1 UJ	<1 UJ	<1 UJ
Acetone	50	<1.5	<1.5	1.5 J	<1.5
Benzene	1	<0.16	<0.16	<0.16	<0.16
Bromodichloromethane	50	<0.19	<0.19	<0.19	<0.19
Bromoform	50	<0.65	<0.65	<0.65	<0.65
Bromomethane	5	<0.7	<0.7	<0.7	<0.7
Carbon disulfide	60	<1	<1	<1	<1
Carbon tetrachloride	5	<0.13	<0.13	<0.13	<0.13
Chlorobenzene	5	<0.7	<0.7	<0.7	<0.7
Chloroethane	5	<0.7	<0.7	<0.7	<0.7
Chloroform	7	<0.7	<0.7	<0.7	<0.7
Chloromethane	NL	<0.7	<0.7	<0.7	<0.7
cis-1,2-Dichloroethene	5	<0.7	<0.7	<0.7	<0.7
cis-1,3-Dichloropropene	0.4	<0.14	<0.14	<0.14	<0.14
Cyclohexane	NL	<0.27	<0.27	<0.27	<0.27
Dibromochloromethane	50	<0.15	<0.15	<0.15	<0.15
Dichlorodifluoromethane	5	<1	<1	<1	<1
Ethylbenzene	5	<0.7	<0.7	<0.7	<0.7
Freon-113	5	<0.7	<0.7	<0.7	<0.7
Isopropylbenzene	5	<0.7	<0.7	<0.7	<0.7
Methyl Acetate	NL	<0.23	<0.23	<0.23	<0.23
Methyl cyclohexane	NL	<0.4	<0.4	<0.4	<0.4
Methyl tert butyl ether	10	<0.7	<0.7	<0.7	<0.7
Methylene chloride	5	<0.7	<0.7	<0.7	<0.7
n-Butylbenzene	5	<0.7	<0.7	<0.7	<0.7
n-Propylbenzene	5	<0.7	<0.7	<0.7	<0.7
Naphthalene	10	<0.7 UJ	<0.7 UJ	<0.7 UJ	<0.7 UJ
o-Xylene	5	<0.7	<0.7	<0.7	<0.7
p-Isopropyltoluene	5	<0.7	<0.7	<0.7	<0.7
p/m-Xylene	5	<0.7	<0.7	<0.7	<0.7
sec-Butylbenzene	5	<0.7	<0.7	<0.7	<0.7
Styrene	930	<0.7	<0.7	<0.7	<0.7
tert-Butylbenzene	5	<0.7	<0.7	<0.7	<0.7
Tetrachloroethene	5	<0.18	<0.18	<0.18	<0.18
Toluene	5	<0.7	<0.7	<0.7	<0.7
trans-1,2-Dichloroethene	5	<0.7	<0.7	<0.7	<0.7
trans-1,3-Dichloropropene	0.4	<0.16	<0.16	<0.16	<0.16
Trichloroethene	5	<0.18	2.9	2.8	<0.18
Trichlorofluoromethane	5	<0.7	<0.7	<0.7	<0.7
Vinyl chloride	2	<0.07	<0.07	<0.07	<0.07
Total VOCs		-	2.9	4.3	-

Notes:

All values displayed in micrograms per liter (ug/l), equal to parts per billion (ppb)
 c - Indicates compound was not detected above the indicated laboratory method detection limit (MDL).
 Bold font indicates the concentration exceeds the MDL.
 VOCs analyzed by USEPA Method 8260D
 NL indicates Not Listed
 J indicates an estimated value (above the MDL but below the laboratory method reportable limit (RL))
 Data has been validated
 Blue font represents a change made in the DUSR
 UJ indicates nondetect with estimated quantitation limits

Table 4B
Phase II Environmental Site Assessment
42 York Street, Rochester, New York 14614
Summary of Semi-Volatile Organic Compounds in Groundwater
LaBella Project # 2230119



Well Location: Sample ID: Laboratory ID Screen Depth (ft bgs): Sample Date:	NY-TOGS-GA	MW-01	YS-MW-2023-01	
		MW-01 072623	YS-MW-2023-01 072623	YS-MW-2023-01 072623
		L2343170-02	L2343170-01	L2343170-03
		3.5 - 10.5	3.0 - 11.0	
		7/26/2023	7/26/2023	7/26/2023
SVOCs				
1,2,4,5-Tetrachlorobenzene	5	<0.62	<0.62	<0.62
2,3,4,6-Tetrachlorophenol	NL	<0.47	<0.47	<0.47
2,4,5-Trichlorophenol	NL	<0.38	<0.38	<0.38
2,4,6-Trichlorophenol	NL	<0.49	<0.49	<0.49
2,4-Dichlorophenol	2	<0.53	<0.53	<0.53
2,4-Dimethylphenol	2	<1.1	<1.1	<1.1
2,4-Dinitrophenol	2	<3.6	<3.6	<3.6
2,4-Dinitrotoluene	5	<0.38	<0.38	<0.38
2,6-Dinitrotoluene	5	<0.37	<0.37	<0.37
2-Chlorophenol	NL	<0.4	<0.4	<0.4
2-Methylphenol	NL	<1.1	<1.1	<1.1
2-Nitroaniline	5	<0.52	<0.52	<0.52
2-Nitrophenol	NL	<0.46	<0.46	<0.46
3,3-Dichlorobenzidine	5	<0.85	<0.85	<0.85
3-Methylphenol/4-Methylphenol	NL	<0.55	<0.55	<0.55
3-Nitroaniline	5	<0.57	<0.57	<0.57
4,6-Dinitro-o-cresol	NL	<5.4	UJ	<5.4
4-Bromophenyl phenyl ether	NL	<0.63	<0.63	<0.63
4-Chloroaniline	5	<0.65	<0.65	<0.65
4-Chlorophenyl phenyl ether	NL	<0.8	<0.8	<0.8
4-Nitroaniline	5	<0.58	<0.58	<0.58
4-Nitrophenol	NL	<1.1	<1.1	<1.1
Acetophenone	NL	<0.98	<0.98	<0.98
Atrazine	7.5	<1.7	<1.7	<1.7
Benzaldehyde	NL	<0.9	<0.9	<0.9
Biphenyl	NL	<0.64	<0.64	<0.64
Bis(2-chloroethoxy)methane	5	<1.5	<1.5	<1.5
Bis(2-chloroethyl)ether	1	<0.88	<0.88	<0.88
Bis(2-chloroisopropyl)ether	5	<1.8	<1.8	<1.8
Bis(2-ethylhexyl)phthalate	5	<1.5	<1.5	<1.5
Butyl benzyl phthalate	50	<2.2	<2.2	<2.2
Caprolactam	NL	<1.3	<1.3	<1.3
Carbazole	NL	<0.76	<0.76	<0.76
Di-n-butylphthalate	50	<0.58	<0.58	<0.58
Di-n-octylphthalate	50	<2.4	<2.4	<2.4
Dibenzofuran	NL	<0.82	<0.82	<0.82
Diethyl phthalate	50	<4.3	<4.3	<4.3
Dimethyl phthalate	50	<4.4	<4.4	<4.4
Hexachlorocyclopentadiene	5	<0.61	<0.61	<0.61
Isophorone	50	<0.66	<0.66	<0.66
n-Nitrosodi-n-propylamine	NL	<0.77	<0.77	<0.77
NDPA/DPA	50	<0.65	<0.65	<0.65
Nitrobenzene	0.4	<0.66	<0.66	<0.66
p-Chloro-m-cresol	NL	<0.41	<0.41	<0.41
Phenol	2	<1.3	<1.3	<1.3
SVOCs (GC/MS-SIM)				
2-Chloronaphthalene	10	<0.04	<0.04	<0.04
2-Methylnaphthalene	NL	<0.05	<0.05	<0.05
Acenaphthene	20	<0.04	<0.04	<0.04
Acenaphthylene	NL	<0.04	<0.04	<0.04
Anthracene	50	<0.04	<0.04	<0.04
Benzo(a)anthracene	0.002	<0.02	0.02 J	<0.02
Benzo(a)pyrene	0	<0.04	<0.04	<0.04
Benzo(b)fluoranthene	0.002	<0.02	0.03 J	<0.02
Benzo(ghi)perylene	NL	<0.04	<0.04	<0.04
Benzo(k)fluoranthene	0.002	<0.04	<0.04	<0.04
Chrysene	0.002	<0.04	<0.04	<0.04
Dibenzo(a,h)anthracene	NL	<0.04	<0.04	<0.04
Fluoranthene	50	<0.04	0.05 J	<0.04
Fluorene	50	<0.04	<0.04	<0.04
Hexachlorobenzene	0.04	<0.03	<0.03	<0.03
Hexachlorobutadiene	0.5	<0.04	<0.04	<0.04
Hexachloroethane	5	<0.03	<0.03	<0.03
Indeno(1,2,3-cd)pyrene	0.002	<0.04	<0.04	<0.04
Naphthalene	10	<0.04	<0.04	<0.04
Pentachlorophenol	2	1.9	<0.22	0.27 J
Phenanthrene	50	<0.02	0.02 J	<0.02
Pyrene	50	<0.04	0.04 J	<0.04
Total SVOCs		1.9	0.16	0.27

Notes:

All values displayed in micrograms per liter (ug/l), equal to parts per billion (ppb)

*< - Indicates compound was not detected above the indicated laboratory method detection limit (MDL).

Bold font indicates the concentration exceeds the method detection limit (MDL).

Yellow highlight indicates that the compound was detected at a concentration above its respective 6 NYCRR Part 703 Groundwater Quality Standard or Technical and Operational Guidance Series (TOGS 1.1.1) Guidance Value

SVOCs analyzed by USEPA Method 8270E

NL indicates Not Listed

J indicates an estimated value (above the MDL but below the laboratory method reportable limit (RL))

Data has been validated

Blue font represents a change made in the DUSR.

UJ indicates nondetect with estimated quantitation limits

Table 4C
Phase II Environmental Site Assessment
42 York Street, Rochester, New York 14614
Summary of Metals in Groundwater
LaBella Project # 2230119



Well Location:	NY-TOGS-GA	MW-01	YS-MW-2023-01	
Sample ID:		MW-01 072623	YS-MW-2023-01 072623	YS-MW-2023-BD-072623
Laboratory ID		L2343170-02	L2343170-01	L2343170-03
Screen Depth (ft bgs):		3.5 - 10.5	3.0 - 11.0	
Sample Date:		7/26/2023	7/26/2023	7/26/2023
Metals				
Aluminum, Total	2000	113	251	123
Antimony, Total	6	0.8 J	<0.42	<0.42
Arsenic, Total	50	2.37	1.51	1.25
Barium, Total	2000	30.53	152.9	95.36
Beryllium, Total	3	<0.1	<0.1	<0.1
Cadmium, Total	10	0.11 J	0.15 J	<0.05
Calcium, Total	NL	53100	115000	107000
Chromium, Total	100	0.52 J	1.05	0.84 J
Cobalt, Total	NL	0.72	0.31 J	0.18 J
Copper, Total	1000	6.11	102.2	12.16
Iron, Total	600	1170	681	247
Lead, Total	50	1.11	15.77	2.36
Magnesium, Total	35000	9760	25900	27100
Manganese, Total	600	533.6	81.69	42.68
Mercury, Total	1.4	<0.09	<0.09	<0.09
Nickel, Total	200	1.99 J	3.57	1.09 J
Potassium, Total	NL	3520	7320	6720
Selenium, Total	20	<1.73	5.38	5.02
Silver, Total	100	<0.16	<0.16	<0.16
Sodium, Total	NL	23700	5880	6210
Thallium, Total	0.5	<0.14	<0.14	<0.14
Vanadium, Total	NL	1.89 J	1.87 J	<1.57
Zinc, Total	5000	101.6	125	19.36

Notes:

All values displayed in micrograms per liter (ug/l), equal to parts per billion (ppb)

"<" - Indicates compound was not detected above the indicated laboratory method detection limit (MDL).

Bold font indicates the concentration exceeds the method detection limit (MDL).

Yellow highlight indicates that the compound was detected at a concentration above its respective 6 NYCRR Part 703 Groundwater Quality Standard or Technical and Operational Guidance Series (TOGS 1.1.1) Guidance Value

* indicates no Part 703 Standard, TOGS 1.1.1 Guidance Value is listed

Metals analyzed by USEPA Method 6020B/7470A

NL indicates Not Listed

J indicates an estimated value (above the MDL but below the laboratory method reportable limit (RL))

Table 5
Phase II Environmental Site Assessment
42 York Street, Rochester, New York 14614
Summary of Waste Characterization Analytical Results
LaBella Project # 2230119



Sample Location:	TCLP Standard	TP-01	TP-06	TP-08	TP-09		TP-10
Sample ID:		WC-01-0.25-5 FT	WC-08-0.4-3 FT	WC-03-1-6 FT	WC-02-0.75-2 FT	WC-05-4-5 FT	WC-04-3.5-6.5 FT
Laboratory ID:		L2339895-01	L2339895-08	L2339895-03	L2339895-02	L2339895-05	L2339895-04
Sample Depth (ft bgs):		0.25-5	0.4-3	1-6	0.75-2	4-5	3.5-6.5
Sample Date:		7/11/2023	7/12/2023	7/12/2023	7/11/2023	7/11/2023	7/12/2023
TCLP VOCs							
1,1-Dichloroethene	0.7	<0.0017	<0.0017	<0.0017	<0.0017	<0.0017	<0.0017
1,2-Dichloroethane	0.5	<0.0013	<0.0013	<0.0013	<0.0013	<0.0013	<0.0013
1,4-Dichlorobenzene	7.5	<0.0019	<0.0019	<0.0019	<0.0019	<0.0019	<0.0019
2-Butanone	200	<0.019	<0.019	<0.019	<0.019	<0.019	<0.019
Benzene	0.5	<0.0016	<0.0016	<0.0016	<0.0016	<0.0016	<0.0016
Carbon tetrachloride	0.5	<0.0013	<0.0013	<0.0013	<0.0013	<0.0013	<0.0013
Chlorobenzene	100	<0.0018	<0.0018	<0.0018	<0.0018	<0.0018	<0.0018
Chloroform	6	<0.0022	<0.0022	<0.0022	<0.0022	<0.0022	<0.0022
Tetrachloroethene	0.7	<0.0018	<0.0018	<0.0018	<0.0018	<0.0018	<0.0018
Trichloroethene	0.5	<0.0018	<0.0018	<0.0018	<0.0018	<0.0018	<0.0018
Vinyl chloride	0.2	<0.00071	<0.00071	<0.00071	<0.00071	<0.00071	<0.00071
TCLP SVOCs							
2,4,5-Trichlorophenol	400	<0.0019	<0.0019	<0.0019	<0.0019	<0.0019	<0.0019
2,4,6-Trichlorophenol	2	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025
2,4-Dinitrotoluene	0.13	<0.0019	<0.0019	<0.0019	<0.0019	<0.0019	<0.0019
2-Methylphenol	200	<0.0055	<0.0055	<0.0055	<0.0055	<0.0055	<0.0055
3-Methylphenol/4-Methylphenol	200	<0.0028	<0.0028	<0.0028	<0.0028	<0.0028	<0.0028
Hexachlorobenzene	0.13	<0.0034	<0.0034	<0.0034	<0.0034	<0.0034	<0.0034
Hexachlorobutadiene	0.5	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
Hexachloroethane	3	<0.0022	<0.0022	<0.0022	<0.0022	<0.0022	<0.0022
Nitrobenzene	2	<0.0033	<0.0033	<0.0033	<0.0033	<0.0033	<0.0033
Pentachlorophenol	100	<0.0098	<0.0098	<0.0098	<0.0098	<0.0098	<0.0098
Pyridine	5	<0.0045	<0.0045	<0.0045	<0.0045	<0.0045	<0.0045
PCBs							
Aroclor 1016	NL	<0.00516	<0.0064	<0.00474	<0.00647	<0.00594	<0.00596
Aroclor 1221	NL	<0.00582	<0.00722	<0.00535	<0.0073	<0.00671	<0.00673
Aroclor 1232	NL	<0.0123	<0.0153	<0.0113	<0.0154	<0.0142	<0.0142
Aroclor 1242	NL	<0.00783	<0.00972	<0.00719	<0.00982	<0.00902	<0.00905
Aroclor 1248	NL	<0.00871	<0.0108	<0.008	<0.0109	<0.01	<0.0101
Aroclor 1254	NL	<0.00635	<0.00789	<0.00584	<0.00797	<0.00732	<0.00734
Aroclor 1260	NL	<0.0107	<0.0133	<0.00986	0.0212 J	<0.0124	<0.0124
Aroclor 1262	NL	<0.00738	<0.00916	<0.00678	<0.00926	<0.0085	<0.00852
Aroclor 1268	NL	<0.00602	<0.00747	<0.00553	<0.00755	<0.00693	<0.00695
PCBs, Total	NL	<0.00516	<0.0064	<0.00474	0.0212 J	<0.00594	<0.00596
TCLP Metals							
Arsenic, TCLP	5	<0.019	<0.019	<0.019	0.0223 J	<0.019	<0.019
Barium, TCLP	100	0.458 J	0.219 J	0.215 J	0.147 J	0.388 J	0.647
Cadmium, TCLP	1	<0.01	0.0188 J	<0.01	<0.01	<0.01	<0.01
Chromium, TCLP	5	<0.021	<0.021	<0.021	<0.021	<0.021	<0.021
Lead, TCLP	5	0.323 J	<0.027	<0.027	0.344 J	<0.027	0.575
Mercury, TCLP	0.2	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
Selenium, TCLP	1	<0.035	<0.035	<0.035	<0.035	<0.035	<0.035
Silver, TCLP	5	<0.028	<0.028	<0.028	<0.028	<0.028	<0.028
General Chemistry							
Cyanide, Reactive	NL	<10	<10	<10	<10	<10	<10
Sulfide, Reactive	NL	<10	<10	<10	<10	<10	<10
Corrosivity (pH)	<2 or >12.5	7.91	7.07	7.54	7.6	8.32	7.39
Ignitability	<140 F	NI	NI	NI	NI	NI	NI

NOTES:

All values displayed in milligrams per Liter (mg/L), equal to parts per million (ppm)

"<" - Indicates compound was not detected above the indicated laboratory method detection limit (MDL).

Bold font indicates the concentration exceeds the MDL.

NL indicates Not Listed

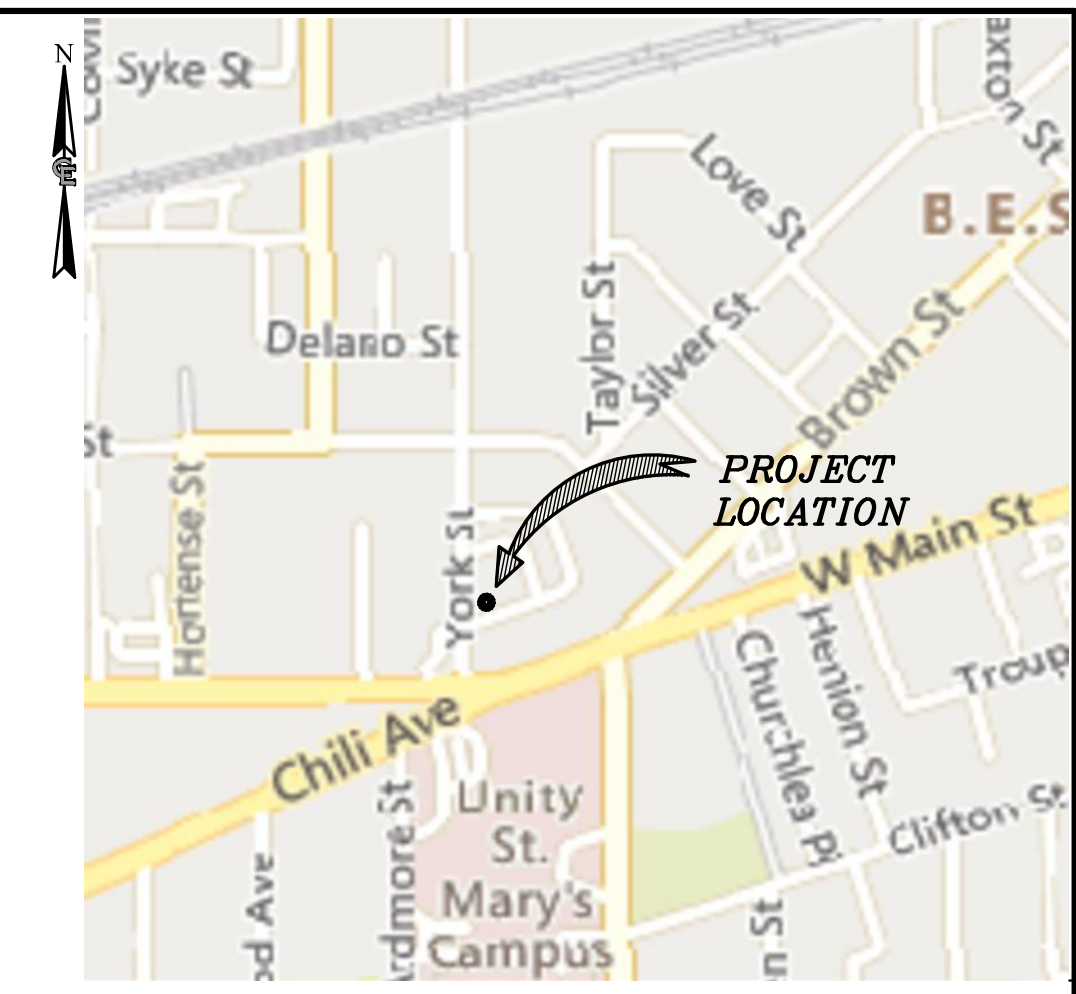
NI indicates that the sample is non-ignitable.

J indicates an estimated value (above the MDL but below the laboratory reportable limit (RL)).



APPENDIX 1

York Street Environmental Investigation Exhibit (Survey) – Existing Features
(By Costich Engineering, DPC)



LOCATION SKETCH

SYMBOL LEGEND

⊙	DRAINAGE MANHOLE	⊕	ELECTRIC MANHOLE
⊕	INLET DRAINAGE MANHOLE	⊕	ELECTRIC METER
⊕	CATCH BASIN	⊕	TELEPHONE MANHOLE
⊕	END SECTION	⊕	TELEPHONE PEDESTAL
⊕	END OF PIPE	⊕	PEDESTRIAN POLE
⊕	MANHOLE	⊕	TRAFFIC CONTROL CABINET
⊕	SANITARY MANHOLE	⊕	LAMP POST
⊕	CLEAN OUT	⊕	LIGHT POLE
⊕	GAS VALVE	⊕	UTILITY POLE WITH LIGHT
⊕	GAS SERVICE	⊕	FLAG POLE
⊕	GAS METER	⊕	MAILBOX
⊕	SPRINKLER VALVE	⊕	BOLLARD
⊕	SPRINKLER HEAD	⊕	POST
⊕	WATER VALVE	⊕	SIGN
⊕	WATER SERVICE	⊕	SIGN
⊕	HYDRANT	⊕	SIGN
⊕	WATER METER	⊕	TURNING ARROW
⊕	WELL	⊕	HANDICAP
⊕	BORE	⊕	STOP BAR
⊕	CABLE TV PEDESTAL	⊕	TREE DECIDUOUS
⊕	SIGNAL POLE	⊕	TREE CONIFEROUS
⊕	UTILITY POLE	⊕	BUSH
⊕	GUY WIRE	⊕	AIR CONDITIONING UNIT
⊕	PULL BOX	⊕	SANITARY UTILITY LATH
⊕	ELECTRIC PULL BOX	⊕	GAS UTILITY LATH
⊕	TELEPHONE PULL BOX	⊕	WATER UTILITY LATH
⊕	TRANSFORMER	⊕	TELEPHONE UTILITY LATH
		⊕	ELECTRIC UTILITY LATH
		⊕	CABLE UTILITY LATH

SURVEY NOTES

- ALL BOUNDARY SHOWN IS APPROXIMATE PER CITY OF ROCHESTER TAX MAP OVERLAY.
- LOCATIONS SHOWN FROM A FIELD SURVEY BY COSTICH ENGINEERING ON 8/3/2013 HORIZONTAL AND VERTICAL DATA OBTAINED THROUGH NYS DOT CORS NETWORK REFERENCED TO THE FOLLOWING MONUMENT
 PITTSFORD CORS STATION
 -LATITUDE: 43-05-35.48461 (N) NAD 83 (CORS)
 -LONGITUDE: 077-31-31.11244 (W)
 -ELLIP HEIGHT: 113.481 METERS NAVD 88 (CORS)

LINE LEGEND

---	SECTION/PARCEL BOUNDARY
---	MIN. BUILDING SETBACK
---	CENTER LINE
---	EXIST. EASEMENT LINE
---	EXIST. RIGHT-OF-WAY LINE
---	EXIST. EDGE OF PAVEMENT
---	EXISTING WATER MAIN, VALVE, & HYDRANT.
---	EXISTING SANITARY SEWER, & MANHOLE.
---	EXISTING DRAINAGE SEWER, FIELD INLET, INLET MANHOLE, MANHOLE, & END SECTION.
---	EXISTING OVERHEAD UTILITIES
---	EXISTING TELEPHONE
---	EXISTING UNDERGROUND UTILITIES
---	EXISTING GAS
---	EXISTING ELECTRIC
---	EXISTING GUARD RAIL
---	TREE, HEDGE, EDGE OF WOODS
---	EXISTING SWALE
---	BARBED WIRE, STOCKADE, CHAIN LINKED FENCE
---	EXISTING CONTOUR
---	EXISTING SPOT ELEVATION @ X
---	CONCRETE PAD/ CONCRETE SIDEWALK

CERTIFICATION

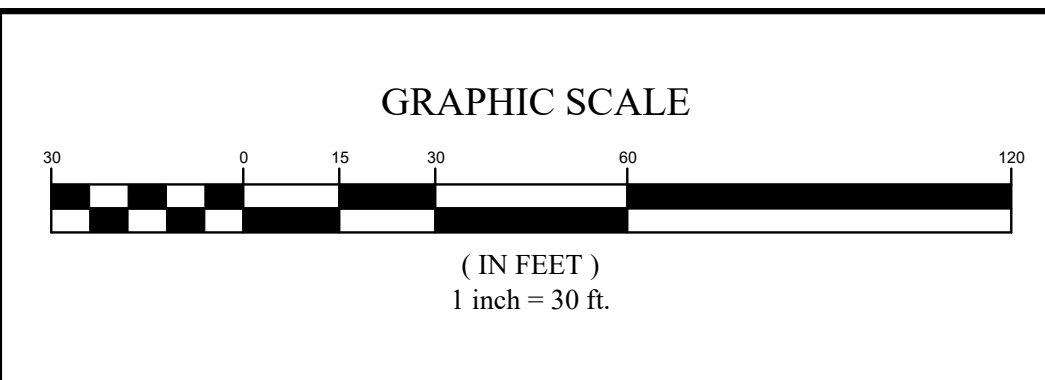
WE, COSTICH ENGINEERING, D.P.C., HEREBY CERTIFY TO

THAT THIS MAP WAS PREPARED FROM NOTES OF AN INSTRUMENT SURVEY COMPLETED ON 08/03/2023 AND FROM THE REFERENCE(S) LISTED HEREON. NO SEARCH OF RECORDS, OTHER THAN THOSE REFERENCED, WAS MADE FOR EASEMENTS OR ENCUMBRANCES AFFECTING THIS PARCEL.

By: *Daniel T. Hickok* Date: 08-11-23
 Daniel T. Hickok, N.Y.S. L.S., No. 050449

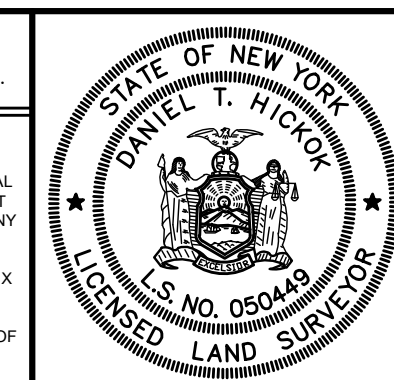


EXISTING UTILITIES (LOCATION, SIZES AND INVERTS) SHOWN ON THE PLANS ARE APPROXIMATE AND ARE NOT CERTIFIED AS TO THE ACCURACY OF THEIR LOCATION OR COMPLETENESS. THE CONTRACTOR SHALL BE RESPONSIBLE FOR DETERMINING THE EXACT LOCATIONS AND DEPTHS OF ALL UTILITIES AND STRUCTURES IN THE PATH OF, OR CLOSELY PARALLEL TO, OR UNDER, THE PROPOSED CONSTRUCTION. THE CONTRACTOR SHALL BE RESPONSIBLE FOR ANY DELAYS OR DAMAGES OCCURRING AS A RESULT OF INCORRECTLY LOCATED UTILITIES. IT IS THE CONTRACTORS RESPONSIBILITY TO NOTIFY THE VARIOUS UTILITY OWNERS IN AMPLI TIME FOR THEM TO LOCATE AND MARK THEIR FACILITIES. THE CONTRACTOR SHALL ALSO NOTIFY UNDERGROUND UTILITY LOCATION SERVICE AT LEAST 48 HOURS IN ADVANCE OF COMMENCING ANY WORK.

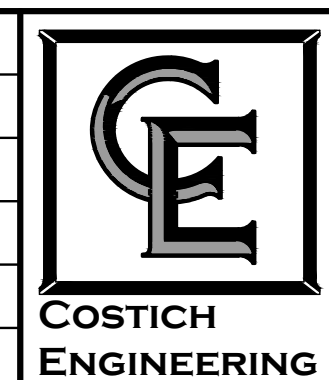


NO.	DATE	REVISION	BY	CHKD.	APVLS.

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PROJECT MANAGER: D.T.H.
 DRAWN BY: T.F.R.
 BOUNDARY: ---
 TOPBASE: T.F.R.
 DATE: 08/10/2023
 SCALE: 1"=30'



• CIVIL ENGINEERING
 • LAND SURVEYING
 • LANDSCAPE ARCHITECTURE
 217 LAKE AVENUE
 ROCHESTER, NY 14608
 (585) 458-3020

TITLE OF PROJECT YORK STREET ENVIORNMENTAL INVESTIGATION EXHIBIT
TITLE OF DRAWING EXISTING FEATURES
LOCATION OF PROJECT TAX PARCEL NO.'S 120-42-1-46, 120-42-1-47, 120-42-2-65, 120-42-2-70, 120-42-2-71, 120-42-2-72, 001 CITY OF ROCHESTER, COUNTY OF MONROE, STATE OF NEW YORK
CLIENT LABELLA ASSOCIATES 300 STATE STREET - SUITE 201 ROCHESTER, NEW YORK 14614
DWG. # 8694 VE100 SHEET 1 OF 1



APPENDIX 2

Field Logs



TEST PIT LOG

Test Pit No.	TP-01
Sheet	1 of 1
Project No.:	2230119
Chkd By:	DB
Start Date:	7/11/2023
Finish Date:	7/11/2023

Project Name:	Phase II ESA
Location:	42 York Street, Rochester, New York
Client:	City of Rochester / NYSDEC / USEPA
Contractor:	LaBella ENV LLC
Operator:	Andrew LeFebvre

Key:		Equipment: Kubota Excavator
_____	Geologic Strata Change	Logged By: A. daSilva
_____	Gradation Change Within Strata	Time Start: 1225
_____	End of Test Pit	LaBella Rep.: A. daSilva
		Time End: 1530

Test Pit Location: North portion of Site.

Depth (ft.)	Sample Number / ID	Depth of Change (FT)	VISUAL-MANUAL MATERIAL DESCRIPTION <small>trace (1 - 10%), little (11 - 20%), some (21 - 35%), and (36-50%); WOH = weight of hammer; WOR = weight of rod</small>	PID (parts per million)	COMMENTS
1	WC-01-0.25-5 FT	0.25	ASPHALT	0	Possible native material starting at 5 feet bgs; however, no oxidation or oxidation-transition zone was identified to verify native materials. Additionally, no apparent bedding to aid in identifying native soil.
2		1.5	ASPHALT SUBBASE GRAVEL AND possible cinders / slag (glass and copper slag)	0	
3			Brown reworked F to M SAND ; little brick ; little cobble ; little silt ; moist to wet	0	
4		3.0	ASH FILL LAYER - ash ; glass ; wood ; moist	0	
5		5.0	Light tan-brown F SAND ; some silt ; little SR gravel (limestone, red sandstone, dolostone, trace chert) ; trace clay (plastic) ; trace cobbles ; massive ; moist	0	
6				0	
7				0	
8				0	
9		9.0	Apparent Bedrock refusal at 9 feet bgs	0	
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

DEPTH (FT)				ADDITIONAL NOTES:	
WATER LEVEL DATA			BOTTOM OF TEST PIT	GROUNDWATER ENCOUNTERED	
Date	Time	Elapsed Time			
NA	NA	NA			
			9.0	NA	

GENERAL NOTES

- STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL.
- WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER

BGS = Below Ground Surface and = 35 - 50% C = Coarse R = Rounded
 NA = Not Applicable some = 20 - 35% M = Medium A = Angular
 little = 10 - 20% F = Fine SR = Subrounded
 trace = 1 - 10% VF = Very Fine SA = Subangular



TEST PIT LOG

Test Pit No.	TP-04
Sheet	1 of 1
Project No.:	2230119
Chkd By:	DB
Start Date:	7/12/2023
Finish Date:	7/12/2023

Project Name:	Phase II ESA
Location:	42 York Street, Rochester, New York
Client:	City of Rochester / NYSDEC / USEPA
Contractor:	LaBella ENV LLC
Operator:	Andrew LeFebvre

Key:	Geologic Strata Change	Equipment:	Kubota Excavator
-----	Gradation Change Within Strata	Logged By:	A. daSilva
-----	End of Test Pit	LaBella Rep.:	A. daSilva
		Time Start:	0710
		Time End:	0800

Test Pit Location: Western portion of Site.

Depth (ft.)	Sample Number / ID	Depth of Change (FT)	VISUAL-MANUAL MATERIAL DESCRIPTION trace (1 - 10%), little (11 - 20%), some (21 - 35%), and (36-50%); WOH = weight of hammer ; WOR = weight of rod	PID (parts per million)	COMMENTS
1	TP-04-0.5-2ft T: 0800	0.25	ASPHALT		Possible native material starting at 4.5 feet bgs; however, no oxidation or oxidation-transition zone was identified to verify native materials. Additionally, no apparent bedding to aid in identifying native soil.
		0.5	SUBBASE GRAVEL	0	
2		2.0	Black stained URBAN FILL MATERIAL (apparent brick road with ash and terracotta subbase on northeast end of test pit beneath asphalt subbase gravel; apparent dolostone building foundation blocks located on west end of trench)	0	
3			Reworked brown F SAND ; some silt ; little SR gravel ; little cobbles (possible foundation material based on layering) ; moist	0	
4		4.5	Light tan-brown F SAND ; some silt ; little SR gravel (limestone, red sandstone, dolostone, trace chert) ; trace clay (plastic) ; trace cobbles ; massive ; moist	0	
5				0	
6				0	
7				0	
8				0	
9				0	
10	9.75	Apparent Bedrock refusal at 9.75 feet bgs	0		
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

DEPTH (FT)			ADDITIONAL NOTES:	
WATER LEVEL DATA			BOTTOM OF TEST PIT	GROUNDWATER ENCOUNTERED
Date	Time	Elapsed Time		
NA	NA	NA	9.75	NA

GENERAL NOTES

- STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL.
- WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER

BGS = Below Ground Surface and = 35 - 50% C = Coarse R = Rounded
 NA = Not Applicable some = 20 - 35% M = Medium A = Angular
 little = 10 - 20% F = Fine SR = Subrounded
 trace = 1 - 10% VF = Very Fine SA = Subangular

TEST PIT: TP-04



TEST PIT LOG

Test Pit No.	TP-05
Sheet	1 of 1
Project No.:	2230119
Chkd By:	DB
Start Date:	7/12/2023
Finish Date:	7/12/2023

Project Name:	Phase II ESA
Location:	42 York Street, Rochester, New York
Client:	City of Rochester / NYSDEC / USEPA
Contractor:	LaBella ENV LLC
Operator:	Andrew LeFebvre

Key:	_____ Geologic Strata Change	Equipment:	Kubota Excavator
_____ Gradation Change Within Strata		Logged By:	A. daSilva
_____ End of Test Pit		LaBella Rep.:	A. daSilva
		Time Start:	0848
		Time End:	0930

Test Pit Location: Eastern portion of Site.

Depth (ft.)	Sample Number / ID	Depth of Change (FT)	VISUAL-MANUAL MATERIAL DESCRIPTION trace (1 - 10%), little (11 - 20%), some (21 - 35%), and (36-50%); WOH = weight of hammer ; WOR = weight of rod	PID (parts per million)	COMMENTS
1	TP-05-0.5-2.5ft T: 0930	0.25	ASPHALT	0	Possible native material starting at 2.5 feet bgs; however, no oxidation or oxidation-transition zone was identified to verify native materials. Additionally, no apparent bedding to aid in identifying native soil.
		0.5	ASPHALT SUBBASE GRAVEL	0	
		2.5	URBAN FILL - ASH intermixed with dark brown F to M SAND, AND SILT ; little brick ; little metal slag and wire ; little SA to SR gravel ; trace glass ; moist	0	
			Possible reworked Light tan-brown F SAND ; some silt ; little SR gravel (limestone, red sandstone, dolostone, trace chert) ; trace clay (plastic) ; trace cobbles ; massive ; moist	0	
4.5	Refusal at 4.5 feet bgs - possible bedrock. Moved test pit 3 feet towards to north to avoid blockage - could not pass stone.	0			
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

WATER LEVEL DATA			DEPTH (FT)	BOTTOM OF TEST PIT	GROUNDWATER ENCOUNTERED	ADDITIONAL NOTES:
Date	Time	Elapsed Time				
NA	NA	NA				

GENERAL NOTES

- STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL.
 - WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER
- BGS = Below Ground Surface and = 35 - 50% C = Coarse R = Rounded
 NA = Not Applicable some = 20 - 35% M = Medium A = Angular
 little = 10 - 20% F = Fine SR = Subrounded
 trace = 1 - 10% VF = Very Fine SA = Subangular

TEST PIT: TP-05



TEST PIT LOG

Test Pit No.	TP-08
Sheet	1 of 1
Project No.:	2230119
Chkd By:	DB
Start Date:	7/12/2023
Finish Date:	7/12/2023

Project Name:	Phase II ESA
Location:	42 York Street, Rochester, New York
Client:	City of Rochester / NYSDEC / USEPA
Contractor:	LaBella ENV LLC
Operator:	Andrew LeFebvre

Key:	Equipment: Kubota Excavator	Time Start: 1202	
----- Geologic Strata Change	Logged By: A. daSilva	Time End: 1245	
----- Gradation Change Within Strata	LaBella Rep.: A. daSilva		
----- End of Test Pit			

Test Pit Location: Eastern portion of Site.

Depth (ft.)	Sample Number / ID	Depth of Change (FT)	VISUAL-MANUAL MATERIAL DESCRIPTION <small>trace (1 - 10%), little (11 - 20%), some (21 - 35%), and (36-50%); WOH = weight of hammer ; WOR = weight of rod</small>	PID (parts per million)	COMMENTS
1	WC-03-1-6 FT	0.25	ASPHALT		
		1.0	ASPHALT SUBBASE GRAVEL	0	
		1.3	ASH AND F brown SAND ; some silt ; dry		
2		2.0	Brown F SAND ; some silt ; some metal slag and charcoal ; dry	0	
3			URBAN FILL - F brown SAND AND URBAN FILL (little brick ; little metal) ; dry to moist	0	
4				0	
5				0	
6		7.0	SATURATION at 6.0 feet bgs	0	
7			Apparent bedrock refusal at 7 feet bgs		
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

DEPTH (FT)			ADDITIONAL NOTES:
WATER LEVEL DATA			
Date	Time	Elapsed Time	
NA	NA	NA	BOTTOM OF TEST PIT 7.0
			GROUNDWATER ENCOUNTERED 6.0

GENERAL NOTES

- 1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL.
- 2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER

BGS = Below Ground Surface and = 35 - 50% C = Coarse R = Rounded
 NA = Not Applicable some = 20 - 35% M = Medium A = Angular
 little = 10 - 20% F = Fine SR = Subrounded
 trace = 1 - 10% VF = Very Fine SA = Subangular



TEST PIT LOG

Test Pit No.	TP-09
Sheet	1 of 1
Project No.:	2230119
Chkd By:	DB
Start Date:	7/11/2023
Finish Date:	7/11/2023

Project Name:	Phase II ESA
Location:	42 York Street, Rochester, New York
Client:	City of Rochester / NYSDEC / USEPA
Contractor:	LaBella ENV LLC
Operator:	Andrew LeFebvre

Key:	Geologic Strata Change	Equipment:	Kubota Excavator
-----	Gradation Change Within Strata	Logged By:	A. daSilva
-----	End of Test Pit	LaBella Rep.:	A. daSilva
		Time Start:	1100
		Time End:	1200

Test Pit Location: Northwestern portion of Site (adjacent to TP-13 advanced in 2018).

Depth (ft.)	Sample Number / ID	Depth of Change (FT)	VISUAL-MANUAL MATERIAL DESCRIPTION trace (1 - 10%), little (11 - 20%), some (21 - 35%), and (36-50%); WOH = weight of hammer; WOR = weight of rod	PID (parts per million)	COMMENTS
1	WC-02-0.75-2 FT	0.25	ASPHALT		Possible native material starting at 5 feet bgs; however, no oxidation or oxidation-transition zone was identified to verify native materials. Additionally, no apparent bedding to aid in identifying native soil.
		0.75	ASPHALT SUBBASE GRAVEL	0	
2		2.0	URBAN FILL MATERIAL (ASH, glass, F to M brick chunks) ; some C sand ; moist to dry	0	
3			Brown F SAND ; some silt ; little SA gravel ; trace C sand ; moist ; Not native	0	
4				0	
5		5.0		Brown F to M SAND ; little C SA sand ; little SR gravel ; little cobble ; trace silt ; moist to wet ; massive	
6				0	
7				0	
8				0	
9		9.0	Apparent bedrock refusal at 9 feet bgs	0	
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

DEPTH (FT)			ADDITIONAL NOTES: Only waste characterization samples collected.	
WATER LEVEL DATA			BOTTOM OF TEST PIT	GROUNDWATER ENCOUNTERED
Date	Time	Elapsed Time	9.0	NA
NA	NA	NA		

GENERAL NOTES

1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL.

2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER

BGS = Below Ground Surface and = 35 - 50% C = Coarse R = Rounded
 NA = Not Applicable some = 20 - 35% M = Medium A = Angular
 little = 10 - 20% F = Fine SR = Subrounded
 trace = 1 - 10% VF = Very Fine SA = Subangular

TEST PIT: TP-09



TEST PIT LOG

Test Pit No.	TP-10
Sheet	1 of 1
Project No.:	2230119
Chkd By:	DB
Start Date:	7/12/2023
Finish Date:	7/12/2023

Project Name:	Phase II ESA
Location:	42 York Street, Rochester, New York
Client:	City of Rochester / NYSDEC / USEPA
Contractor:	LaBella ENV LLC
Operator:	Andrew LeFebvre

Key:		Equipment: Kubota Excavator
_____	Geologic Strata Change	Logged By: A. daSilva
_____	Gradation Change Within Strata	Time Start: 1315
_____	End of Test Pit	LaBella Rep.: A. daSilva
		Time End: 1400

Test Pit Location: Southeastern portion of Site (adjacent to TP-14 advanced in 2018).

Depth (ft.)	Sample Number / ID	Depth of Change (FT)	VISUAL-MANUAL MATERIAL DESCRIPTION <small>trace (1 - 10%), little (11 - 20%), some (21 - 35%), and (36-50%); WOH = weight of hammer ; WOR = weight of rod</small>	PID (parts per million)	COMMENTS	
1	WC-04-3.5-6.5 FT	0.25	ASPHALT			
		0.5	ASPHALT SUBBASE GRAVEL	0		
2		2.0	Brown F SAND ; some silt ; little SR gravel ; some dolostone blocks (apparent building footer wall) ; trace cobble ; moist to dry	0		
3			Fill material - Dark brown F to M SAND ; little silt ; little fill material (brick chunks, metal, glass, and ash) ; moist to dry	0		
4				0		
5				0		
6				0		
7			6.5	Possible reworked brown F SAND ; some silt ; some dolostone footer wall blocks ; little SR gravel ; moist	0	
8					0	
9			9.5	Apparent bedrock refusal at 9.5 feet bgs	0	
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

DEPTH (FT)			ADDITIONAL NOTES:	
WATER LEVEL DATA			BOTTOM OF TEST PIT	GROUNDWATER ENCOUNTERED
Date	Time	Elapsed Time		
NA	NA	NA	9.5	NA

GENERAL NOTES

- STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL.
- WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER

BGS = Below Ground Surface and = 35 - 50% C = Coarse R = Rounded
 NA = Not Applicable some = 20 - 35% M = Medium A = Angular
 little = 10 - 20% F = Fine SR = Subrounded
 trace = 1 - 10% VF = Very Fine SA = Subangular



BORING LOG

Boring No. YS-MW-2023-01

Sheet 1 of 1

Project No.: 2230119

Project Name: Phase II ESA - Subtask 1.2

CHKD BY:

Location: 42 York Street, Rochester, New York

Start Date: 7/17/2023

Client: City of Rochester / NYSDEC / EPA

Finish Date: 7/17/2023

Drilling Firm: LaBella LLC

Driller: Mike / Matt Pepe

Key:	Drill Rig: Geoprobe 7822DT	Core Type: Auger
_____ Geologic Strata Change	Casing: 4.25" interior / 8" exterior diameter auger	Time Start: 840
----- Gradation Change Within Strata	Sampler: Macro-Core - 5ft length, 2 in diameter	Time End: 1100
_____ End of Boring or Overpacked	Sampling Method: Direct Push	LaBella Rep. A. daSilva

Boring Location: _____ **Hammer:** _____ **Other:** _____

Depth (ft)	Sample ID	Depth of Change (ft)	VISUAL-MANUAL MATERIAL DESCRIPTION	PID (parts per million)	COMMENTS (e.g., Native, core run, RQD, % recovered)
1		0.25	Asphalt	0	possible background
		0.75	Asphalt Subbase gravel	0	
2			F brown SAND ; some silt ; little SR-SA gravel (limestone & red sandstone); trace clay (little placity) ; moist	0	
3				0	
4				0	
5				0	
6				0	
7				0	
8				0.1	
9		8.83		0	
		9.5	Start of weathered bedrock between 8.83 and 9-ft bgs	0	
10			Apparent competent Bedrock	0	
11			Augered to approximately 11-ft bgs		
12					
13					
14					
15					
16					
17					
18					
19					
20					

WATER LEVEL DATA			DEPTH (FT)			ADDITIONAL NOTES: Observations taken from auger cuttings. YS-MW-2023-01 monitoring well installed with 8-ft of screen and 2.62-ft of riser. Well installed with flush mount road box.
Date	Time	Elapsed Time	BOTTOM OF CASING	BOTTOM OF BORING	GROUNDWATER ENCOUNTERED	
7/17/2023	1120	45 minutes	11	11	6.52	

GENERAL NOTES

1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL.

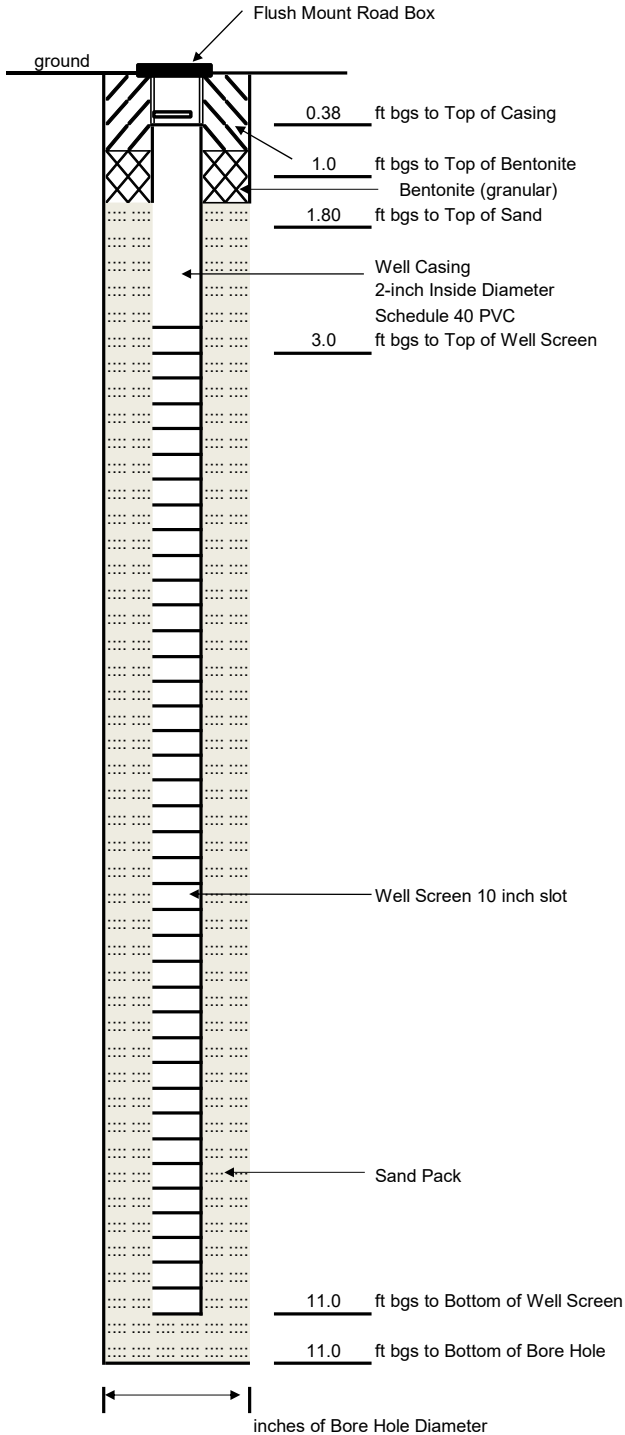
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER

BGS = Below Ground Surface and = 35 - 50% C = Coarse R = Rounded
 NA = Not Applicable some = 20 - 35% M = Medium A = Angular
 BC = Blow counts per 6" sampler little = 10 - 20% F = Fine SR = Subrounded
 NR = No Sample Recovery trace = 1 - 10% VF = Very Fine SA = Subangular

BORING: YS-MW-2023-01

WELL CONSTRUCTION LOG

Well ID.: **YS-MW-2023-01**



Project: Phase II ESA - 42 York Street

Address: 42 York Street

Town/City: City of Rochester **State:** New York

Project No. 2230119 **County:** Monroe

Installation Date: July 17, 2023

Drilling Method: Hollow Stem Auger

Drilling Contractor: LaBella ENV, LLC **Driller:** M. Pepe

Drill Rig: Geoprobe 7822DT

Drilling Fluid: None

Datum: NAVD 88 **Elevation:** 534.74 **ft TOC**

Well Development Information

Finished with protective flush mount well cover, j-plug

Turbid at first, clear after three gallons of water purged, no odors

Static Water Level: 6.52 **feet from top of casing/ground/other**

Fluid Lost During Drilling: None **gallons**

Water Removed During Development: ~5 **gallons**

Date(s) of Development: July 19, 2023

Purging Method: Submersible pump **Sampling Method:** Low Flow (Bladder)

Well Cover Size/Tools Needed to Open: Socket Wrench 5/16"

Notes: ft = feet, bgs = below the ground surface

Weathered bedrock encountered between 8.83ft to 9ft bgs.

8ft of screen was installed.

Oversight By: LaBella Associates (A. daSilva)

Development By : Lu Engineers

Groundwater Development Field Record

 Project Name 42 York St.
 Location ID YS-MW-2023-01
 Activity Time 9:15am

 Field Sample ID N/A
 Sample Time N/A

 Job # 4262
 Sampling Event # __
 Date 07/19/2023

SAMPLING NOTES

 Initial Depth to Water 5.71 feet Measurement Point TOR
 Final Depth to Water Dry feet Well Depth 10.51 feet
 Screen Length 8 feet Pump Intake Depth _____
 Total Volume Purged 5 gallons PID Well Head _____

 Well Diameter 2"
 Well Integrity:
 Cap Yes
 Casing Yes
 Locked Yes
 Collar Yes

[purge volume (milliliters per minute) x time duration (minutes) x 0.00026 gal/milliliter]
 Volume of Water in casing – 2" diameter = 0.163 gallons per foot of depth
 One well volume = 0.78 gallons, Three well volumes = 2.35 gallons

PURGE DATA

Time	Depth to Water (ft)	Purge Rate (ml/min)	Temp. (deg. C)	pH (units)	Dissolved O2 (mg/L)	Turbidity (NTU)	Cond. (mS/cm)	ORP (mV)	Comments
Parameters were not collected as per the QAPP Subtask 1.2									

Purge Observations: Turbid at first, clear after 3-gallons of water purged, development ceased after 5-gallons purged, at which point well was completely dry, no odors.
 Purge Water Containerized: Yes, new drum provided by client.

EQUIPMENT DOCUMENTATION

 Type of Pump: Submersible Pump
 Type of Tubing: 1/4" HDPE
 Type of Water Quality Meter: N/A

 Calibrated: N/A

ANALYTICAL PARAMETERS

Parameter	Volumes	Sample Collected

LOCATION NOTES

 Signature: Klajdi Macolli
 Checked By: _____

Groundwater Sampling Field Record

Project Name 42 York St. Job # 4262
 Location ID YS-MW-2023-01 Field Sample ID YS-MW-2023-01 072623 Sampling Event # -
 Activity Time 9:50 Sample Time 10:45 Date 7/26/23

SAMPLING NOTES

Initial Depth to Water 4.90 feet Measurement Point TOR Well Diameter 2"
 Final Depth to Water 5.10 feet Well Depth 10.61 feet Well Integrity: _____
 Screen Length _____ feet Pump Intake Depth ~ 7 feet Cap
 Total Volume Purged ~ 1 gallons PID Well Head _____ Casing
 [purge volume (milliliters per minute) x time duration (minutes) x 0.00026 gal/milliliter] Locked
 Volume of Water in casing - 2" diameter = 0.163 gallons per foot of depth, 4" diameter = 0.653 gallons per foot of depth Collar

PURGE DATA

Time	Depth to Water (ft)	Purge Rate (ml/min)	Temp. (deg. C)	pH (units)	Dissolved O2 (mg/L)	Turbidity (NTU)	Cond. (mS/cm)	ORP (mV)	Comments
9:55	4.90		20.5	7.54	6.62	163.41	0.681	125.9	
10:00	4.90		21.1	7.51	6.89	149.67	0.678	131.8	
10:05	5.10		21.2	7.52	6.99	140.51	0.681	134.2	
10:10	5.10		21.4	7.48	7.23	91.74	0.679	139.3	
10:15	5.10		21.3	7.44	7.28	81.61	0.677	140.1	
10:20	5.10		21.3	7.42	7.03	48.76	0.675	145.1	
10:25	5.10		21.4	7.41	7.27	24.18	0.674	148.6	
10:30	5.10		21.3	7.40	7.28	22.17	0.671	154.1	
10:35	5.10		21.2	7.40	7.29	18.62	0.674	153.7	
10:40	5.10		21.1	7.43	7.30	16.73	0.678	151.9	

Purge Observations: Clear water
 Purge Water Containerized: Yes

EQUIPMENT DOCUMENTATION

Type of Pump: Bladder Pump
 Type of Tubing: 1/4" HDPE
 Type of Water Quality Meter: YSI ProDSS

Calibrated: Yes

ANALYTICAL PARAMETERS

Parameter	Volumes	Sample Collected
TCL/CP-SVOCs	3x40mL	<input checked="" type="checkbox"/>
TCL/CP-SVOCs	2x1000mL	<input checked="" type="checkbox"/>
TAL Metals	1x250mL	<input checked="" type="checkbox"/>

LOCATION NOTES

Signature: [Signature]
 Checked By: _____

Groundwater Sampling Field Record

 Project Name 42 York St.
 Location ID MW-01
 Activity Time 12:10

 Field Sample ID MW-01 072623
 Sample Time 13:10

 Job # 4262
 Sampling Event #
 Date 7/26/23

SAMPLING NOTES

 Initial Depth to Water 4.89 feet
 Final Depth to Water 5.31 feet
 Screen Length feet
 Total Volume Purged ~3.75 gallons
 Measurement Point TOR
 Well Depth 10.38 feet
 Pump Intake Depth ~7 feet
 PID Well Head

 Well Diameter 2"
 Well Integrity:
 Cap
 Casing
 Locked
 Collar

[purge volume (milliliters per minute) x time duration (minutes) x 0.00026 gal/milliliter]

Volume of Water in casing – 2" diameter = 0.163 gallons per foot of depth, 4" diameter = 0.653 gallons per foot of depth

PURGE DATA

Time	Depth to Water (ft)	Purge Rate (ml/min)	Temp. (deg. C)	pH (units)	Dissolved O2 (mg/L)	Turbidity (NTU)	Cond. (mS/cm)	ORP (mV)	Comments
12:15	4.89		18.3	6.82	2.18	70.60	0.334	126.6	
12:20	4.89		18.2	6.85	2.33	58.16	0.318	123.5	
12:25	4.89		18.1	6.86	2.23	51.17	0.316	121.6	
12:30	5.07		17.9	6.82	1.93	38.65	0.328	119.7	
12:35	5.07		17.9	6.83	1.87	27.15	0.336	117.6	
12:40	5.16		17.9	6.84	1.69	22.09	0.339	117.7	
12:45	5.21		17.8	6.83	1.66	20.61	0.344	117.1	
12:50	5.31		17.9	6.82	1.41	19.06	0.358	114.2	
12:55	5.31		17.8	6.83	1.37	16.19	0.357	113.6	
13:00	5.31		17.9	6.81	1.34	13.06	0.359	112.3	
13:05	5.31		17.9	6.83	1.36	12.01	0.361	111.9	

 Purge Observations: Orange-ish water

 Purge Water Containerized: Yes

EQUIPMENT DOCUMENTATION

 Type of Pump: Bladder Pump
 Type of Tubing: 1/4" HDPE
 Type of Water Quality Meter: YSI ProDSS

 Calibrated: Yes

ANALYTICAL PARAMETERS

Parameter Volumes Sample Collected

TCL/CP-SIVOCs	3x40ml	<input checked="" type="checkbox"/>
TCL/CP-SI SIVOCs	2x1000ml	<input checked="" type="checkbox"/>
TAL Metals	1x250ml	<input checked="" type="checkbox"/>

LOCATION NOTES

 Signature: K. [Signature]
 Checked By:



APPENDIX 3

Photo Log



Fill Layer



DESCRIPTION: TP-01

DATE: 7/11/2023



DESCRIPTION: TP-02 (no urban fill identified)

DATE: 7/11/2023



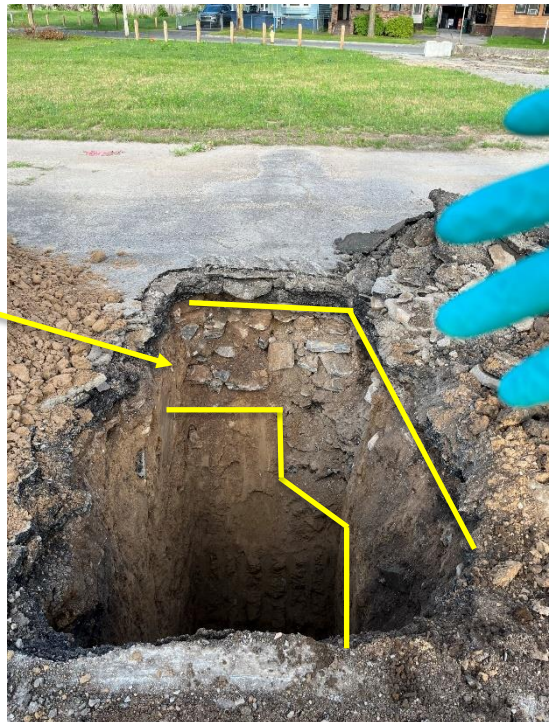
Fill Layer



DESCRIPTION: TP-03

DATE: 7/11/2023

Fill Layer



DESCRIPTION: TP-04

DATE: 7/12/2023



Fill Layer



DESCRIPTION: TP-05

DATE: 7/12/2023

Fill Layer



DESCRIPTION: TP-06

DATE: 7/12/2023



Fill Layer



DESCRIPTION: TP-07

DATE: 7/12/2023

Fill Layer



DESCRIPTION: TP-08 (note: possible urban fill from 2 - 7 feet bgs
Brown F SAND AND URBAN FILL (little brick, little metal) moist

DATE: 7/12/2023



Fill Layer



DESCRIPTION: TP-09

DATE: 7/11/2023



DESCRIPTION: Installation of monitoring well YS-MW-2023-01

DATE: 7/17/2023



APPENDIX 4

CAMP Data

This Appendix Only Available in the Digital Version of the Report



APPENDIX 5

Laboratory Reports

This Appendix Only Available in the Digital Version of the Report



APPENDIX 6

Data Usability Summary Reports (DUSR)

This Appendix Only Available in the Digital Version of the Report



APPENDIX 3

Health and Safety Plan

Site-Specific Health and Safety Plan (HASP)



Project Title:

Remedial Work Plan 42 York Street NYSDEC Spill #2206496

Location:

42 York Street, Rochester, New York 14614

Prepared For:

City of Rochester

LaBella Project No. 2230119

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ATTACHMENTS

APPENDICES

APPENDIX A - Directions to Medical Facility

APPENDIX B - Task Hazard Analysis Forms

APPENDIX C - Daily Tailgate Safety Meeting Form

0.0 HASP Acknowledgment

All LaBella project personnel, contractors, and subcontractors are required to sign the following agreement prior to conducting work:

1. I have read and fully understand the requirements of this site-specific HASP including my individual responsibilities listed above.
2. I agree to abide by the provisions of the HASP and participate in any health and safety meetings or modifications to the HASP criteria during the implementation of work.

Name	Company	Date

1.0 Introduction

The purpose of this Health and Safety Plan (HASP) is to provide guidelines for responding to potential health and safety issues that may be encountered at the project site, located at 42 York Street, Rochester, New York 14614. This HASP only reflects the policies of LaBella Associates D.P.C. and its affiliated company LaBella Environmental, LL, collectively referred to as "LaBella". The requirements of this HASP are applicable to all approved LaBella personnel, contractors and subcontractors at the work site. This document's project specifications are to be consulted for guidance in preventing and quickly abating any threat to human safety or the environment. The provisions of the HASP do not replace or supersede any federal, state or local regulatory requirements.

2.0 Responsibilities

This HASP presents guidelines to minimize the risk of injury to project personnel, and to provide rapid response in the event of injury. The HASP is applicable only to activities of approved LaBella personnel and their authorized visitors specific to this project. The Project Manager shall implement the provisions of this HASP for the duration of the project. It is the responsibility of LaBella employees to follow the requirements of this HASP, and all applicable company safety procedures.

3.0 Daily Pre-Job Safety Meetings

Prior to the beginning of work each day the Field Supervisor/Foreman or on-site Project Manager will review upcoming daily job requirements, anticipated hazards and hazard control measures with the project team members. At this meeting information such as personal protective equipment, site conditions, emergency procedures, and other applicable topics may be addressed. A copy of the **Daily Pre-Job Safety Tailgate/Toolbox Meeting Form** is attached to this HASP.

4.0 Site Information

Project Name:	Remedial Work Plan 42 York Street NYSDEC Spill #2206496
LaBella Project No.:	2230119
Project Location:	42 York Street, Rochester, New York 14614
Current Use of Project Location:	Vacant Lot
Uses of Surrounding Areas (Res Vacant Land, Commercial, etc.):	Residential and Vacant Lots
Proposed Date(s) of Field Activity - Start:	2024-10-07

Proposed Date(s) of Field Activity - End:	2024-12-20
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5.0 Scope of Work

The proposed field work covered under this HASP includes the following:

- Excavation using an excavator and related heavy equipment. Off-site transport/disposal of non-hazardous solid waste (soil/fill). Refer to parent document for additional details.

6.0 Emergency Information

The personnel and emergency response contacts associated with the proposed scope of work are presented below and are to be posted onsite during all field activities. The Site Safety Officer (SSO) is the primary authority for directing site operations and relaying communications under emergency conditions. During the SSO's absence, the Project Manager or Site Supervisor will lead emergency operations.

Project Personnel		
Contact	Name	Phone
LaBella Project Manager	Drew Brantner	585-287-9089
LaBella Site Supervisor	TBD	TBD
Corporate Safety Manager	Catherine Monian	845-486-1557
Environmental Division Safety Program Manager	Tim Ruddy	315.440.5125
Site Safety Officer	TBD	TBD
Site Contact	TBD	TBD
Emergency Personnel including Police and Fire Dept and Ambulance - Dial 911		
Hospital- see Hospital Route Section below for directions		
Poison Control		800-336-6997
NYSDEC Spill Response Hotline		800-457-7362

First Aid

A First Aid Kit will be located as follows: LaBella Company Vehicle or Personal Vehicle The injured person may be transported to a trained medical center for further examination and treatment. The preferred transport method is a professional emergency transportation service; however, if this option is not readily available or would result in excessive delay, other transport is authorized.

Under no circumstances should an injured person transport themselves to a medical facility for treatment, no matter how minor the injury may appear.

Incident Reporting

Employees shall report all incidents and injuries to their supervisor as soon as possible, including those involving employees operating vehicles and other equipment. All reporting procedures contained in LaBella Safety Policy 1.22 must be followed.

During emergencies employees should seek medical care immediately. When contacting their Supervisor/Safety Manager/HR, employees should discuss medical care options. If an employee is asked by medical personnel for a worker's compensation number they should tell them that LaBella should be billed directly.

When emergency medical care is not imminent, employees shall immediately report events to their immediate Supervisor, the Safety Manager and Human Resources, and participate in the investigation process as well as the corrective action process, as needed. An Accident-Incident-Near Miss-Hazard Form must be submitted online or by e-mail to the Supervisor, Safety Manager and HR as soon as possible but no later than 24 hours after the event. The Form can be found on LaBella's intranet under "Operations".

7.0 Potential Health and Safety Hazards and Controls

This section lists potential health and safety hazards that project personnel may encounter at the project site and actions to be implemented by approved personnel to control and reduce the associated risk to health and safety. This is not intended to be a complete listing of any and all potential health and safety hazards. New or different hazards may be encountered as site environmental and site work conditions change. The suggested actions to be taken under this plan are not to be substituted for good judgment on the part of project personnel. At all times, the Site Safety Officer has responsibility for site safety and their instructions must be followed.

<i>Physical Hazards</i>		
Work Action or Condition	Potential Safety Hazard	Controls (including PPE)
Concrete Dust	Inhalation of respirable silica dust	<ul style="list-style-type: none"> • Use wet suppression systems to minimize dust. • Do not use compressed air to clean surfaces. • Wear appropriate PPE and refer to exposure control plan for silica.
Electrical - Overhead Power Lines	Struck by injury, Electric shock and electrocution	<p>Overhead power lines pose a danger of shock or electrocution if the power line is contacted during site operations. The following hazard control measures will be applied:</p> <ul style="list-style-type: none"> • Equipment should not come within at least 10-feet of power lines to avoid arcing. • Prior to conducting work in areas where overhead lines could be impacted, the appropriate utility company will be notified and information will be obtained regarding the line voltage(s) and the minimum separation distance necessary to create a safe-work environment. • If work may come close to the minimum separation distance ask the utility company if the lines can be shut down for the duration of the work period. If this cannot be done, ask them if they can put a rubber cover over the line or if the minimum clearance distance can be reduced. • Use paint to mark a line on the ground underneath the overhead lines to help project staff stay aware of the hazard. • Use smaller equipment. • Use grounding cables. • Ensure equipment operators are properly trained on the equipment and know exactly where the overhead lines are. • Ensure all site staff are aware of the hazard and stay clear of the work zone (which should be demarcated as best as possible). Employees who are too close to equipment which becomes electrified can ALSO be electrocuted.
Excavations and Trenches	Injury from fall into or cave-in of trench/excavation. Asphyxiation, engulfment, or	An open excavation or trench may be present during site activity, or could be present during demolition or remediation activities. No Labella employees should enter a trench or excavation unless authorized to by the designated Competent Person. During heavy precipitation,

	explosion (if pipe bursts)	<p>excessive runoff may create slippery surfaces and also weaken the excavation sidewalls making the excavation more susceptible to collapse. The following hazard control measures will be applied:</p> <ul style="list-style-type: none"> • All materials must be placed greater than 2 feet from the edge of the trench and LaBella employees should remain at least 2-feet from the edge of any excavation or trench. • LaBella employees are not to enter excavations greater than 4-feet in depth unless they have received appropriate training, stabilization measures are in place and a competent person has determined that the conditions are safe. • Any samples must be collected from the equipment bucket or from the spoils pile.
Hand Tools	Physical injury	<ul style="list-style-type: none"> • Do not use a tool if you have not been trained. Inspect tool before use and do not use damaged tools. • Maintain tools in good condition and follow manufacturers' instructions. • Wear gloves, safety glasses and appropriate PPE /apparel, avoiding loose clothing; secure long hair. • When using a cutting tool hold its handle firmly and cut away from your body, never towards it. • If working on a ladder or scaffold raise and lower tools using a bucket and hand line; never carry tools in a way that prevents using both hands on a ladder (maintain three points of contact)
Heavy Equipment - Working Near	Struck by, Caught in between, Causing an obstruction on existing roadway, Rollaway, and hearing damage.	<p>Working near heavy equipment presents struck-by and caught-in or in-between risks. Heavy equipment can also rollaway or obstruct roadways, limiting visibility. The following hazard control measures will be applied:</p> <ul style="list-style-type: none"> • Maintain 360 degrees of awareness of your surroundings. • Meet the Operator, discuss work operations, and stay in line of sight. • Wear high visibility clothing (outer layer), hard hat, safety glasses, work boots. • Stand in safe zone away from blind areas. Never walk behind or to the side of heavy equipment without the operator's knowledge. Have an escape plan. • Stay out of the swing zone of heavy equipment

		<p>such as excavators or traditional auger rigs. The swing zone is defined as an entire 360 degree circle equipment may move within as measured from a central location point.</p> <ul style="list-style-type: none"> • Only approach drill rig after auger has stopped rotating and the operator has given the OK for you to approach to collect a sample. • Wear hearing protection when working near heavy or moving equipment.
High Crime Area	Potential theft or risk of safety	<p>Working in high crime areas requires vigilance to protect personnel and project assets. The following hazard control measures will be applied:</p> <ul style="list-style-type: none"> • Workers will be accompanied by a site representative or another employee. • Workers should stay in well-lit areas and maintain awareness of their surroundings. • If significant risk is evident, vacate the area.
Hot Weather & Sun, Other Heat Hazards	Prickly Heat (Heat rash), Heat Cramps, Heat Exhaustion, Heat Fatigue, Heat Collapse, Heat Stroke, Sunburn	<p>Environmental heat hazards, whether indoors or outdoors, present physical injury risks. Exercise caution when working in hot temperatures or around hot tar or other materials, hot ovens or other equipment, heat absorbing surfaces such as roofs and roads, and reflective surfaces such as water or metal. The following hazard control measures will be applied:</p> <ul style="list-style-type: none"> • Have sunscreen available for ultraviolet protection on sunny days. • Have water or electrolyte drinks for dehydration. • Check the weather and adjust work schedules if heat is excessive. Work early or later in day. • Perform work during cooler hours of the day or at night if adequate lighting can be provided. • Utilize shelter (air-conditioned, if possible) or shaded areas to protect personnel during rest periods. • Use cooling devices such as fans and water misters. • Allow workers to take breaks in air-conditioned vehicles.
Parking Vehicle	Struck by, caught in between, casing an obstruction on existing roadway. Fire from plants under hot exhaust	<ul style="list-style-type: none"> • Workers will park far enough off the edge of the road to stay well clear of traffic. • Put on hi-visibility vest before exiting parked car. • Leave Field Card on dashboard. • Use appropriate number of cones to mark for oncoming traffic as needed.

		<ul style="list-style-type: none"> • Do not park on/in flammable vegetation. • Keys stay on field person.
Power Tools	Injury from improper use Electrical shock and electrocution	<ul style="list-style-type: none"> • Unplug power tools when not in use. • Do not use a tool if you have not been trained. Inspect tool and cord before use and do not use damaged tools. • Maintain tools in good condition and follow manufacturers' instructions. • Wear gloves, safety glasses and appropriate PPE /apparel, avoiding loose clothing; secure long hair. • Never remove a safety guard when a tool is being used. • Only plug electric tools into a grounded receptacle with a GFCI. Stop using tool if slight shock or tingling is felt. • Secure work with clamps to have both hands free to use the tool. • Keep power tool cords away from heat, oil and sharp edges. • Tag all damaged tools with "Do Not Use".
Slip-Trip-Fall	Injury	<ul style="list-style-type: none"> • Reduce and avoid slippery (wet, icy, oily, muddy, etc.) surfaces. • Workers will watch where they step and wear proper footwear. • Keep work areas free of obstructions and debris.
Underground Utilities	Damage to utility infrastructure, Electrocution, Explosion	<ul style="list-style-type: none"> • Utility marking is needed for this project. • Prior to the commencement of ground intrusive activities, underground utilities will be located by a third-party locator. • Workers will not stand within 20-feet of any active excavations or boreholes if not actively working in those areas.

<i>Biological and Environmental Hazards</i>		
Work Action or Condition	Potential Safety Hazard	Controls (including PPE)

<i>Ergonomic Hazards</i>		
Work Action or Condition	Potential Safety Hazard	Controls (including PPE)
Lifting Heavy Objects	Injury from Improper Lifting/Lifting weights that are too heavy	<ul style="list-style-type: none"> • When lifting heavy objects, keep the load close to the body and use the leg muscles instead of the back muscles to perform lifting tasks. • Do not attempt to lift large, heavy (especially over 50-lbs), or awkwardly shaped objects without assistance from another employee or from a manual lifting devise.
Noise (Loud, Sustained)	Hearing Damage	<ul style="list-style-type: none"> • Ear protection will be worn at all times when personnel are within 20-feet of operating equipment or when noise level becomes consistently loud enough to have to raise voice to communicate with someone. • Hearing protection will also be worn in the vicinity of generators, concrete cutters, and any other high noise emitting equipment.

<i>Chemical Hazards (General)</i>		
Work Action or Condition	Potential Safety Hazard	Controls (including PPE)
Chemical Exposure - Heavy Metals	<i>Contaminants identified in testing locations at the Site include low-level heavy metals, primarily associated with Site contamination. Heavy metal-impacted media including fill material may be encountered during subsurface activities at the project work site.</i>	<p>The presence of heavy metals in site media may be difficult to ascertain in the field. Heavy metal concentrations at this site are not anticipated to exceed PELs. The following hazard control measures will be applied, however:</p> <ul style="list-style-type: none"> • Workers shall wear appropriate PPE and follow listed decontamination procedures to prevent exposures. Refer to the relevant sections of this HASP for more detail regarding PPE and decontamination procedures.
Chemical Exposure - Semi-Volatile Organic	<i>Contaminants identified in testing locations at the</i>	The presence of SVOCs in site media may be detected by their odor and monitoring instrumentation. SVOC concentrations at this Site

<p>Compounds (SVOC)</p>	<p><i>Site include SVOCs. SVOC-impacted media including fill material may be encountered during subsurface activities at the project work site.</i></p>	<p>are not anticipated to exceed PELs. The following hazard control measures will be applied, however:</p> <ul style="list-style-type: none"> • Workers should be wearing appropriate PPE and following listed decontamination procedures to prevent exposures. Refer to the relevant sections of this HASP for more detail regarding PPE and decontamination procedures.
<p>Chemical Exposure - Volatile Organic Compounds (VOC)</p>	<p><i>Contaminants identified in testing locations at the Site include various volatile organic compounds (VOCs), primarily VOCs associated with Site contamination. Volatile organic vapors may be encountered during subsurface activities at the project work site. Inhalation of high concentrations of volatile organic vapors can cause headache, stupor, drowsiness, confusion and other health effects. Skin contact can cause irritation, chemical burn, or dermatitis. Relevant Safety Data Sheets are included as Appendix 1.</i></p>	<p>Volatile Organic Compound (VOC) gases may be emitted from a number of materials and products. The presence of organic vapors may be detected by their odor and by monitoring instrumentation and can lead to physical harm. VOC concentrations at this Site are not anticipated to exceed PELs. The following hazard control measures will be applied, however:</p> <ul style="list-style-type: none"> • Workers should be wearing appropriate PPE, following listed decontamination procedures and be periodically screening the work zone to prevent against and evaluate for unexpected exposures. Refer to the relevant sections of this HASP for more detail regarding PPE, decontamination procedures and work zone screening.
<p>Lead</p>	<p><i>Injury, Illness</i></p>	<ul style="list-style-type: none"> • Lead exposure, which occurs most commonly by breathing in particles, can result in long term physical illness and disability (See 4.04 LEAD SAFETY POLICY in Labella's Safety Manual for information on Exposure Controls).

<p>Sample Collection - Soil or Groundwater</p>	<p><i>Exposure to contaminants. Hand injury from cutting, crushing, tool or glass breakage. Back strain from lifting cooler.</i></p>	<ul style="list-style-type: none"> • When collecting samples, workers will utilize nitrile gloves, safety glasses or goggles. If material being sampled potentially contains fill or other sharp material, use a stainless steel spoon (or similar) as a tool to collect the sample. Any such tools should be dedicated or properly decontaminated between samples. • When lifting sample coolers, workers will use proper lifting techniques and get assistance when possible, especially for containers heavier than 50 lbs.
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8.0 Personal Protective Equipment (PPE)

All site workers will have appropriate training as identified in Section 7.0. Training includes the identification of PPE necessary for various tasks; how to don, doff, adjust, and wear PPE; limitations of PPE; and proper care, inspection, testing, maintenance, useful life, storage, and disposal of the PPE. PPE will be inspected on a regular basis.

<p>Level D: A work uniform affording minimal protection, used for nuisance contamination, only.</p>	<ul style="list-style-type: none"> • Coveralls or long-sleeves and pants • Gloves • Nitrile sampling gloves (as needed) • Boots/shoes, chemical-resistant steel toe and shank • Safety glasses or chemical splash goggles • Hard hat
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9.0 Employee Training

All workers and other personnel shall receive appropriate training prior to engaging in site activities. All workers must recognize and understand the potential hazards to health and safety that are associated with the proposed scope of work and must be thoroughly familiar with programs and procedures contained in this Safety Plan.

The following training levels were determined to be needed:

- OSHA 40 Hour - HAZWOPER

10.0 Exposure Monitoring

No - VOC Exposure Monitoring not required or applicable

11.0 Site Control

No - Contaminant Exclusion or Reduction zone not required or applicable at the site.

12.0 Recordkeeping

An electronic or hardcopy version of this HASP will be present at the Site during all field work activities. Copies of field logs, including daily pre-job safety meeting logs, will be filed by LaBella and available for the duration of the project.

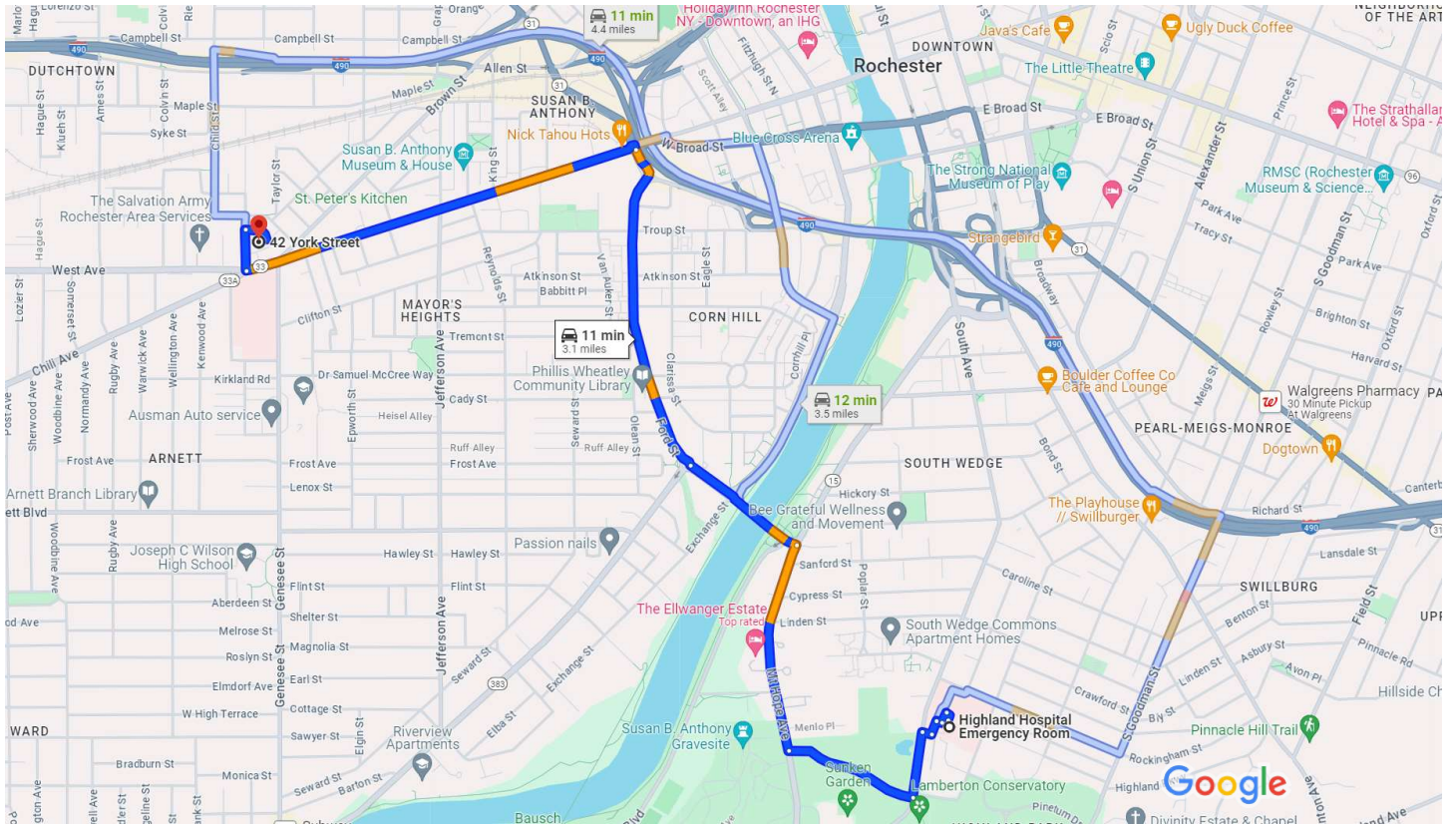
Employees will be able to provide physical or electronic copies of required training certificates.

Incident reporting will be completed in accordance with LaBella policies.



APPENDIX A

Directions to Nearest Medical Facility



Map data ©2024 Google 1000 ft

Highland Hospital Emergency Room
 1000 South Ave, Rochester, NY 14620

Continue to South Ave

- 3 min (0.1 mi)
- ↑ 1. Head north toward Bellevue Dr
 - 112 ft
- ↶ 2. Turn left
 - 131 ft
- ↷ 3. Turn right
 - 66 ft
- ↶ 4. Turn left
 - 174 ft
- ↷ 5. Turn right onto Bellevue Dr
 - 112 ft
- ↶ 6. Turn left onto South Ave
 - 34 sec (0.1 mi)

➤ 7. Turn right onto Robinson Dr
_____ 59 sec (0.3 mi)

Take Ford St and NY-33 W/W Main St to York St

_____ 7 min (2.4 mi)

➤ 8. Turn right onto NY-15 N/Mt Hope Ave
_____ 0.5 mi

↶ 9. Turn left onto Ford St
_____ 0.3 mi

🔄 10. At the traffic circle, continue straight to stay on Ford St
_____ 0.7 mi

↶ 11. Turn left onto NY-33 W/W Main St
_____ 0.9 mi

Continue on York St to your destination

_____ 35 sec (0.1 mi)

➤ 12. Turn right onto York St

_____ 479 ft

➤ 13. Turn right
📘 Destination will be on the right
_____ 312 ft

42 York St



APPENDIX B

Task Hazard Analysis Forms

6.02 TASK HAZARD ANALYSIS (THA) FORM

THA Title:		THA ID #:	Date: <input type="checkbox"/> New <input type="checkbox"/> Revised
Work Activity:		Risk Code (Table Page 2):	Division:
Person Preparing THA:		Person Assisting with THA:	
Sequence of Steps or Activities	Materials, Equipment & Tools Needed	Hazards	Recommended Controls Measures / PPE/ Training
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			

Risk Assessment Codes (RACs) Likelihood & Severity Classification			
Likelihood of Harm (People, Environment, Facility)	Severity of Harm/Consequences (People, Environment, Facility, Supply Chain Disruption, Brand Impact)		
	Slight Harm	Moderate Harm	Extreme Harm
Very Unlikely	Very low risk	Very low risk	High risk
Unlikely	Very low risk	Medium risk	Very high risk
Likely	Low risk	Medium risk	Very high risk
Very Likely	Low risk	High risk	Very high risk
Definitions			
<u>Likelihood of Harm Categories:</u> -Very Unlikely: Will not occur except in rare instances under certain conditions -Unlikely: Typically would not occur -Likely: May occur on a regular basis -Very Likely: Will occur in most instances		<u>Severity of Harm Categories:</u> -Slight harm: Only first aid required -Moderate harm: Injury or illness resulting in inability to work for a short period of time -Extreme harm: Death or serious injury or illness resulting in inability to work indefinitely	

PREPARATION SIGN OFF			
Role	Name	Signature	Date
Preparer			
Reviewer with Relevant Task Technical Experience or Safety Expertise			
Safety Manager – Needed for High Risk or Very High Risk THAs			

ACKNOWLEDGEMENT IF THA IS USED AS A TRAINING RESOURCE			
By signing I am indicating that I have read and understand the contents of this Task Hazard Assessment and the controls required to mitigate the risks from identified hazards.			
Name	Signature	Company	Date



APPENDIX C

Daily Tailgate Safety Meeting Form

6.08 PRE-JOB SAFETY TAILGATE/TOOLBOX MEETING FORM

Date		Time	
Location or Address		Temperature	
Project Number		Humidity	
Conducted by		Conditions	
Were all workers reminded that COVID is still prevalent and that appropriate measures should be taking to prevent infection of themselves and others?			Yes <input type="checkbox"/> No <input type="checkbox"/>

911	If 911 is unavailable at this location, please state the procedure for reporting emergencies _____
------------	--

List Safety Topic of Discussion and/or Any Specific Hazards for the Work Being Performed Today	
1	
2	
3	
4	
5	
6	
7	

List Control Measures for Each Specific Hazard Listed Above	
1	
2	
3	
4	
5	
6	
7	

PLEASE SIGN THE BACK OF THIS SHEET

The presenter and all attendees shall print and sign in the appropriate areas on the back of this sheet



By signing, you declare that you understand the information presented in today's meeting, and that you have had the opportunity to ask questions and to clarify any uncertainty regarding such information.

All Visitors and Contractors Must Print Their Company Name

Name	Signature	Company





APPENDIX 4

NYSDOH Generic CAMP

Appendix 1A

New York State Department of Health Generic Community Air Monitoring Plan

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., off-site receptors including residences and businesses and on-site workers not directly involved with the subject work activities) from potential airborne contaminant releases as a direct result of investigative and remedial work activities. The action levels 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 off-site 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

Volatile organic compounds (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 $150 \text{ mcg}/\text{m}^3$ 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 $150 \text{ mcg}/\text{m}^3$ 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 $150 \text{ mcg}/\text{m}^3$ 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.

December 2009



APPENDIX 5

Quality Assurance Project Plan

Quality Assurance Project Plan

42 York Street

Remedial Work Plan

Location:

42 York Street
Rochester, NY 14611
USEPA Grant No. 96242500

Prepared for:

City of Rochester
30 Church Street
Rochester, NY 14614

LaBella Project No. 2230119

August 2024

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Section 4	Project Timeline	Template #4
Measurement/Data Acquisition		
Section 5	Sampling and Analytical Requirements	Templates #5a, #5b, #5c, #5d, #5e
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Section 7	Field Equipment Calibration/Corrective Action	Template #7
Section 8	Laboratory Equipment Calibration/Corrective Action	Template #8
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Appendices:

Appendix A – Staff Resumes

Appendix B – Field Logs

Appendix C – Laboratory Quality Assurance Manual

Appendix D – Laboratory Standard Operating Procedures (SOPs)

Appendix E – LaBella SOPs

**Brownfields QAPP Template #1
Title and Approval Page**

Title: Quality Assurance Project Plan (QAPP) 42 York Street – Remedial Work Plan

Project Name/Property Name: 42 York Street

Property/Site Location: 42 York Street, Rochester, New York 14614

Revision Number: 0

Revision Date: 8/19/24

Brownfields Cooperative Agreement Number: 96242500

City of Rochester

Brownfields Recipient

Drew Brantner, LaBella Associates, D.P.C.

Preparer's Name and Organizational Affiliation

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Preparer's Address, Telephone Number, and E-mail Address

6/3/2024

Preparation Date (Day/Month/Year)

Brownfields Recipient Program Manager:

Signature

Rick Rynski, City of Rochester, rick.rynski@cityofrochester.gov

Printed Name/Organization/Date

Environmental Consultant Quality Assurance Officer:
(QAO)

Signature

Dan Noll, LaBella Associates, D.P.C.

Printed Name/Organization/Date

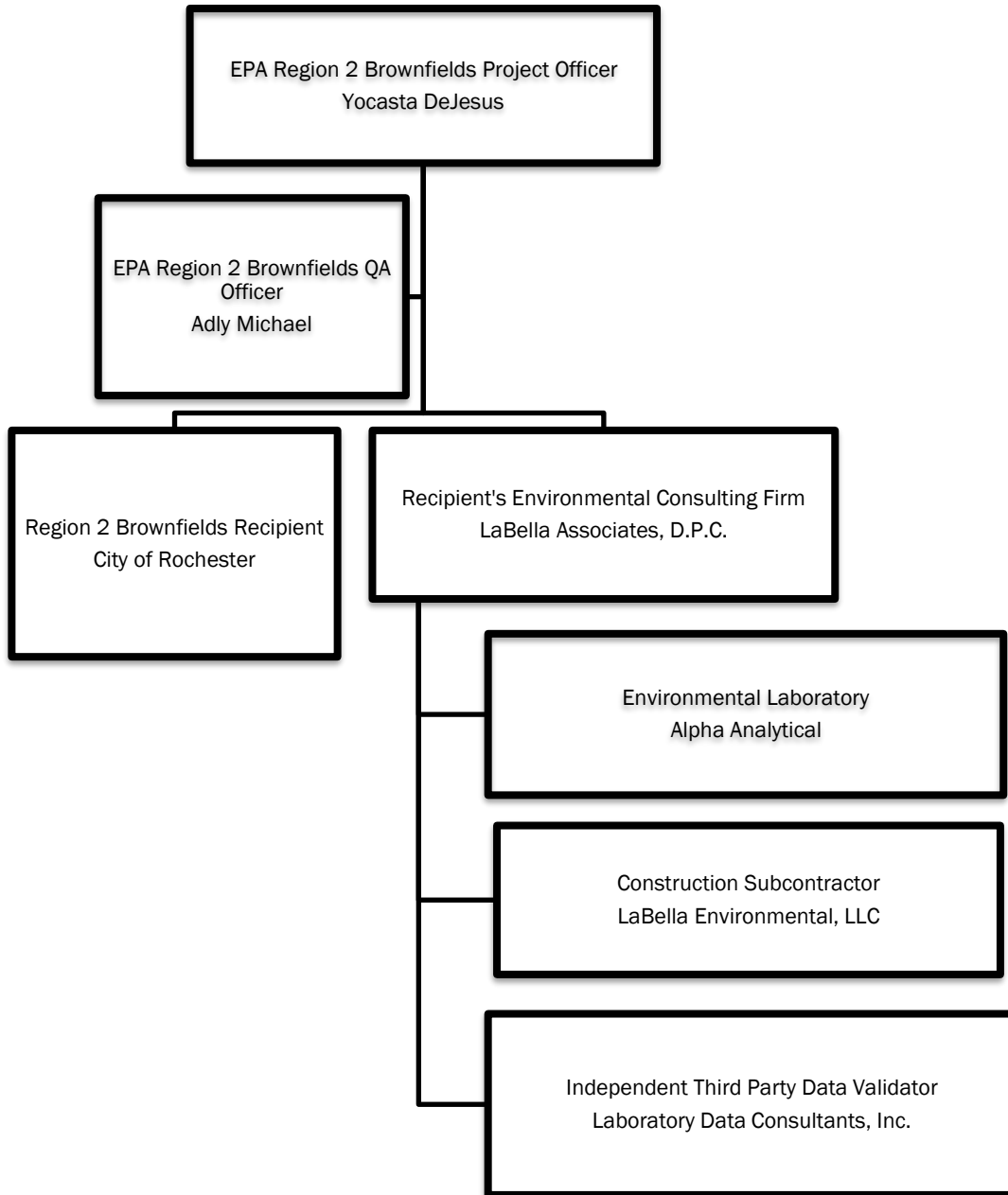
EPA Region 2 Brownfields Project Officer:

Signature

Yocasta DeJesus, USEPA

Printed Name/Organization/Date

Brownfields QAPP Template #2a Project Organizational Chart



**Brownfields QAPP Template #2b
Personnel Responsibilities**

Name	Title	Telephone Number	Organizational Affiliation	Responsibilities
Drew Brantner	Project Manager	585-287-9089	LaBella Associates, D.P.C.	Coordinate planning, sampling, reporting tasks, client liaison, project oversight, coordinate sampling and reporting
Ann Barber	Assistant Project Manager and Engineer	585-295-6289	LaBella Associates, D.P.C.	Client liaison and assist with planning and reporting.
Dan Noll	LaBella Quality Assurance Officer (QAO)	585-295-6611	LaBella Associates, D.P.C.	Provide input on quality of technical work completed
Katherine Truong	Project Coordinator	585-402-7049	LaBella Associates, D.P.C.	Assist the project manager as needed and coordinate with field staff and subcontractors
Alex daSilva	Environmental Geologist	585-295-6268	LaBella Associates, D.P.C.	Perform oversight and soil sampling in accordance with QAPP, project reporting
Rick Rynski	Grant Recipient	315-338-0393	City of Rochester	Manage grant budget and schedule, select and coordinate consultants
Yocasta DeJesus	EPA Brownfields Project Officer (BPO)	212-637-4340	EPA Region 2	Provide grant administration and technical assistance as needed
Adly Michael	EPA Brownfields Quality Assurance Officer (QAO)	732-906-6161	EPA Region 2	Provide input on quality of technical work completed
Melissa Deyo	Analytical Laboratory	716-427-5229	Alpha Analytical	Analyze environmental samples collected during field portion of the projects
Stella Cuenco <i>et. al.</i>	Third Party Data Validator	760-827-1100	Laboratory Data Consultants, Inc.	Assess the validity of analytical data generated by the laboratory
Steve Rinker	Environmental Construction Subcontractor	585-303-9403	LaBella Environmental, LLC	Complete the remedial action (excavation, etc.)

**Brownfields QAPP Template #3a
Problem Definition/Project Description**

PROBLEM DEFINITION

The Site is located within the Bull's Head BOA and associated with the City of Rochester's Bull's Head Revitalization Project. Remedial efforts targeting the removal of fill material and the associated SVOCs and metals impacts are necessary to provide a clean site that promotes redevelopment, in accordance with the Bull's Head Revitalization Plan.

PROJECT DESCRIPTION

Site Location and Description

The Site is comprised of one approximate 0.48-acre parcel (SBL #120.42-2-72.001) located at 42 York Street, in the City of Rochester, Monroe County, New York. Refer to Figure 1 for the approximate Site location (map) and Figure 2 for a local site plan. The Site is within the Bull's Head redevelopment area and is currently an unused paved parking lot. The Site is located in an urban setting.

Site History

The Site appeared to be first developed with several residential dwellings and sheds/barns on portions of the parcel from 1892 to at least 1935.

On aerial photographs dated 1988, 1993, and 2003, approximately 15 vehicles are parked on the Site. In addition, apparent dark staining and miscellaneous items (which may be indicative of debris) appear to be located throughout the Site. The staining and debris on the Site may also be indicative of current or former industrial/manufacturing use of the property or effects from surrounding properties. Potential concerns associated with an industrial/manufacturing use of a property include the contamination of soil and/or groundwater if leaks/spills and/or improper handling/disposal of hazardous materials, petroleum products, and/or hazardous wastes has occurred.

Remedial Action

The remedy will consist of the excavation and off-Site disposal of soil-fill material from the Site. For further information, refer to the body of the RWP.

Confirmatory/Documentation Sampling

Prior to backfilling the excavation, confirmatory/documentation samples will be collected from the sidewalls and portions of the bottom of the excavation with soil. Documentation samples will refer to samples collected from the excavation that are not final endpoint samples (i.e., additional soil must be removed to meet SCOs/SCLs). Confirmatory samples will refer to endpoint samples that meet SCOs/SCLs. Confirmatory/ documentation samples will not be collected from the bottom of the excavation in areas of exposed bedrock.

The confirmatory/documentation samples will be collected in accordance with DER-10; one (1) sidewall confirmatory/documentation sample will be collected for every 40 linear feet of excavation perimeter, and one (1) bottom confirmatory/documentation sample will be collected for every 1,600-sq.ft. of excavation bottom area. While it is currently anticipated that some of the excavation bottom will consist of bedrock, the bottom confirmatory/

documentation sample quantities will be calculated based on the area of exposed soils at the excavation bottom. If minimal soil remains at the bottom of the excavation (less than 6 inches), it will be removed to bedrock.

Since the perimeter of the remedial excavation is currently anticipated to measure approximately 700-ft, up to eighteen (18) sidewall confirmatory/documentation soil samples will be collected. While the exact area of exposed soil at the bottom of the excavation is unknown, it is estimated that thirteen (13) bottom confirmatory/documentation soil samples will be collected. Quantities of confirmatory/documentation samples are subject to change based on actual excavation perimeter/ area. Each confirmatory/documentation soil sample will be submitted for laboratory analysis of the following:

- NYCRR Part 375 and CP-51 List VOCs using USPA Method 8260
- NYCRR Part 375 and CP-51 List SVOCs using USEPA Method 8270
- TAL Metals using USEPA Method 6010/7470

A blind duplicate and matrix spike/matrix spike duplicate sample will be collected from the confirmatory/documentation samples at a rate of one (1) per twenty (20) samples, respectively.

Samples will be sent under standard Chain of Custody procedures to a NYSDOH ELAP-certified laboratory. To reduce the amount of time the excavation will remain open without backfill, all confirmatory samples will be submitted with a rush turnaround time of approximately 3 to 5 business days.

Reporting

At the conclusion of the project, a Remedial Construction/Closure Report (RCCR) will be prepared for the Site. This report will document all remedial actions implemented, and include the following (at minimum):

1. Project background and pertinent history;
2. Remedial objectives;
3. Summary of all remedial work performed;
4. Field documentation in a field notebook and daily summaries;
5. Scaled drawings showing the Site location and layout, previous testing locations, confirmatory soil sample locations, and actual limits of excavation(s);
6. All quantities (tonnage) of all media disposed of;
7. Tabulated data for analytical results comparing to applicable cleanup criteria (i.e., Restricted Residential Use SCOs);
8. Laboratory analytical reports in ASP Category B format;
9. DUSRs;
10. CAMP data;
11. NYSDEC approvals of work plans, requests to import/reuse material, etc.;
12. Imported material documentation including weight tickets, laboratory data etc.;
13. Disposal documentation including weight tickets, landfill approval, laboratory data, etc.; and,
14. Photographs of the work performed with summary and date of each photograph.

Throughout the project, LaBella will keep a record of all remedial excavation limits, depths, and soil sample locations utilizing a GPS unit capable of recording locations on the US State Plane 1983 (New York Western Zone) coordinate system. The RCCR will be submitted as draft to the City for review prior to submittal to NYSDEC. LaBella will address any comments from the City and NYSDEC.

**Brownfields QAPP Template #3b
Project Quality Objectives/Systematic Planning Process Statements**

Overall project objective:

The Site is located within the Bull's Head BOA and associated with the City of Rochester's Bull's Head Revitalization Project. Remedial efforts targeting the removal of fill material and the associated SVOCs and metals impacts are necessary to provide a clean site that promotes redevelopment, in accordance with the Bull's Head Revitalization Plan.

Who will use the data?

The data generated during the completion of the Remedial Work will be used by the City of Rochester and future developers.

What will the data be used for?

The data will be used to verify the remedial action is complete and no contamination in excess of applicable New York State SCGs remain at the Site.

What types of data are needed?

The data required includes field screening and analytical results for the media to which people utilizing the property may be exposed. As invasive activities may be required for any future Site development, the screening and analysis of soil will be required as part of the remedial process. The field screening will include the use of a PID. The sampling will include the collection of soil/fill samples, including the use of an excavator to collect soil/fill samples.

How "good" does the data need to be in order to support the environmental decision?

The data must be of a quality necessary to effectively allow for comparison to NYSDEC soil cleanup objectives. The samples must be representative of the soil and of sufficient quantity to adequately characterize the soil conditions across the Site.

How much data are needed?

The quantity of data must be sufficient to adequately characterize the soil and groundwater across the Site and allow for comparison to New York State soil cleanup objectives/standards.

Where, when, and how should the data be collected/generated?

The data will be collected from the Site. The work will be conducted in fall of 2024. Template #3A, Project Description, Planned Assessment describes in detail the data collection methods.

Who will collect and generate the data?

LaBella Associates, D.P.C.

How will the data be reported?

Template #3A, Project Description, Planned Assessment describes in detail the data reporting methods.

How will the data be archived?

LaBella and the City of Rochester will retain hard and electronic copies of the data and associated report. An administrative record will also be established in accordance with 40 CFR 300.800(a).

**Brownfields QAPP Template #4
Project Schedule/Timeline**

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Preparation of Draft QAPP	LaBella Associates, D.P.C.	5/1/2024	6/3/2024	Draft QAPP	6/3/2024
Review of Draft QAPP	United States EPA	6/3/2024	8/5/2024	Approved Draft QAPP by EPA Region BPO	8/5/2024
QAPP-Finalization	LaBella Associates, D.P.C.	8/5/2024	8/12/2024	Final QAPP	8/12/2024
Procurement of Field/Lab Supplies	LaBella Associates, D.P.C.	8/26/2024	9/3/2024	N/A	N/A
Subsequent Collection of Field Samples	LaBella Associates, D.P.C.	9/10/2024	10/21/2024	N/A	N/A
Laboratory Package Received	LaBella Associates, D.P.C.	11/12/2024	11/12/2024	Unvalidated data package	11/12/2024
Validation of Laboratory Results	Laboratory Data Consultants, Inc.	11/12/2024	1/6/2025	Validated data Packages	1/6/2025
Data Evaluation/ Preparation of Draft RCCR	LaBella Associates, D.P.C.	11/12/2024	1/20/2025	Draft Phase II ESA Report	1/20/2025
Review of Draft Phase II ESA	United States EPA	1/20/2025	3/7/2025	Approved Draft Phase II Report by EPA Region BPO	3/7/2025
Final Phase II ESA Report	LaBella Associates, D.P.C.	3/10/2025	3/17/2025	Final Phase II ESA Report	3/17/2025

**Brownfields QAPP Template #5a
Sampling Methods and Locations**

The following table identifies the sampling methods and locations at the Site.

Matrix	Sampling Location(s)	Depth (feet)	Analytical Group	No. of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
<i>Soil</i>	<i>Excavation Perimeter and Bottom</i>	<i>Variable (Typ. 2 to 9)</i>	<i>CP-51 & TCL VOCs, CP-51 & TCL SVOCs, TAL Metals</i>	<i>31 field samples, 2 MS/MSD, and 2 blind duplicates</i>	<i>LaBella Field QAQC Samples, Sample Packaging, and Shipping SOP and Subsurface Soil Sampling SOP</i>	<i>Confirmatory/ Documentation samples at perimeter and bottom of excavation</i>

Template #3A, Project Description, Planned Assessment describes in greater detail the sampling methods.

**Brownfields QAPP Template #5b
Analytical Methods and Requirements**

This section provides a list of sampling methods and requirements for the Remedial Work.

Matrix	Analytical Group	Concentration Level	Analytical & Preparation Method/SOP Reference	Sample Volume	Containers	Preservation Requirements	Extraction Time	Maximum Sample Holding Time
Soil	VOCs	Low	8260D, Method 5035	15 grams	2, 40-ml Glass vial with 5-mL water, 1, 40-mL Glass vial with 15-mL methanol	Cool to 4 °C	48 hours To freeze	14 days
Soil	SVOCs	Low	8270E, 3546	8 oz.	Glass 250ml/8oz unpreserved	Cool to 4 °C	14 days	40 days
Soil	TAL Metals	Low	6010D,3050B/7471B, 7471B	2 oz.	Glass 60mL/2oz unpreserved	Cool to 4 °C	N/A	180 Days (Hg 28 days)

Brownfields QAPP Template #5c Reference Limits and Evaluation Table

This section identifies the Standards, Criteria and Guidelines (SCGs) to which the analytical results generated during the Remedial Work will be compared. The SCGs identified are used in order to quantify the extent of contamination at the Site that may require remedial work. The NYSDEC has promulgated SCGs for soil for different end-use scenarios, and the most appropriate SCGs for the proposed future redevelopment of the Site are:

- NYCRR Subpart 375-6 Remedial Program Soil Cleanup Objectives (SCOs) for Restricted Residential and Commercial Use
- NYSDEC Petroleum-Contaminated Soil Guidance CP-51, Tables 2 and 3, Hazardous Waste Determination and Regulatory Levels, October 21, 2010

Soil - VOCs 8260

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	NYSDEC CP-51 (ug/kg)	NYSDEC Part 375 Restricted Residential (ug/kg)	NYSDEC Part 375 Commercial (ug/kg)
1,1,1-Trichloroethane	71-55-6	1	0.2698	ug/kg	70-130	30	70-130	30	30	N/A	X	100,000	500,000
1,1,2,2-Tetrachloroethane	79-34-5	1	0.2402	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
1,1,2-Trichloro-1,2,2-Trifluoroethane	76-13-1	20	0.3972	ug/kg	70-130	30		30	30	N/A	X	X	X
1,1,2-Trichloroethane	79-00-5	1.5	0.393	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
1,1-Dichloroethane	75-34-3	1.5	0.2952	ug/kg	70-130	30	70-130	30	30	N/A	X	26,000	240,000
1,1-Dichloroethene	75-35-4	1	0.2598	ug/kg	65-135	30	65-135	30	30	N/A	X	100,000	500,000
1,2,4-Trichlorobenzene	120-82-1	5	0.7898	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
1,2,4-Trimethylbenzene	95-63-6	5	0.573	ug/kg	70-130	30	70-130	30	30	N/A	3,600	52,000	190,000
1,2-Dibromo-3-chloropropane	96-12-8	5	0.8366	ug/kg	68-130	30	68-130	30	30	N/A	X	X	X
1,2-Dibromoethane	106-93-4	4	0.4088	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
1,2-Dichlorobenzene	95-50-1	5	0.3642	ug/kg	70-130	30	70-130	30	30	N/A	X	100,000	500,000
1,2-Dichloroethane	107-06-2	1	0.2274	ug/kg	70-130	30	70-130	30	30	N/A	X	3,100	30,000
1,2-Dichloropropane	78-87-5	3.5	0.255	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
1,3,5-Trimethylbenzene	108-67-8	5	0.6016	ug/kg	70-130	30	70-130	30	30	N/A	8,400	52,000	190,000
1,3-Dichlorobenzene	541-73-1	5	0.3996	ug/kg	70-130	30	70-130	30	30	N/A	X	49,000	280,000
1,4-Dichlorobenzene	106-46-7	5	0.4198	ug/kg	70-130	30	70-130	30	30	N/A	X	13,000	130,000
2-Butanone	78-93-3	10	3.8772	ug/kg	70-130	30	70-130	30	30	N/A	X	100,000	500,000
2-Hexanone	591-78-6	10	0.3964	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
4-Methyl-2-pentanone	108-10-1	10	0.8164	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
Acetone	67-64-1	10	3.235	ug/kg	54-140	30	54-140	30	30	N/A		100,000	500,000
Benzene	71-43-2	1	0.2972	ug/kg	70-130	30	70-130	30	30	N/A	60	4,800	44,000
Bromodichloromethane	75-27-4	1	0.3848	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
Bromoform	75-25-2	4	0.4954	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	NYSDEC CP-51 (ug/kg)	NYSDEC Part 375 Restricted Residential (ug/kg)	NYSDEC Part 375 Commercial (ug/kg)
Bromomethane	74-83-9	2	0.6478	ug/kg	57-147	30	57-147	30	30	N/A	X	X	X
Carbon disulfide	75-15-0	10	0.3754	ug/kg	59-130	30	59-130	30	30	N/A	X	X	X
Carbon tetrachloride	56-23-5	1	0.2112	ug/kg	70-130	30	70-130	30	30	N/A	X	2,400	22,000
Chlorobenzene	108-90-7	1	0.1862	ug/kg	70-130	30	70-130	30	30	N/A	X	100,000	500,000
Chloroethane	75-00-3	2	0.4384	ug/kg	50-151	30	50-151	30	30	N/A	X	X	X
Chloroform	67-66-3	1.5	0.3246	ug/kg	70-130	30	70-130	30	30	N/A	X	49,000	350,000
Chloromethane	74-87-3	5	0.7832	ug/kg	52-130	30	52-130	30	30	N/A	X	X	X
cis-1,2-Dichloroethene	156-59-2	1	0.3014	ug/kg	70-130	30	70-130	30	30	N/A	X	100,000	500,000
cis-1,3-Dichloropropene	10061-01-5	1	0.2672	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
Cyclohexane	110-82-7	20	20	ug/kg	70-130	30	N/A	30	30	N/A	X	X	X
Dibromochloromethane	124-48-1	1	0.3078	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
Dichlorodifluoromethane	75-71-8	10	0.3888	ug/kg	30-146	30	30-146	30	30	N/A	X	X	X
Ethylbenzene	100-41-4	1	0.2214	ug/kg	70-130	30	70-130	30	30	N/A	100	41,000	390,000
Isopropylbenzene	98-82-8	1	0.177	ug/kg	70-130	30	70-130	30	30	N/A	2,300	X	X
Methyl Acetate	79-20-9	20	20	ug/kg	70-130	30	N/A	30	30	N/A	X	X	X
Methyl cyclohexane	108-87-2	4	4	ug/kg	70-130	30	N/A	30	30	N/A	X	X	X
Methyl tert butyl ether	1634-04-4	2	0.487	ug/kg	66-130	30	66-130	30	30	N/A	930	100,000	500,000
Methylene chloride	75-09-2	10	0.816	ug/kg	70-130	30	70-130	30	30	N/A	X	100,000	500,000
Naphthalene	91-20-3	5	0.7696	ug/kg	70-130	30	70-130	30	30	N/A	12,000	X	X
n-Butylbenzene	104-51-8	1	0.3144	ug/kg	70-130	30	70-130	30	30	N/A	12,000	100,000	500,000
n-Propylbenzene	103-65-1	1	0.284	ug/kg	70-130	30	70-130	30	30	N/A	3,900	100,000	500,000
o-Xylene	95-47-6	2	0.4174	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
p/m-Xylene	179601-23-1	2	0.43	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
p-Isopropyltoluene	99-87-6	1	0.2732	ug/kg	70-130	30	70-130	30	30	N/A	10,000	X	X
sec-Butylbenzene	135-98-8	1	0.2756	ug/kg	70-130	30	70-130	30	30	N/A	11,000	100,000	500,000
Styrene	100-42-5	2	0.726	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
tert-Butylbenzene	98-06-6	5	0.6032	ug/kg	70-130	30	70-130	30	30	N/A	5,900	100,000	500
Tetrachloroethene	127-18-4	1	0.3062	ug/kg	70-130	30	70-130	30	30	N/A	X	19,000	150,000
Toluene	108-88-3	1.5	0.2416	ug/kg	70-130	30	70-130	30	30	N/A	700	100,000	500,000
trans-1,2-Dichloroethene	156-60-5	1.5	0.3916	ug/kg	70-130	30	70-130	30	30	N/A	X	100,000	500,000
trans-1,3-Dichloropropene	10061-02-6	1	0.3006	ug/kg	70-130	30	70-130	30	30	N/A	X	X	X
Trichloroethene	79-01-6	1	0.224	ug/kg	70-130	30	70-130	30	30	N/A	X	21,000	200,000
Trichlorofluoromethane	75-69-4	5	0.3914	ug/kg	70-139	30	70-139	30	30	N/A	X	X	X
Vinyl chloride	75-01-4	2	0.7534	ug/kg	67-130	30	67-130	30	30	N/A	X	900	13,000
1,2-Dichloroethane-d4	17060-07-0	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	70-130			

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	NYSDEC CP-51 (ug/kg)	NYSDEC Part 375 Restricted Residential (ug/kg)	NYSDEC Part 375 Commercial (ug/kg)
Toluene-d8	2037-26-5	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	70-130			
4-Bromofluorobenzene	460-00-4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	70-130			
Dibromofluoromethane	1868-53-7	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	70-130			

Soil - SVOCs 8270

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	NYSDEC CP-51 (ug/kg)	NYSDEC Part 375 Restricted Residential (ug/kg)	NYSDEC Part 375 Commercial (ug/kg)
1,2,4-Trichlorobenzene	120-82-1	167	19.1048	ug/kg	38-107	50	38-107	50	50	N/A	X	X	X
1,2-Dichlorobenzene	95-50-1	167	29.9932	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
1,3-Dichlorobenzene	541-73-1	167	28.724	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
1,4-Dichlorobenzene	106-46-7	167	29.1582	ug/kg	28-104	50	28-104	50	50	N/A	X	X	X
2,4,5-Trichlorophenol	95-95-4	167	31.9972	ug/kg	30-130	50	30-130	50	50	N/A	X	X	X
2,4,6-Trichlorophenol	88-06-2	100.2	31.6632	ug/kg	30-130	50	30-130	50	50	N/A	X	X	X
2,4-Dichlorophenol	120-83-2	150.3	26.8536	ug/kg	30-130	50	30-130	50	50	N/A	X	X	X
2,4-Dimethylphenol	105-67-9	167	55.11	ug/kg	30-130	50	30-130	50	50	N/A	X	X	X
2,4-Dinitrophenol	51-28-5	801.6	77.822	ug/kg	4-130	50	4-130	50	50	N/A	X	X	X
2,4-Dinitrotoluene	121-14-2	167	33.4	ug/kg	40-132	50	40-132	50	50	N/A	X	X	X
2,6-Dinitrotoluene	606-20-2	167	28.6572	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
2-Chloronaphthalene	91-58-7	167	16.5664	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
2-Chlorophenol	95-57-8	167	19.7394	ug/kg	25-102	50	25-102	50	50	N/A	X	X	X
2-Methylnaphthalene	91-57-6	200.4	20.1736	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
2-Methylphenol	95-48-7	167	25.885	ug/kg	30-130.	50	30-130.	50	50	N/A	X	100,000	500,000
2-Nitroaniline	88-74-4	167	32.1976	ug/kg	47-134	50	47-134	50	50	N/A	X	X	X
2-Nitrophenol	88-75-5	360.72	62.792	ug/kg	30-130	50	30-130	50	50	N/A	X	X	X
3,3'-Dichlorobenzidine	91-94-1	167	44.422	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
3-Methylphenol/4-Methylphenol	106-44-5	240.48	26.1522	ug/kg	30-130	50	30-130	50	50	N/A	X	100,000	500,000
3-Nitroaniline	99-09-2	167	31.4962	ug/kg	26-129	50	26-129	50	50	N/A	X	X	X
4,6-Dinitro-o-cresol	534-52-1	434.2	80.16	ug/kg	10-130	50	10-130	50	50	N/A	X	X	X
4-Bromophenyl phenyl ether	101-55-3	167	25.4842	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
4-Chloroaniline	106-47-8	167	30.394	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
4-Chlorophenyl phenyl ether	7005-72-3	167	17.869	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	NYSDEC CP-51 (ug/kg)	NYSDEC Part 375 Restricted Residential (ug/kg)	NYSDEC Part 375 Commercial (ug/kg)
4-Nitroaniline	100-01-6	167	69.138	ug/kg	41-125	50	41-125	50	50	N/A	X	X	X
4-Nitrophenol	100-02-7	233.8	68.136	ug/kg	11-114	50	11-114	50	50	N/A	X	X	X
Acenaphthene	83-32-9	133.6	17.3012	ug/kg	31-137	50	31-137	50	50	N/A	20,000	100,000	500,000
Acenaphthylene	208-96-8	133.6	25.7848	ug/kg	40-140	50	40-140	50	50	N/A	100,000	100,000	500,000
Acetophenone	98-86-2	167	20.6746	ug/kg	14-144	50	14-144	50	50	N/A	X	X	X
Anthracene	120-12-7	100.2	32.565	ug/kg	40-140	50	40-140	50	50	N/A	100,000	100,000	500,000
Benzo(a)anthracene	56-55-3	100.2	18.8042	ug/kg	40-140	50	40-140	50	50	N/A	1,000	1,000	5,600
Benzo(a)pyrene	50-32-8	133.6	40.748	ug/kg	40-140	50	40-140	50	50	N/A	1,000	1,000	1,000
Benzo(b)fluoranthene	205-99-2	100.2	28.1228	ug/kg	40-140	50	40-140	50	50	N/A	1,000	1,000	5,600
Benzo(ghi)perylene	191-24-2	133.6	19.6392	ug/kg	40-140	50	40-140	50	50	N/A	100,000	100,000	500,000
Benzo(k)fluoranthene	207-08-9	100.2	26.72	ug/kg	40-140	50	40-140	50	50	N/A	800	3,900	56,000
Benzoic Acid	65-85-0	541.08	169.004	ug/kg	10-110	50	10-110	50	50	N/A	X	X	X
Benzyl Alcohol	100-51-6	167	51.102	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Biphenyl	92-52-4	380.76	21.71	ug/kg	37-127	50	37-127	50	50	N/A	X	X	X
Bis(2-chloroethoxy)methane	111-91-1	180.36	16.7334	ug/kg	40-117	50	40-117	50	50	N/A	X	X	X
Bis(2-chloroethyl)ether	111-44-4	150.3	22.6452	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Bis(2-chloroisopropyl)ether	108-60-1	200.4	28.5236	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Bis(2-Ethylhexyl)phthalate	117-81-7	167	57.782	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Butyl benzyl phthalate	85-68-7	167	42.084	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Carbazole	86-74-8	167	16.2324	ug/kg	54-128	50	54-128	50	50	N/A	X	X	X
Chrysene	218-01-9	100.2	17.368	ug/kg	40-140	50	40-140	50	50	N/A	1,000	3,900	56,000
Dibenzo(a,h)anthracene	53-70-3	100.2	19.3052	ug/kg	40-140	50	40-140	50	50	N/A	330	330	5,600
Dibenzofuran	132-64-9	167	15.7982	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Diethyl phthalate	84-66-2	167	15.4642	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Dimethyl phthalate	131-11-3	167	35.07	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Di-n-butylphthalate	84-74-2	167	31.6632	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Di-n-octylphthalate	117-84-0	167	56.78	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Fluoranthene	206-44-0	100.2	19.1716	ug/kg	40-140	50	40-140	50	50	N/A	100,000	100,000	500,000
Fluorene	86-73-7	167	16.2324	ug/kg	40-140	50	40-140	50	50	N/A	30,000	100,000	500,000
Hexachlorobenzene	118-74-1	100.2	18.704	ug/kg	40-140	50	40-140	50	50	N/A		12,000	6,000
Hexachlorobutadiene	87-68-3	167	24.4488	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Hexachlorocyclopentadiene	77-47-4	477.62	151.302	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Hexachloroethane	67-72-1	133.6	27.0206	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Indeno(1,2,3-cd)Pyrene	193-39-5	133.6	23.2798	ug/kg	40-140	50	40-140	50	50	N/A	500	500	5,600
Isophorone	78-59-1	150.3	21.6766	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
Naphthalene	91-20-3	167	20.3406	ug/kg	40-140	50	40-140	50	50	N/A	12,000	100,000	500,000
Nitrobenzene	98-95-3	150.3	24.716	ug/kg	40-140	50	40-140	50	50	N/A	X	X	X
NitrosoDiPhenylAmine(NDPA)/DPA	86-30-6	133.6	19.0046	ug/kg	36-157	50	36-157	50	50	N/A	X	X	X

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	NYSDEC CP-51 (ug/kg)	NYSDEC Part 375 Restricted Residential (ug/kg)	NYSDEC Part 375 Commercial (ug/kg)
n-Nitrosodi-n-propylamine	621-64-7	167	25.7848	ug/kg	32-121	50	32-121	50	50	N/A	X	X	X
P-Chloro-M-Cresol	59-50-7	167	24.883	ug/kg	26-103	50	26-103	50	50	N/A	X	X	X
Pentachlorophenol	87-86-5	133.6	36.74	ug/kg	17-109	50	17-109	50	50	N/A	X	6,700	6,700
Phenanthrene	85-01-8	100.2	20.3072	ug/kg	40-140	50	40-140	50	50	N/A	100,000	100,000	500,000
Phenol	108-95-2	167	25.217	ug/kg	26-90	50	26-90	50	50	N/A	X	100,000	500,000
Pyrene	129-00-0	100.2	16.5998	ug/kg	35-142	50	35-142	50	50	N/A	100,000	100,000	500,000
1,4-Dioxane	123-91-1	25.05	7.682	ug/kg	40-140	50	40-140	50	50	N/A	X	13,000	130,000

Soil – Metals 6010/7471

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	NYSDEC CP-51 (ug/kg)	NYSDEC Part 375 Restricted Residential (ug/kg)	NYSDEC Part 375 Commercial (ug/kg)
Aluminum, Total	7429-90-5	4	1.08	mg/kg	48-151	N/A	75-125	20	20	N/A	X	X	X
Antimony, Total	7440-36-0	2	0.152	mg/kg	1-208	N/A	75-125	20	20	N/A	X	X	X
Arsenic, Total	7440-38-2	0.4	0.0832	mg/kg	79-121	N/A	75-125	20	20	N/A	X	1,600	1,600
Barium, Total	7440-39-3	0.4	0.0696	mg/kg	83-117	N/A	75-125	20	20	N/A	X	400,000	400,000
Beryllium, Total	7440-41-7	0.2	0.0132	mg/kg	83-117	N/A	75-125	20	20	N/A	X	72,000	590,000
Cadmium, Total	7440-43-9	0.4	0.0392	mg/kg	83-117	N/A	75-125	20	20	N/A	X	4,300	9,300
Calcium, Total	7440-70-2	4	1.4	mg/kg	81-119	N/A	75-125	20	20	N/A	X	X	X
Chromium, Total	7440-47-3	0.4	0.0384	mg/kg	80-120	N/A	75-125	20	20	N/A	X	180,000	1,500,000
Cobalt, Total	7440-48-4	0.8	0.0664	mg/kg	84-115	N/A	75-125	20	20	N/A	50	X	X
Copper, Total	7440-50-8	0.4	0.1032	mg/kg	81-118	N/A	75-125	20	20	N/A	X	270,000	270,000
Iron, Total	7439-89-6	2	0.3612	mg/kg	45-155	N/A	75-125	20	20	N/A	2,000,000	X	X
Lead, Total	7439-92-1	2	0.1072	mg/kg	81-117	N/A	75-125	20	20	N/A	X	400	1,000
Magnesium, Total	7439-95-4	4	0.616	mg/kg	76-124	N/A	75-125	20	20	N/A	X	X	X
Manganese, Total	7439-96-5	0.4	0.0636	mg/kg	81-117	N/A	75-125	20	20	N/A	X	2,000,000	10,000,000
Nickel, Total	7440-02-0	1	0.0968	mg/kg	83-117	N/A	75-125	20	20	N/A	X	310,000	310,000
Potassium, Total	7440-09-7	100	5.76	mg/kg	71-129	N/A	75-125	20	20	N/A	X	X	X
Selenium, Total	7782-49-2	0.8	0.1032	mg/kg	78-122	N/A	75-125	20	20	N/A	X	180,000	1,500,000
Silver, Total	7440-22-4	0.2	0.1132	mg/kg	75-124	N/A	75-125	20	20	N/A	X	180,000	1,500,000

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	NYSDEC CP-51 (ug/kg)	NYSDEC Part 375 Restricted Residential (ug/kg)	NYSDEC Part 375 Commercial (ug/kg)
Sodium, Total	7440-23-5	80	1.26	mg/kg	72-127	N/A	75-125	20	20	N/A	X	X	X
Thallium, Total	7440-28-0	0.8	0.126	mg/kg	80-120	N/A	75-125	20	20	N/A	5,000	X	X
Vanadium, Total	7440-62-2	0.4	0.0812	mg/kg	78-122	N/A	75-125	20	20	N/A	100,000	X	X
Zinc, Total	7440-66-6	2	0.1172	mg/kg	82-118	N/A	75-125	20	20	N/A	X	10,000,000	10,000,000
Mercury, Total	7439-97-6	0.08	0.05216	mg/kg	72-128	N/A	80-120	20	20	N/A	X	0.81	2.8

**Brownfields QAPP Template #5d
Analytical Laboratory Sensitivity and Project Criteria**

The sensitivity and project criteria for laboratory sample analysis is described below. The analytical methods to be employed during the Remedial Work have been based on sensitivities that allow for the comparison of the results to appropriate NYSDEC standards, criteria and guidance.

Soil - VOCs 8260

Laboratory: Alpha Analytical, Westborough, MA

Matrix: Soil

Analytical Group or Method: 8260

Concentration Level: LOW

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 30%
Analytical Precision (laboratory)	Matrix Spike Duplicates (at client's request)	RPD ≤ 30%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Generally, 70-130%R – analyte specific
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/ Matrix Spike Duplicates (at client's request)	Generally, 70-130%R – analyte specific
Accuracy/Extraction efficiency	Surrogates	70-130%R
Analytical Accuracy	Internal Standards	50-200% of the IS area count in the CCV
Analytical Accuracy	Initial Calibration Verification (ICV)	%D ≤ 30 (exclusions apply – see SOP) Prepared using standard source different than used for initial calibration
Analytical Accuracy	Continuing Calibration Verification (CCV)	%D ≤ 20 except for 20% of compounds may be > 20 but ≤ 30%D Area counts of internal standards must be within 50–200% of the mid-level initial calibration standard
Overall accuracy/bias (contamination)	Method Blank	No target compounds ≥ RL
Sensitivity	Method Detection Limit	MDL < RL

Soil - SVOCs 8270

Laboratory: Alpha Analytical, Westborough, MA

Matrix: Soils and Waters

Analytical Group or Method: 8270

Concentration Level: LOW

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 50%

Analytical Precision (laboratory)	Matrix Spike Duplicates (at client's request)	RPD \leq 50%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Generally, 40-140%R for Base Neutrals; 30-130%R for Acids, analyte specific control limits for difficult compounds
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/ Matrix Spike Duplicates (at client's request)	Generally, 40-140%R for Base Neutrals; 30-130%R for Acids, analyte specific control limits for difficult compounds
Accuracy/Extraction efficiency	Surrogates	Analyte specific control limits
Analytical Accuracy	Internal Standards	50-200% of the IS area count in the CCV
Analytical Accuracy	ICV	70-130%, 40-160%R for difficult analytes. Prepared using standard source different than used for initial calibration
Analytical Accuracy	CCV	%D \leq 20 except difficult compounds may exhibit %D < 60; Minimum RF per Table 4 in SW-846 8270E. Area counts of internal standards must be within 50-200% of the mid-level initial calibration standard
Overall accuracy/bias (contamination)	Method blank	No target compounds \geq RL
Sensitivity	Method Detection Limit	MDL < RL

Soil – Metals 6010D

Laboratory: Alpha Analytical, Mansfield, MA

Matrix: Soils

Analytical Group or Method: 6010D

Concentration Level: Analyte-specific

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field Duplicates	RPD \leq 20%
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD \leq 20%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Recovery range 80-120%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Recovery range 75-125%
Analytical Accuracy	ICV/CCV	90-110% 90-110% on continuing
Analytical Accuracy	Initial Calibration Blank (ICB)/ Continuing Calibration Blank (CCB)	< RL > 2x RL if below zero
Analytical Accuracy	Method Blank	< RL > 2x RL if below zero
Overall Accuracy/Bias (contamination)	Equipment Blanks	No target analyte concentrations \geq $\frac{1}{2}$ LOQ

Sensitivity Precision	LOQ verification sample (spiked at LOQ)	Recovery within $\pm 25\%$ of LOQ
Sensitivity Qualitative	LOD verification sample (spiked at 3-5X MDL)	Qualitative response (estimated J qualified data only)
Completeness	Project-specific	Data completeness check

Soil – Metals 7470A

Laboratory: Alpha Analytical, Westborough, MA

Matrix: Soil

Analytical Group or Method: 7470A

Concentration Level: LOW

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field Duplicates	RPD $\leq 20\%$
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD $\leq 20\%$
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Recovery range 80-120%
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Recovery range 70-130%
Analytical Accuracy	ICV/CCV	90-110% 80-120% on continuing
Analytical Accuracy	ICB/CCB	< RL > 2x RL if below zero
Analytical Accuracy	Method Blank	< RL > 2x RL if below zero
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations $\geq \frac{1}{2}$ LOQ
Sensitivity Precision	LOQ verification sample (spiked at LOQ)	Recovery within $\pm 25\%$ of LOQ
Sensitivity Qualitative	LOD verification sample (spiked at 3-5X MDL)	Qualitative response (estimated J qualified data only)

**Brownfields QAPP Template #5e
Secondary Data Criteria and Limitations Table**

Secondary data exists for the Site. The following discusses this information:

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
Environmental Screen	Day Environmental Inc., Environmental Screen, September 2009	Day Environmental, Background including Site history	Help identify environmental concerns at site and create objectives for Phase II ESA	Information is limited to historical records and site observations. No analytical data was generated.
Phase I ESA	Day Environmental Inc., Phase I Environmental Site Assessment 894-898 West Main Street, and 42 York Street Rochester, New York, August 16, 2016	Day Environmental, Background including Site history	Help identify environmental concerns at site and create objectives for Phase II ESA	Information is limited to historical records and site observations. No analytical data was generated.
Phase II ESA and Geotechnical Study Report	Day Environmental Inc., Pre-Development Phase II ESA and Geotechnical Study Report, July 2019	Day Environmental, Soil/Fill Samples, Groundwater Samples, Geotechnical Assessment, February 2018 to April 2018	To assess existing subsurface conditions and depth of fill materials onsite.	<ol style="list-style-type: none"> 1. Data generated for the report was unvalidated. 2. Limited number of wells exist (1)
Phase II ESA	LaBella Associates D.P.C., Phase II ESA, dated October 9, 2023	LaBella Associates D.P.C., soil/fill samples, groundwater samples, July-August 2023	To assess existing subsurface conditions and depth/location of fill materials onsite.	None

**Brownfields QAPP Template #6
Project Specific Method and Standard Operating Procedures (SOPs) Reference Table**

The following SOPs, analytical method references, and corresponding analytical laboratory SOPs will be used for this project. These include:

ANALYTICAL METHOD REFERENCE
1a. USEPA 8260B VOCs, SW 846 Solid & Hazardous Waste Methods, Update IVB, 1/3/2008
2a. USEPA 8270 SVOCs, SW 846 Solid & Hazardous Waste Methods, Update IVB, 7/2014
3a. USEPA 6010/7471 RCRA Metals, SW 846 Solid & Hazardous Waste Methods, Update IVB, 1/3/2008
ANALYTICAL LABORATORY SOPs
1b. Mercury in Solid or Semisolid Waste, Alpha Analytical, Inc. July 7, 2022
2b. Inductively Coupled Plasma – Atomic Emission Spectrometry (6010D), Alpha Analytical, Inc. December 23, 2022
3b. Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS), Alpha Analytical, Inc. April 29, 2021
4b. Volatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS), Alpha Analytical, Inc. November 8, 2022
FIELD SAMPLING SOPs
1c. Soil Identification and Description
2c. Subsurface Soil Sampling
3c. Equipment Decontamination
4c. Sample Packaging and Shipping

**Brownfields QAPP Template #7
Field Equipment Calibration, Maintenance, Testing, and Inspection**

The following table identifies the equipment and instruments that are expected to be utilized during the performance of this Phase II ESA. The table also identifies the required calibration, maintenance, testing or inspection and the SOP reference number for each type of equipment.

Field Equipment	Calibration Activity	Maintenance Activity	Testing/ Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	SOP Reference
MiniRAE 3000 Photoionization Detector	Calibrate with isobutylene gas	NA	NA	By supplier prior to arrival on-site	Response should be within 0.5 ppm of calibration gas standard	Replace any filters: clean lamp: return for service	MiniRAE 3000 Users' Manual

The equipment used during the course of the project will be rented, and prior to shipment to the Site, the rental company/facility will ensure that proper maintenance and periodic testing is performed on the equipment. The rental company will provide equipment calibration records that shall be reviewed prior to use and retained for reference. If additional equipment and instruments are necessary for the performance of the Remedial Action, the field equipment manufacturer's recommendations will be adhered to.

Brownfields QAPP Template #8

Analytical Laboratory Instrument and Equipment Maintenance, Testing, and Inspection

The analytical methods and the equipment to perform those analyses will be maintained, tested, and inspected in accordance with the manufacturer’s guidelines and the requirements of the particular analytical method to be performed. The following tables identify all of the equipment the laboratory uses for each analytical method and the SOPs for maintenance, testing, and inspection. For additional information, refer to the Laboratory Quality Assurance Manual (Appendix C). For the USEPA-funded Site Remediation, the laboratory will ensure adherence to the SOPs and the requirements for each analytical method.

VOCs

Instrument/Equipment	Maintenance Activity	Testing/Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	Analytical SOP Reference
HP GC/MS	Injection port maintenance	Preventive maintenance	Daily	Tune and CCV pass criteria	Inspect injector port, cut column, retune instrument, run calibration	Analyst/Supervisor	P255-Maintenance-05
	Detector maintenance	Unable to tune instrument	When tune fails or responses drop	Tune and calibration pass criteria, no air or water in tune scan	Disassemble detector, check parts, check heating element, rerun tune		

SVOCs

Instrument/Equipment	Maintenance Activity	Testing/Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	Analytical SOP Reference
GC/MS	Injection port maintenance	Preventive maintenance	Daily	Tune and CCV pass criteria	Inspect injector port, cut column, retune instrument, run calibration	Analyst/Supervisor	P255-Maintenance-04
	Detector maintenance	Unable to tune instrument	When tune fails or responses drop	Tune and calibration pass criteria, no air or water in tune scan	Disassemble detector, check parts, check heating element, rerun tune		

Metals

Instrument/Equipment	Maintenance Activity	Testing/Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	Analytical SOP Reference
ICP	Tubing	Preventive maintenance	Daily	Calibration passes criteria	Replace tubing	Analyst/Supervisor	P255-Maintenance-05
	Nebulizer		Monthly		Clean nebulizer		
	Torch		Annual		Replace torch		

Mercury

Instrument/ Equipment	Maintenance Activity	Testing/Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	Analytical SOP Reference
CV	Tubing Lamp, optic cell Mercury lamp	Preventive maintenance	Daily Monthly Annual	Calibration passes criteria	Replace tubing Clean lamp and optic cell Replace mercury lamp	Analyst/Supervisor	P255- Maintenance- 05

Analytical Laboratory Instrument Calibration

The protocols for calibrating the laboratory equipment will be performed in accordance the manufacturer's guidelines and the requirements of the particular analytical method to be performed. The following tables identify all of the equipment the laboratory uses for each analytical method and the SOPs for calibration. For additional information, refer to the Laboratory Quality Assurance Manual (Appendix C). For this USEPA-funded Site Remediation, the laboratory will ensure adherence to the SOPs and the requirements for each analytical method.

VOCs

Instrument/Equipment	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Responsible Person	Analytical SOP Reference
GC/MS (VOC)	BFB Tuning	Prior to initial calibration and calibration verification (every 12 hours)	Refer to criteria listed in the method	Retune instrument and verify	Lab analyst	M8260D-SWGCMSSVOA-26
	Multipoint initial calibration (minimum five points)	Prior to sample analysis, or when calibration verification fails	All analytes <20% RSD or correlation coefficient \geq 0.990	Correct the problem and repeat the initial calibration	Lab analyst	
	Second-source calibration verification	Once for each multipoint initial calibration	All analytes within \pm 30% of expected value	Correct the problem and repeat initial calibration	Lab analyst	
	Continuing calibration verification	At start of each analytical sequence and every 12 hours thereafter	All analytes within \pm 20% of expected value	Correct problem, then recalibrate and reanalyze all samples since the last acceptable continuing calibration verification	Lab analyst	

SVOCs

Instrument/Equipment	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Responsible Person	Analytical SOP Reference
GC/MS	DFTPP Tuning	Prior to initial calibration and calibration verification (every 12 hours)	Refer to criteria listed in the method	Retune instrument and verify	Lab analyst	M8270E-BNA-28
	Multipoint initial calibration (minimum five points)	Prior to sample analysis, or when calibration verification fails	RSD \leq 15; (8270C) RSD \leq 20; (8270D) Correlation Coefficient (R^2) \geq 0.990	Correct the problem and repeat the initial calibration	Lab analyst	
	Second-source calibration verification	Once for each multipoint initial calibration	All analytes within \pm 30% of expected value	Correct the problem and repeat initial calibration	Lab analyst	
	Continuing calibration verification	At start of each analytical sequence and every 12 hours thereafter	All analytes within \pm 20% of expected value	Correct problem, then recalibrate and reanalyze all samples since the last acceptable continuing calibration verification	Lab analyst	

Metals

Instrument/ Equipment	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Responsible Person	Analytical SOP Reference
ICP (Metals)	Initial calibration	Before sample analysis, every 24 hours, whenever modifications are made to the system, or when continuing calibration verification fails	If more than one standard is used, correlation coefficient must be > 0.995	Correct problem and repeat initial calibration	Lab analyst	M6010B-C-Trace Elements-23
	Second-source calibration verification	Immediately following each initial calibration	All analytes within $\pm 10\%$ of expected value	Correct problem and repeat initial calibration	Lab analyst	
	Calibration Blank	After every 10 samples and at the end of the sequence	No analytes detected at or above $\frac{1}{2}$ reporting limit	Correct problem, then reanalyze previous 10 samples	Lab analyst	
	Continuing calibration verification	After every 10 samples and at the end of the sequence	All analytes within $\pm 10\%$ of expected value	Recalibrate and reanalyze all samples since the last acceptable continuing calibration verification	Lab analyst	

Mercury

Instrument/ Equipment	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Responsible Person	Analytical SOP Reference
CVAA (Mercury)	Initial calibration	Before sample analysis, every 24 hours, whenever modifications are made to the system, or when continuing calibration verification fails	Correlation coefficient must be > 0.995	Correct problem and repeat initial calibration	Lab analyst	M7470A-Mercury-17, M7471A-B-Mercury-16
	Second-source calibration verification	Immediately following each initial calibration	All analytes within $\pm 10\%$ of expected value	Correct problem and repeat initial calibration	Lab analyst	
	Calibration Blank	Before any sequence, after every 10 samples and at the end of the sequence	No analytes detected at or above $\frac{1}{2}$ reporting limit	Correct problem, then reanalyze previous 10 samples	Lab analyst	
	Continuing calibration verification	After every 10 samples and at the end of the sequence	All analytes within $\pm 20\%$ of expected value	Recalibrate and reanalyze all samples since last acceptable continuing calibration verification	Lab analyst	

**Brownfields QAPP Template #9a
Sample Handling System**

The following table identifies the components of the sample handling system.

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Alex daSilva, Katherine Truong, Drew Brantner, LaBella Associates, D.P.C.
Sample Packaging (Personnel/Organization): Alex daSilva, Katherine Truong, Drew Brantner, LaBella Associates, D.P.C.
Coordination of Shipment (Personnel/Organization): Alex daSilva, Katherine Truong, Drew Brantner, LaBella Associates, D.P.C.
Type of Shipment/Carrier: Alpha Analytical courier or 3 rd -Party Shipping Service (i.e., UPS or Fedex)
SAMPLE RECEIPT AND ANALYSIS (Alpha Analytical, Inc.)
Sample Receipt (Personnel/Organization): Alpha Log-in/Custody Staff
Sample Custody and Storage (Personnel/Organization): Alpha Log-in/Custody Staff
Sample Preparation (Personnel/Organization): Alpha Staff
Sample Determinative Analysis (Personnel/Organization): Alpha Staff
SAMPLE ARCHIVING
Field Sample Storage: As per analytical methodology; See Template #5b
Sample Extract/Digestate Storage (No. of days from extraction/digestion): As per analytical methodology; See Template #5b.
SAMPLE DISPOSAL
Personnel/Organization: Alpha Staff
Number of Days from Analysis: Until analysis and QA/QC checks are completed per agreement between LaBella Associates, D.P.C. and Alpha.

Brownfields QAPP Template #9b Sample Custody Requirements

The procedures that will be used to maintain sample custody and integrity for the site-specific project include the use of chain-of-custody forms, sample identification, custody seals, laboratory sample receipt forms, and laboratory sample transfer forms. The following describes the sample custody procedures that will be implemented during the Remedial Action:

Sample Identification

- All samples collected for the project will be identified using the following format:

SOIL-XX-Yft

XX = indicates the sample type

XX = identify the sample number/location

Y-Yft = sample depth in feet

- Each sample will be labeled, chemically preserved, if required and sealed immediately after collection
- Sample labels will be filled out using waterproof ink, firmly affixed to the sample containers, and protected by Mylar tape
- The sample label will give the following information:
 - Site name
 - Project number
 - Name of sampler
 - Date and time of collection
 - Sample number
 - Analysis required and preservative

Sample Custody

- Sample identification documents must be prepared so that sample identification and chain-of-custody can be maintained and sample disposition controlled
- Sample identification documents include:
 - field notebooks
 - sample label
 - custody seals
 - chain-of-custody

Chain-of-Custody

1. Field Custody Procedures
 - As few persons as possible should handle samples
 - Sample bottles will be obtained certified / precleaned and coolers or boxes containing cleaned bottles should be sealed with a custody tape during transport or while in storage

- The collector is personally responsible for the care and custody of samples until they are transferred under chain-of-custody rules
- The collector will record sample data in the notebook
- The site manager will determine whether proper custody procedures were followed during fieldwork and decide if additional samples are required

2. Sample Tags

- Sample tags attached to or affixed around the sample container must be used to properly identify all samples collected in the field
- Field identification must be sufficient to enable cross referencing with the logbook
- All QC samples are subject to exactly the same custodial procedures and documentation as “real” samples

3. Transfer of Custody Procedures

- Coolers in which samples are packed must be accompanied by a chain-of-custody record
- Individuals relinquishing and receiving samples must sign, date, and note the time on the chain-of-custody record
- Shipping containers must be sealed with custody seals for shipment and be accompanied by the chain-of-custody record placed in a plastic bag and taped to the interior bottom of the cooler lid.
- Shipping containers will remain sealed during shipment, which will be accomplished via courier or 3rd-party shipping service (i.e., UPS or FedEx)

4. Chain-of-Custody Record

- Must be fully completed in duplicate by the field technician who has been designated by the project manager as responsible for sample shipment
- Note special handling instructions or lab requests in the remarks section of the record

Sample Handling, Packaging, and Shipping

1. Sample Packaging

- Sample bottle lids should never be mixed.
- Sample volume level can be marked so the laboratory can determine if any of the sample has leaked during transport to the laboratory.
- Sample bottles are placed in a plastic bag to minimize the potential for vermiculite contamination.
- Shipping coolers must be partially filled with packing materials and ice when required, to prevent the bottles from moving during shipment.
- Sample bottles should be placed in the cooler such that they don't contact one another.
- The samples should be cooled with “blue ice” or ice.
- Any remaining space in the cooler should be filled with inert packing material.
- A duplicate custody record and traffic reports, if required, must be placed in a plastic bag and taped to the interior bottom of the cooler lid. Custody seals are affixed to the sample cooler.

2. Shipping Containers – should be custody sealed prior to shipping.

3. Marking and Labeling

- Use abbreviations only where specified.
- "This end up" or "This side up" must be clearly printed on the top of the outer package. Upward pointing arrows should be placed on the sides of the package.
- After a sample container has been sealed, two (2) chain-of-custody seals are placed on the container, one on the front and one on the back.
- If samples are designated as medium or high hazard, they must be sealed in metal paint cans, placed in the cooler with vermiculite and labeled and placarded in accordance with DOT regulations.
- Coolers must also be labeled and placarded in accordance with DOT regulations if shipping medium and high hazard samples.

Laboratory Procedures

- Following receipt of the samples, the laboratory will accept, log, and maintain chain of custody in accordance custody procedures are described in Section 11 of the attached Laboratory Quality Assurance Manual (Appendix C).

**Brownfields QAPP Template #10
Field Quality Control Summary**

The quality control procedures for the field data collection are described below. These procedures will follow appropriate standards, guidelines, and SOPs, and will include:

- The collection and handling of samples in accordance with the appropriate SOPs
- The collection of an appropriate number of Quality Assurance/Quality Control (QA/QC) samples, which will include, as appropriate:
 - Duplicate samples
 - Matrix spike/matrix spike duplicates
- The analysis of samples in accordance with the prescribed methods
- The performance of all appropriate corrective actions when necessary to ensure quality data

The following tables summarize the QA/QC procedures to be followed for each analyte group.

Soil - VOCs

Matrix	Soil
Analytical Group	VOCs
Concentration Level	Low/Medium - ug/kg (ppb)
Sampling SOP(s)	LaBella SOPs: -Soil Identification and Description -Subsurface Soil Sampling -Equipment Decontamination -Sample Packaging and Shipment
Analytical Method/SOP Reference	SW 846-8260D/M8260B-C-SWGCMSVOA-26
Sampling Team Lead	Drew Brantner
Sampler's Name(s)	Alex daSilva, Katherine Truong
Field Sampling Organization	LaBella Associates
Analytical Organization	Alpha
No. of Sample Locations	Estimate 31

Quality Control (QC) Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Field Duplicate	Two	RPD ≤ 20%	Investigate cause of excess RPD and take appropriate action, if necessary.	Sampling Team Lead	Accuracy/Precision
MS/MSD	Two	See Template #5d	Investigate cause of excess RPD and take appropriate action, if necessary.	Sampling Team Lead and Laboratory	Accuracy/Bias

Soil -SVOCs

Matrix	Soil
Analytical Group	SVOCs
Concentration Level	Low/Medium - ug/kg (ppb)
Sampling SOP(s)	LaBella SOPs: -Soil Identification and Description -Subsurface Soil Sampling -Equipment Decontamination -Sample Packaging and Shipment
Analytical Method/SOP Reference	SW 846-8270E/M8270E-BNA-27
Sampling Team Lead	Drew Brantner
Sampler's Name(s)	Alex daSilva, Katherine Truong
Field Sampling Organization	LaBella Associates, D.P.C.
Analytical Organization	Alpha
No. of Sample Locations	Estimate 31

Quality Control (QC) Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Field Duplicate	Two	RPD ≤ 20%	Investigate cause of excess RPD and take appropriate action, if necessary.	Sampling Team Lead	Accuracy/Precision

Quality Control (QC) Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
MS/MSD	Two	See Template #5d	Investigate cause of excess RPD and take appropriate action, if necessary.	Sampling Team Lead and Laboratory	Accuracy/Bias

Soil - Metals (6010)

Matrix	Soil
Analytical Group	Metals
Concentration Level	Low/medium - mg/kg (ppm)
Sampling SOP(s)	LaBella SOPs: -Soil Identification and Description -Subsurface Soil Sampling -Equipment Decontamination -Sample Packaging and Shipment
Analytical Method/SOP Reference	SW-846 6010D/SOP#26796rev.2
Sampling Team Lead	Drew Brantner
Sampler's Name(s)	Alex daSilva, Katherine Truong
Field Sampling Organization	LaBella Associates, D.P.C.
Analytical Organization	Alpha
No. of Sample Locations	Estimate 31

Quality Control (QC) Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Field Duplicate	One	RPD ≤ 20%	Investigate cause of excess RPD and take appropriate action, if necessary.	Sampling Team Lead	Accuracy/Precision
MS/MSD	One	See Template #5d	Investigate cause of excess RPD and take appropriate action, if necessary.	Sampling Team Lead and Laboratory	Accuracy/Bias

Soil - Metals (7471)

Matrix	Soil
Analytical Group	Metals
Concentration Level	Low/medium - mg/kg (ppm)
Sampling SOP(s)	LaBella SOPs: -Soil Identification and Description -Subsurface Soil Sampling -Equipment Decontamination -Sample Packaging and Shipment
Analytical Method/SOP Reference	SW-846-7471B
Sampling Team Lead	Drew Brantner
Sampler's Name(s)	Alex daSilva, Katherine Truong
Field Sampling Organization	LaBella Associates, D.P.C.
Analytical Organization	Alpha
No. of Sample Locations	Estimate 31

Quality Control (QC) Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Field Duplicate	Two	RPD ≤ 20%	Investigate cause of excess RPD and take appropriate action, if necessary.	Sampling Team Lead	Accuracy/Precision
MS/MSD	Two	See Template #5d	Investigate cause of excess RPD and take appropriate action, if necessary.	Sampling Team Lead and Laboratory	Accuracy/Bias

**Brownfields QAPP Template #11a
Data Management and Documentation**

The types of documentation generated, collected, and managed during this Remedial Action are summarized below:

Field Sample Collection Documents and Records	Analytical Laboratory Documents and Records	Data Assessment Documents and Records	Project File
<ul style="list-style-type: none"> • Site and field logbooks • Chain-of-Custody (COC) forms • Field Data Sheets • Photograph logs • 	<ul style="list-style-type: none"> • Sample receipt logs • Internal and external COC forms • Sample preparation worksheets/logs • Sample analysis worksheets/run logs • Telephone/email logs • Corrective action documentation • ASP Category B Laboratory Analytical Data Package 	<ul style="list-style-type: none"> • Data validation reports • Field inspection checklist(s) • Laboratory Audit checklist (if performed) • Review forms for electronic entry of data into database • Corrective action documentation 	<ul style="list-style-type: none"> • Project files will be maintained by LaBella for a minimum of five years following completion of the Remedial Action

Appendix B contains examples of many of the logs and other forms that will be used during the course of the Phase II ESA. Additionally, Appendix D contains the laboratory’s procedures for documentation generation and handling.

**Brownfields QAPP Template #11b
Project Reports**

Over the course of the Remedial Action, a number of reports will be generated. The following table provides a list of the reports that will be created:

Type of Report	Frequency (Daily, weekly, monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation	Report Recipient(s)
Status Reports	Ongoing	Friday each week	Drew Brantner, LaBella Associates, D.P.C.	City of Rochester
RCCR	One at conclusion of project	Refer to Template #4	Drew Brantner, LaBella Associates, D.P.C.	City of Rochester, USEPA Grant Manager
Quarterly Reports	Quarterly	30 days after end of each quarter	Drew Brantner, LaBella Associates, D.P.C.	City of Rochester, USEPA Grant Manager
MBE/WBE Reports	Annually	30 days after end of year	Drew Brantner, LaBella Associates, D.P.C.	City of Rochester, USEPA Grant Manager

**Brownfields QAPP Template #12a
Planned Project Assessments/Audits Table**

No project assessments/audits are planned to be completed during the course of this Remedial Action.

Brownfields QAPP Template #12b
Assessment/Audit Findings and Corrective Action Responses

No project assessments/audits are planned to be completed during the course of this Remedial Action.

**Brownfields QAPP Template #13a
Project Data Verification Process (Step I)**

During the course of this Remedial Action, a variety of data will be generated. To ensure accuracy and completeness, the processes listed below will be followed:

Verification Input	Description	Internal/ External	Responsible for Verification
Site/Field Logbooks and Forms	Field notes will be prepared daily by Field Staff Members and will be complete, appropriate, legible and pertinent. Upon completion of field work, logbooks will be checked by the Environmental Consultant Project Manager and placed in the project files.	Internal	Alex daSilva, LaBella Associates, D.P.C. (Preparation); Drew Brantner, LaBella Associates, D.P.C. (Review)
Chains of custody	Following preparation, COC forms will be reviewed against the samples packed in the specific cooler prior to shipment. The reviewer will initial the form. An original COC will be sent with the samples to the laboratory, while copies will be retained for the Sampling Trip Report and the project files.	Internal	Alex daSilva, LaBella Associates, D.P.C. (Preparation); Drew Brantner, LaBella Associates, D.P.C. (Review)
Laboratory analytical data package	Data packages will be reviewed/verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal.	Internal	Alpha
Laboratory analytical data package	Data packages will be reviewed as to content and sample information upon receipt by the Environmental Consultant Project Manager and the Third Party Data Validation Personnel.	Internal /External	Drew Brantner, LaBella Associates, D.P.C.; Staff at Laboratory Data Consultants, Inc.
Final Sample Report	The project data results will be compiled in a sample report for the project. Entries will be reviewed/verified against hardcopy information.	Internal	Drew Brantner, LaBella Associates, D.P.C.

**Brownfields QAPP Template #13b
Project Data Validation Process (Steps IIa and IIb)**

During the course of the Remedial Action, the processes listed below will be performed to validate project data.

Step IIa/IIb ¹	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	SOPs	Ensure that the sampling methods/procedures outlined in QAPP were followed, and that any deviations were noted/approved.	Drew Brantner, LaBella Associates, D.P.C.
IIb	SOPs	Determine potential impacts from noted/approved deviations, in regard to PQOs.	Drew Brantner, LaBella Associates, D.P.C.
IIa	Chains of custody	Examine COC forms against QAPP and laboratory contract requirements (e.g., analytical methods, sample identification, etc.).	Drew Brantner, LaBella Associates, D.P.C., and Laboratory Data Consultants, Inc.
IIa	Laboratory data package	Examine packages against QAPP and laboratory contract requirements, and against COC forms (e.g., holding times, sample handling, analytical methods, sample identification, data qualifiers, QC samples, etc.).	Drew Brantner, LaBella Associates, D.P.C., and Laboratory Data Consultants, Inc.
IIb	Laboratory data package	Determine potential impacts from noted/approved deviations, in regard to PQOs. Examples include PQLs and QC sample limits (precision/accuracy).	Drew Brantner, LaBella Associates, D.P.C., and Laboratory Data Consultants, Inc.
IIb	Field duplicates	Compare results of field duplicate (or replicate) analyses with RPD criteria	Drew Brantner, LaBella Associates, D.P.C., and Laboratory Data Consultants, Inc.

**Brownfields QAPP Template #13c
Project Matrix and Analytical Validation (Steps IIa and IIb) Summary**

The matrices, analytical groups, and concentration levels that Laboratory Data Consultants, Inc. will be responsible for, as well as criteria that will be used to validate those data, includes:

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa / IIb	Soil	VOCs	Low	Data Validation SOP for Analysis of Low/Medium Concentration VOCs under SOW Revision 1 (HW-33A)	Laboratory Data Consultants, Inc.
IIa / IIb	Soil	SVOCs	Low	Data Validation SOP for Analysis of Low/Medium Concentration SVOCs under SOW Revision 1 (HW-35A)	Laboratory Data Consultants, Inc.
IIa / IIb	Soil	Metals	Low	Data Validation SOPs for the evaluation for the CLP Program under SOW Revision 1 (HW-3a, 3b, 3c)	Laboratory Data Consultants, Inc.

Brownfields QAPP Template #13d Usability Assessment (Step III)

Prior to reliance on and incorporation of the data generated during the Remedial Action into the final reports, the usability of data must be determined. The procedures/methods/activities that will be used to determine whether data are of the right type, quality and quantity to support environmental decision-making for the project are discussed below. Table 1 at the end of this section identifies the data assessment activities that may occur during the Remedial Action.

This section also describes how data quality issues will be addressed and how limitations on the use of the data will be handled.

Usability Assessment Responsibility

The Environmental Consultant Project Manager, Drew Brantner (LaBella Associates, D.P.C.), and the third-party data validator, Laboratory Data Consultants, Inc., will be responsible to assess the usability of the data generated during the course of the USEPA-funded Site Remediation.

Data Usability Assessment Process and Procedures

The data generated during the course of the project will be evaluated against the Data Quality Objectives (DQOs), which are the quantitative and qualitative terms used by USEPA to describe how good the data needs to be in order to meet the project's objectives. DQOs for measurement data (referred to here as data quality indicators) are precision, accuracy, representativeness, completeness, comparability, and measurement range. The overall QA objective for analytical data is to ensure that data of known, acceptable and legally defensible quality are generated. To achieve this goal, data must be reviewed for 1) precision, 2) accuracy or bias, 3) representativeness, 4) comparability, and 5) completeness. These qualities are discussed below:

Precision and Accuracy

Precision is the degree of agreement among repeated measurements of the same characteristic, or parameter, and gives information about the consistency of methods. Accuracy is a measure of confidence that describes how close a measurement is to its "true" value. Replicate measurements will be performed during each testing event, monitoring and training sessions and during annual performance evaluation and re-certification. Replicate analysis acceptability criteria and applicability for each environmental measurement are described in the method's SOP.

Field analytical precision will be evaluated by the relative percent differences (RPD) between field duplicate samples and/or replicate readings using the following formula:

$$RPD = \frac{(R1 - R2)}{((R1 + R2)/2)} \times 100$$

Where: R1 = the larger of the two replicate values
R2 = the smaller of the two replicate values

Field accuracy will be routinely checked according to the instrument and analytical method accuracy requirements of each parameter.

Commercial laboratory Accuracy and Precision: QC samples for accuracy and precision in a laboratory setting may include the analysis of the following: duplicate samples, laboratory control check and laboratory control check duplicate samples (LCS/LCSD) and/or matrix spike and matrix spike duplicate (MS/MSD) sample analyses and addition of surrogate spike. LCS and LCSD analyses are blank samples (from the lab) injected (spiked) with a known concentration of target compounds processed on the same date as the routine samples and analyzed with the routine samples. LCS/LCSD are usually performed in cases where insufficient amount of routine samples are available for the MS/MSD QC analyses. MS/MSD analyses are routine samples injected with a known concentration of target compounds processed on the same date and same way and analyzed with the routine samples. Surrogate spike is a compound that is not one of the target compounds but belongs to same chemical category and has the same characteristics as the target compounds. Accuracy are determined by calculating the recoveries (%R) of the target compounds spiked into the LCS/LCSD and/or MS/MSD samples or the surrogate spiked into the sample using the following formula:

$$\% R = \frac{SQ - NS}{\text{Spike}} \times 100$$

Where:

%R = percent recovery

SQ = the concentration of the spiked compound measured in the routine or blank sample

NS = concentration of the target compound native to the unspiked routine or blank sample

Spike = the concentration of the target compound spiked in the routine or blank sample

Laboratory precision is calculated as follows:

$$RPD = \frac{(R1 - R2)}{((R1 + R2)/2)} \times 100$$

Where:

RPD= Relative Percent Difference and

R1 and R2 are the initial and duplicate measurement values, respectively

In case of MS/MSD and/or LCSD/LCSD

R1 = % Recovery of the target compound in the initial analysis (from MS or LCS)

R2 = % Recovery of the target compound in duplicate analysis (from MSD of LCSD)

Data Representativeness

Representativeness is the extent to which measurements actually represent the true environmental condition. It is the degree to which data from the project accurately represent a particular characteristic of the watershed that is being tested. Representativeness of samples is ensured by adherence to standard field sampling and measurement and laboratory protocols. The design of the sampling scheme and number of samples for this project provide representativeness of the part of the watershed being monitored. As a whole, representativeness of the samples collected for this project will be determined during data assessment and data interpretation phase.

Data Comparability

Comparability is the degree to which data can be compared directly to similar studies. Using standardized sampling, analytical methods and units of reporting with comparable sensitivity helps ensure comparability. The USEPA-funded Site Investigation will select testing methods that are EPA-approved and/or currently being employed by other water quality monitoring programs throughout the country. Efforts will be made to duplicate the effort of past studies where possible.

Data Completeness

Completeness is the comparison between the amounts of usable data collected versus the amount of data called for in the sampling plan. Completeness is the percentage of valid results obtained compared to the total number of samples taken for a parameter. The target completeness goal for this project shall be 75% or better. %Completeness is calculated using the following formula:

$$\% \text{ Completeness (per parameter)} = \frac{\# \text{ of valid results}}{\# \text{ of samples taken}} \times 100$$

Assessment of Analytical Results

In addition to the evaluation above, the actual analytical results will be used to help determine usability. This procedure includes determining if any detectable amounts of contaminant(s) are present.

- If no detectable amounts are indicated and all data are acceptable for the verification and validation, then the data is usable.
- If verification and validation are not acceptable then corrective action will be necessary. These actions may include determining the cause and the impact to the data; evaluating the impact; and documenting the rationale for corrective action, such as resampling.

Data Usability Reporting

The RCCR will describe the methods and results of the data usability assessment, and incorporate the findings into the discussion of the data generated for the project. The assessment of data usability will be also documented in a DUSR, which will be appended to the final RCCR.

Table 13-D

Data Elements for Data Review Process				
Item	Step I - Data Verification	Step IIa - Data Validation Compliance	Step IIb - Data Validation Comparison	Step III - Data Usability
Planning Documents				
Evidence of approval of QAPP	X			Use outputs from previous steps
Identification of personnel	X			
Laboratory name	X			
Methods (sampling & analytical)	X	X	X	
Performance requirements (including QC criteria)	X	X		
Project quality objectives	X		X	
Reporting forms	X	X		
Sampling plans – locations, maps grids, sample ID numbers	X	X		
Site identification	X			
SOPs (sampling & analytical)	X	X		
Staff training & certification	X			
List of project-specific analytes	X	X		
Analytical Data Package				
Case narrative	X	X	X	Use outputs from previous steps
Internal lab chain of custody	X	X		
Sample condition upon receipt, & storage records	X	X		
Sample chronology (time of receipt, extraction/digestion, analysis)	X	X		
Identification of QC samples (sampling /lab)	X	X		
Associated PE sample results	X	X	X	
Communication Logs	X	X		
Copies of lab notebook, records, prep sheets	X	X		
Corrective action reports	X	X		
Definition of laboratory qualifiers	X	X	X	
Documentation of corrective action results	X	X	X	
Documentation of individual QC results (e.g., spike, duplicate, LCS)	X	X	X	
Documentation of laboratory method deviations	X	X	X	
Electronic data deliverables	X	X		
Instrument calibration reports	X	X	X	
Laboratory name	X	X		

Laboratory sample identification no.	X	X		
QC sample raw data	X	X	X	
QC summary report	X	X	X	
Data Elements for Data Review Process				
Raw data	X	X	X	Use outputs from previous steps
Reporting forms, completed with actual results	X	X	X	
Signatures for laboratory sign-off (e.g., laboratory QA manager)	X	X		
Standards traceability records (to trace standard source form NIST, for example)	X	X	X	
Sampling Documents				
Chain of custody	X	X		Use outputs from previous steps
Communication logs	X	X		
Corrective action reports	X	X	X	
Documentation of corrective action results	X	X	X	
Documentation of deviation from methods	X	X	X	
Documentation of internal QA review	X	X	X	
Electronic data deliverables	X	X		
Identification of QC samples	X	X	X	
Meteorological data from field (e.g., wind, temperature)	X	X	X	
Sampling instrument decontamination records	X	X		
Sampling instrument calibration logs	X	X		
Sampling location and plan	X	X	X	
Sampling notes & test pit logs	X	X	X	
Sampling report (from field team leader to project manager describing sampling activities)	X	X	X	
External Reports				
External audit report	X	X	X	Use outputs from previous steps
External PT sample results	X	X		
Laboratory assessment	X	X		
Laboratory QA plan	X	X		
MDL study information	X	X	X	
NELAP accreditation	X	X		

————— END OF DOCUMENT —————



APPENDIX A

Staff Resumes



DREW BRANTNER

Project Manager

Drew is an environmental project manager responsible for the coordination and successful completion of a wide range of environmental investigation and remediation projects. Drew's background includes Phase I Environmental Site Assessments (ESAs), Phase II ESAs, NYSDEC Brownfield Cleanup Program projects and NYSDEC Spills projects.

EDUCATION

Muhlenberg College:
Environmental Science and
Chemistry, BS

CERTIFICATIONS

OSHA 30-Hour Construction

OSHA 40-Hour HAZWOPER
Construction

NYSDEC Mold Assessor

NYSDEC Asbestos Building
Inspector

Former Gas Station (NYSDEC Brownfield Site), Periodic Groundwater Monitoring: Syracuse, New York

Drew reviewed and compiled historical sampling data and performed or directed field sampling, prepared quarterly monitoring reports, and assisted with the planning, coordination, inspection, and completion of annual summary reports necessary for maintaining the site's status in the NYSDEC Brownfield Cleanup Program.

Vacant Commercial Property (NYSDEC Brownfield Site), Remedial Investigation: Syracuse, New York

Mr. Brantner reviewed and compiled historical sampling data, assisted with the development of a sampling plan, and performed field tasks associated with executing a Remedial Investigation of a NYSDEC Brownfield Site. Prepared the Remedial Investigation Report and assisted with the Remedial Alternatives Analysis.

Abandoned Industrial Facility (NYSDEC Brownfield Site): Johnson City, New York

Drew assisted with the Brownfield Cleanup Program Application process and development of a Supplemental Remedial Investigation Work Plan. Assisted with field tasks and reporting of findings.

Confidential Client, Former Penfield Manufacturing Facility (Moyer Carriage Lofts), Phase I ESA, Asbestos & Hazardous Materials Survey, and Subsurface Investigation: North Salina Street, Syracuse, New York

Drew coordinated the completion of a Phase I ESA and subsequent asbestos, lead-based paint, and PCB caulk building materials surveys, and limited subsurface investigation, for a 200,000+ square foot abandoned manufacturing facility. Assisted with the completion of summary reports, site plans, and building drawings documenting all sample locations.

Confidential Client, Former Industrial Site, Phase I ESA & Surface Soil Investigation: 255 Ship Canal Parkway, Buffalo, New York

Drew reviewed historical records, including investigative assessments and cleanup reports, completed a Phase I ESA, and coordinated the completion of a Phase II ESA involving the investigation of a multi-acre parcel of land contaminated by SVOCs, heavy metals, and PCB. Used GPS, AutoCAD, and related computer software to assist in determining sample locations. Collected soil samples for SVOCs, metals, and PCB analysis and prepared a summary report for a prospective developer.

Confidential Client, Vacant Facility, Soil, Groundwater, and Vapor Intrusion Investigation: Fulton, New York

Mr. Brantner oversaw a targeted GPR survey, collected soil and groundwater samples, and performed vapor intrusion sampling to determine whether TCE contamination from an adjacent Brownfield site was impacting the indoor air quality of a multiple-unit residential facility.

Confidential Client, Former Manufacturing Facility, Phase I & II Environmental Site Assessment: 1900 Bleeker Street, Utica, New York

Drew completed a Phase I ESA for a 200,000+ square foot manufacturing facility and adjacent lot. Identified environmental concerns were further assessed by implementing multiple investigation activities that included: indoor air quality sampling, ground penetrating radar (GPR), exploratory excavation, soil borings and sampling, and the installation and sampling of temporary groundwater monitoring wells.

Confidential Client, Industrial Park, Surface Soil Investigation: DeWitt, New York

Drew assisted with the completion of a Phase II ESA involving the investigation of a multi-acre undeveloped parcel of land contaminated by PCBs. Used GPS and related computer software to assist in determining sample locations and to define the extent of contamination. Collected soil samples for PCB analysis and assisted with writing the final investigation report provided to the NYSDEC, for consideration of the project entering the Brownfield Program.

City of Utica, Demolished Manufacturing Facility, Phase II Environmental Site Assessment: 1732 Erie Street, Utica, New York

Drew developed a multi-step investigative plan to address multiple contaminants from different sources across the 5-acre Site, which had previously been home to a large manufacturing facility. A widespread GPR survey and comprehensive soil and groundwater sampling plan was implemented to identify and then delineate the various contaminant plumes located on the Property.

Confidential Client, Former Gas Station, Subsurface Investigation & Remedial Oversight: 1600 Erie Boulevard East, Syracuse, New York

Mr. Brantner assisted with tank registration and closure, subsurface soil and groundwater sampling, the preparation of a remedial work plan, provided excavation oversight, and coordinated waste transport during remedial excavation at a former gas station. Worked closely with the Owner, Contractor, and NYSDEC officials during the project.

Confidential Client, Redevelopment of Former Industrial/Commercial Facilities for Residential Use, Asbestos & Hazardous Materials Survey, Vapor/Radon Mitigation System Design

Drew assisted real estate and redevelopment firms with redeveloping various former commercial and industrial properties for future residential and/or commercial use. Projects often include hazardous building material surveys prior to renovation, and assisting with the design of sub-slab depressurization systems to mitigate radon and/or other vapor intrusion concerns.

Confidential Client, Construction Management of Excavated Materials: Rochester, New York

Drew prepared and executed a pre-construction soil sampling plan and evaluated the obtained data so that soil spoils generated during the construction project were properly handled for minimal costs. Impacted soils were profiled and accepted for use as cover material at a local landfill, while non-impacted (native) soils were able to be transferred to a 3rd Party Site meeting the requirements of NYSDEC Part 360 Regulations.

Norry Management, Commercial Warehouse: 100 Mushroom Boulevard, Henrietta, New York

Coordinated sub-slab communication testing and design of a subsurface depressurization system (SSDS) for the 20,000+ square foot facility. Oversaw installation of the SSDS and performed post-construction testing to confirm efficacy of the system.

Lead and Copper in Water Testing, Multiple K-12 Schools, Upstate New York

Drew has prepared drinking water sampling plans in accordance with USEPA regulations for multiple K-12 schools throughout upstate New York. Assisted with the execution of the sampling plans and prepared summary reports based on the results.

Residential Well Sampling, Town of Ridgeway, New York

Prepared sampling plan in accordance with NYSDOH guidance for multiple residences within the municipality. Coordinated field staff and scheduling of sampling events. Reviewed data and prepared summary report.



ANN BARBER

Project Manager

Ann is a Project Manager and an Environmental Engineer responsible for coordination and successful completion of environmental investigation and remediation projects and has managed numerous Phase II Environmental Site Assessments (ESAs). Project experience includes Phase I and Phase II ESAs, NYSDEC Brownfield Cleanup Program projects and NYSDEC Spills projects. Ann is proficient in ArcGIS and is 40 hour OSHA HAZWOPER certified.

PE

Professional Engineer, NY

EDUCATION

Stevens Institute of Technology: Bachelors in Environmental Engineering, minors in Green Engineering and Science Communication

Marist College: Masters in Business Administration, Concentration in Ethical Leadership

CERTIFICATION

40-hour OSHA HAZWOPER Certified

ORGANIZATIONS

American Academy of Environmental Engineers and Scientists (AAEES)

Electrical Resistance Heating Project, Manufacturing Facility, Henrietta, NY

Ann was the project engineer responsible for implementing ERH at a former manufacturing facility in Henrietta, NY. The approximate 2,000 square foot ERH treatment area was designed to treat chlorinated solvents in soil and groundwater. The project is in the NYSDEC Brownfield Cleanup Program. Ann was responsible for BCP project deliverables including work plans, Construction Completion Reports, Site Management Plan and progress reports to the NYSDEC.

City of Rochester, Former United Cleaners, 68-92 Genesee Street, Rochester NY

Ann was responsible for managing a Site Investigation and four Interim Remedial Measures at the former dry cleaning and automotive repair facility. The Site Investigation included overburden and bedrock evaluations as well as on-site and off-site soil vapor intrusion investigations. Interim Remedial Measures included removal of underground storage tanks, excavations of metals and petroleum impacted soil as well as installation of a sub-slab depressurization system in the on-site building.

Rochester Housing Authority, 100 Fernwood Ave, Rochester, NY

This site was in the NYSDEC Brownfield Cleanup Program and received a Certificate of Completion in 2009. Ann is responsible for managing annual monitoring and reporting requirements per the Brownfield Cleanup Program including groundwater monitoring, light non-aqueous phase liquid monitoring and removal, period review reports, and annual inspections of engineering controls.

City of Rochester: Phase II ESA and Tank Closure, 32 Webster Avenue - Rochester, NY

Coordinated and conducted a Phase II ESA and removal of two underground storage tanks and impacted soil removal at a former gasoline filling station. Ann was responsible for developing work plans, implementing field work tasks, and completing Phase II ESA and Tank Closure Reports.

City of Rochester: CERCLA Investigation, 527 River Street - Rochester, NY

Completed a CERCLA Site investigation for a vacant land. Was responsible for completion of field work including soil and groundwater sampling,

preparation of summary reporting, an Engineering Evaluation and Cost Analysis, and public notices.

City of Batavia, Wastewater Treatment Facility Improvements - Batavia NY

Designed upgrades to headworks treatment processes at a lagoon WWTF in Batavia. Upgrades included new grit classifier system and new influent screen. Ann was responsible for design, and construction observation and administration.

Anderson Acquisitions LLC: Davis-Howland Oil Corporation - Rochester NY

Responsible for design and oversight of 4 sub-slab depressurization systems at NYSDEC Superfund Site. Completed design and construction completion documentation for the former industrial site including air sampling and system effectiveness testing.

City of Rochester: Former Emerson Street Landfill - Rochester, NY

Designed various phases of a remedial investigation at a former landfill and implemented Remedial Investigation fieldwork including drilling oversight, soil sampling, passive diffusion bag sampling, test pitting, pumping tests, and GPS locating. Developed a successful Delisting Petition for a portion of the landfill from the NYSDEC's registry of Inactive Hazardous Waste Disposal Sites. Conducted a soil vapor intrusion investigation across several properties and provided oversight and reporting for installation of mitigation systems in several buildings. Designed and implemented a Pilot Test consisting of a permeable reactive barrier and completed reporting tasks including a remedial investigation

Report and Feasibility Study. Ann was responsible for management of project GIS files and organization of GIS data into Geodatabases. Generated 2D and 3D models of the conceptual site model in GIS.

Corning Hospital: Former Corning Hospital BCP - Corning, NY

Conducted a Phase II Environmental Site Assessment at a former hospital and eight associated adjacent parcels for admittance into the NYSDEC Brownfield Cleanup Program. Was responsible for conducting all field work tasks including soil boring logging, soil and groundwater sampling, and test pit evaluation. Evaluated laboratory data and organized the findings into a detailed report including GIS mapping. Designed a Remedial Investigation based on the initial assessment and organized the remedial investigation findings into a detailed report.

Canandaigua Lakefront, LLC: Canandaigua Multi-Brownfield Site - Canandaigua, NY

Completed NYSDEC required reporting and documentation of several Interim Remedial Measures for the Brownfield Cleanup Program site to obtain a Certificate of Completion. Responsible for sub-slab depressurization system designs and final reporting including a Construction Completion Report, Final Engineering Report, and Site Management Plan.

Mark IV Enterprises: Former Monoco Oil Brownfield Cleanup Program Site - Pittsford, NY

Developed and implemented a Remedial Action Work Plan for a 7 acre parcel in Pittsford. Responsible for generating final reports to obtain a Certificate of Completion from the NYSDEC

including a Construction Completion Report, Final Engineering Report, and Site Management Plan.

Town of Royalton: Wastewater Treatment Facility Improvements - Royalton, NY

Designed upgrades to a wastewater treatment facility including a new influent screen, secondary clarifiers, oxidation ditch aerators, and replacement of other equipment including sludge pumps and flow meter. Ann was responsible for developing contract drawings and specifications.

Town of Niagara Landfill, Client Nexamp

This project involves design and construction of a solar array on a portion of a 16-acre former landfill. Ann was responsible for development of portions of an Engineering Report in accordance with NYSDEC's guidance for photovoltaic solar projects at closed landfills and order on consent. Responsibilities included development of a Post-Closure Monitoring and Maintenance Manual.

Lancaster Landfill, Client AC Power

Ann is assisting with a cover evaluation on a closed landfill to delineate the area of the landfill cap. Responsibilities include development of a work plan to evaluate cover thickness and characteristics and correspondence with NYSDEC.

City of Rochester, Former Emerson Street Landfill: Rochester, NY

Ann worked to declassify a 13 acre portion of the Former Emerson Street Landfill from the Inactive Hazardous Waste Disposal Site to facilitate installation of a solar array. Ann developed and implement a

site management plan including community air monitoring and soil characterization utilized during excavation for utilities associated with the solar array. Ann is assisting the City with remediation of a portion of the former landfill immediately adjacent to the existing solar array which is contaminated with chlorinated volatile organic compounds.

City of Rochester: Institutional Control Program - Rochester, NY

Assisted the City of Rochester with development of their Institutional Control Program. Worked closely with the City to collect and develop Site Management Plans and site maps for over 175 properties in the City of Rochester with previous environmental investigations and/or remediation. Created a database for properties with environmental related institutional controls consisting of property information and Site Management Plans for use on the City of Rochester's website.

City of Rochester: Phase II ESA, 177 University Avenue - Rochester, NY

Conducted a Phase II ESA to delineate subsurface contamination in soil and groundwater. Conducted soil boring logging, soil and groundwater sampling, reporting, and GIS data management.

City of Rochester: 68-92 Genesee Street Phase II ESA - Rochester, NY

Designed and implemented a Phase II ESA at a former dry cleaning facility. Ann was responsible for all field work tasks including soil and groundwater sampling, a test pitting study to identify underground

storage tanks and a soil vapor intrusion investigation. Ann was responsible for all reporting and GIS data management.

City of Rochester: Phase II ESA, 310 Lyell Avenue - Rochester, NY

Completed a Phase II ESA at a portion of the former Rochester Subway and Canal. Researched historic documentation in order to select soil boring and test pit locations. Conducted soil boring logging, soil and groundwater sampling, GIS data management, and reporting.

New York Air Brake: Sub-slab Depressurization System Design, - Watertown, NY

Assisted with the design of a sub-slab depressurization system for a building addition at NY Air Brake. Was responsible for drafting specifications and construction drawings. Oversaw and documented the system installation.

Norry Management Corp.: Brownfield Cleanup Program, Monroe Avenue - Rochester, NY

Developed a Remedial Investigation Work Plan for implementation at a former industrial facility in the NYSDEC Brownfield Cleanup Program. Developed conceptual site models of historic soil and groundwater data.

MTA: Waste Minimization Plan - New York, NY

Developed a waste minimization plan report for a large quantity generator by analyzing quantities and types of waste streams. Compared annual data from previous years and compiled tables to display data in a detailed report.

City of Rochester: Pump and Treat Groundwater Treatment System Reporting - Rochester, NY

Compiled annual reports for a groundwater treatment system in order to meet regulatory agency requirements. Compiled and interpreted over a decade worth of analytical data to create graphs and identify emission and concentration trends over time. Compiled graphs and summarized findings into detailed reports.

Pre-Development Site Assessment, Kodak Park South - Rochester, NY

Conducted a pre-development site assessment for an approximate 122 acre former industrial site. Ann was responsible for soil and groundwater sampling and GIS data management. Organized the findings of this study and previous environmental studies conducted at the site in a detailed report.



DANIEL NOLL

Vice President

Dan has more than 24 years of experience with environmental projects at industrial/manufacturing facilities and environmental investigation projects for a variety of clients including developers, financial institutions, industrial clients, and municipalities. Dan has managed numerous Phase II Environmental Site Assessments and remediation projects such as groundwater monitoring programs, soil vapor investigations, test pit investigations, geo-probe investigations, underground storage tank removals, soil removals, bio-cell remediations, and in-situ groundwater remediation. He also has experience with the design and installation oversight of mitigation systems. In addition, Dan has assisted industrial, municipal and agricultural clients with permitting and annual reporting for State Pollution Discharge Elimination System (SPDES) permits, Part 360 Land Application permits, Composting permits, and Petroleum Bulk Storage (PBS) registrations.

Compliance Bio:

Dan has more than 24 years of experience with environmental compliance/audits and investigation and remediation projects at industrial/manufacturing and municipal facilities. Dan has worked with a large variety of manufacturing clients from food processing facilities to heavy industrial facilities like steel manufacturing. Dan has worked with all manner of clients to assess their operations and determine applicable regulations and compliance programs applicable to their specific work and location. Dan has assisted clients with a wide variety of permitting including, National and State Pollution Discharge Elimination System (NPDES/SPDES) permits, Petroleum Bulk Storage (PBS) permits, Chemical Bulk Storage (CBS) Permits, Resource Conservation and Recovery Act (RCRA) permits, air permits, Land Application permits, Composting permits, etc., Dan has also worked with clients to develop programs and compliance plans for their facilities including Spill Prevention, Control and Countermeasure (SPCC) Plans, Stormwater Pollution Prevention (SWPP) Plans, Hazardous Waste Contingency Plans, Spill Prevention Reports and other similar compliance plans.

PE

Professional Engineer: NY, ME, OH, NH, AZ, CO, CT, IA, KS, MA, ND, OR, SD, WA

EDUCATION

Clarkson University: B.S. in Chemical Engineering

CERTIFICATIONS/ REGISTRATIONS

OSHA 40-Hour Certified Hazardous Waste Site Worker Training

OSHA 8-Hour Certified Hazardous Waste Site Worker Refresher Training

Wollensack Optical: Rochester NY

Mr. Noll served as the overall engineer in charge of the investigation and remediation work at the former Wollensack Optical facility. The site was entered into the NYSDEC Brownfield Program to address contamination that resulted from historical operations the site. The remedial investigation identified

orphaned underground storage tanks and associated petroleum impacted soil and groundwater, chlorinated solvent impacts to soil and groundwater and radioactive building materials. Mr. Noll guided the technical aspects of the investigation work which included delineation of a chlorinated solvent groundwater plume that extended from the overburden and into a fractured



bedrock network. Subsequent to completing the investigation work, Mr. Noll served as the engineer of record for the remedial analysis and the remedial action work plan. The selected remedy included in-situ chemical treatment to address chlorinated solvents, removal of underground tanks and a sub-slab depressurization system to mitigate potential exposure concerns. The remedial work allowed for the redevelopment of the building into an affordable housing complex.

Electrical Resistance Heating Project, Getinge Manufacturing Facility, Henrietta NY

Dan served as the overall engineer in charge and certifying engineer for the design, installation and operation of an electrical resistance heating system to remediate a source area of chlorinated solvents beneath a former manufacturing building. The ERH approach was selected in order to rapidly and effectively remove significant mass from the source area materials. The ERH operated for 80 days and removed an estimated 168 pounds of Trichloroethylene. The ERH was supplemented with an injection of an amendment to provide further long-term treatment and allow for natural attenuation monitoring as part of the overall remedy. This project successfully obtained a Certificate-of-Completion through the NYSDEC Brownfield Program.

Brenneman Industrial: Oswego NY

Mr. Noll was the engineer of record for the investigation and remediation work at the former Brenneman industrial facility. The site was identified

as a 'catalyst' site through the NYSDEC Brownfield Opportunity Area program and based on that a developer put the site into the NYSDEC Brownfield Program. The remedial investigation identified a plume of chlorinated solvents and significant fill material at the site. Mr. Noll led the team that evaluated remedial options and selected the remedy for the site. Mr. Noll provided technical oversight during the remedy implementation phase and during site management phase as part of the redevelopment of the site. The site was successfully redeveloped into an affordable housing building filling a need for the community.

Marketview Park: Ithaca NY

Mr. Noll served as the technical manager for an affordable housing/commercial redevelopment project in Ithaca NY. The site had significant urban fill material identified during a routine due diligence project. The funding source (Housing and Community Renewal) required specific actions be implemented during construction to allow for redevelopment for the intended use. Due to significant cost for removal and disposal of these materials, Mr. Noll led a project team that developed a beneficial use determination (BUD) for the reuse of the urban fill material that was encountered during construction. Ultimately several 'BUDs' were obtained to minimize disposal cost and allow the project to move forward.

PFAS Investigation at Former Landfill – Orleans County, NY

Mr. Noll managed a project to assess a former landfill in Orleans County NY for Per and Polyfluoroalkyl Substances (PFAS). Due to concerns with the landfill closure (1980s), the

NYSDEC required sampling of nearby residential drinking water wells and an assessment of the soil and groundwater at the landfill. Mr. Noll coordinated an assessment of drinking water wells in proximity of the landfill. Municipal water serviced a majority of the area but four residences still utilized private wells. Mr. Noll coordinated sampling with the NYSDOH, NYSDEC, Orleans County DOH and the property owners. In addition, Mr. Noll managed soil and groundwater sampling within and around the landfill to assess for PFAS sources.

PFAS Investigation at Former Landfill – Palmyra, NY

Mr. Noll currently is managing a project to assess a former landfill in Palmyra NY for Per and Polyfluoroalkyl Substances (PFAS). The landfill was closed in the late 1970s. NYSDEC conducted an initial testing program and identified elevated levels of PFAS in groundwater. Mr. Noll has been working with the Town to evaluate nearby residences for private wells and public water availability. Mr. Noll is also managing an assessment of the landfill history and subsequent to completing that assessment a detailed investigation will be completed to determine any remedial actions required.

PFAS at Brownfield Sites – Various Locations, NY

The NYSDEC is currently undergoing a statewide assessment of Per and Polyfluoroalkyl Substances (PFAS) in groundwater. As part of that assessment NYSDEC has been requesting that active and former Brownfield sites be assessed for PFAS across

the State of New York. This program resulted in numerous old and active remedial sites being further investigated. Mr. Noll was the project manager for over 15 Brownfield sites in NY where such testing was requested. Mr. Noll negotiated the details of the sampling and managed/coordinated the field activities and reporting. In addition to PFAS NYSDEC also required conducting emerging contaminant testing for 1,4-Dioxane.

Former Rock Quarry Water Sampling – Cortland, NY

Mr. Noll coordinated a project to characterize quarry water as part of a larger construction project. The former quarry filled with water after operations ceased. A large natural gas pipeline was being installed near the quarry and required ballast water for the pipeline installation. Mr. Noll coordinated the approvals for baseline sampling of the water through the Town of Cortland who owned the quarry. The sampling included contaminants of concern including Per and Polyfluoroalkyl Substances (PFAS). Mr. Noll negotiated the sampling requirements/scope and coordinated implementation with internally and with the natural gas company, Town and contractor. The sampling included baseline and post discharge of the ballast water to confirm there was no impact to the water since the Town was exploring possible future uses of the quarry.

City of Hornell – Wastewater Plant Aeration Basin Upgrades

Mr. Noll was the project manager for assessing and implementing replacements for the aeration basin aerators. The City's aeration basins had not been upgraded in almost 30 years and the aging

equipment was past its useful life. Mr. Noll worked with the City to assess potential replacement equipment and coordinated a performance contract approach to complete the aeration equipment upgrades. Mr. Noll worked closely with the chief operator to assess the preferred equipment in order to make sure that the equipment would not only meet the process/treatment requirements but to take into account the long-term maintenance and operations for a facility that will utilize the equipment for the next 30 years. Mr. Noll and the LaBella team assessed numerous types of aeration equipment and assisted with selection of the equipment. Mr. Noll also worked with the City to conduct construction administration activities to ensure a successful completion of the project.

City of Hornell – Wastewater Plant Phosphorus Removal Program

Mr. Noll was the project manager to assist the City of Hornell with completing the New York State mandated actions for removal of phosphorus from the wastewater. Initially, Mr. Noll worked with the City of Hornell to evaluate potential chemicals for use in removing phosphorus. Mr. Noll coordinated bench-scale studies with chemical suppliers to assess performance and cost of the chemicals. Based on the bench-scale studies a pilot-test was developed and proposed to NYSDEC. The pilot test was approved and implemented and the results were utilized to design and bid for construction a new chemical feed building. The design included a pre-fabricated building to house the chemicals and associated equipment

(chemical feed pumps, day tank, piping, and controls). Mr. Noll also worked with the City to bid the project in such a way that the City could self-perform some of the construction work and reduce the overall cost of the project. The project was successful in utilizing alum in reducing the WPCP effluent phosphorus concentration to one (1) mg/l to meet new limits in the State Pollution Discharge Elimination System (SPDES) permit.

City of Hornell – Wastewater Plant Filter Building and Drive Upgrades

Mr. Noll worked with the City to apply for funding to complete upgrades to aging equipment. The drives providing mixing for numerous tanks were over 30 years old and beyond their useful life. Mr. Noll worked with the City to obtain the information on the aging drives and coordinate with replacement of similar equipment. Mr. Noll coordinated with the City to assess the sequencing of drive replacements to ensure that the plant processes would be maintained throughout the construction work so that effluent limits would be met. This project also included replacing filter blocks on the sand filter equipment. Similar to the drives, the filter building had not been upgraded in over 30 years and the blocks required replacement. Mr. Noll led the project to provide design drawings, bid specs and work with the City to bid and award the project. Mr. Noll also further assisted the City with the construction administration services.

Enbridge (Spectra Energy, LP): Gas Pipeline Characterization Work

Mr. Noll has worked with Enbridge to coordinate/oversee a program that characterizes natural gas

pipings that has been removed from service. Mr. Noll managed the program to characterized the exterior coating of piping (PCBs and asbestos) as well as the piping interior (PCBs). This work has included the characterization of over 25 miles of line piping and numerous pieces of compressor station equipment and associated piping over various projects in the northeast. The work was completed in accordance with applicable Federal regulations (e.g., 40 CFR 761) and state regulations depending on the project site (included New York, Massachusetts, Connecticut, Rhode Island and Pennsylvania).

Enbridge (Spectra Energy, LP): Wastewater Characterization Work

Mr. Noll was the project manager for the characterization of ballast water used as part of a 1-mile horizontal drilling program to install 42-inch diameter natural gas piping beneath the Hudson River. Mr. Noll coordinated with the regulatory agencies to develop the required sampling program and oversaw the collection and analysis for the sampling of approximately 500,000 gallons of ballast water. Based on the sampling completed a treatment system was developed and the water was directly discharged to surface water. LaBella completed this work in a very short timeframe based on the Client's request in order to accommodate the construction schedule.

Enbridge (Spectra Energy, LP): Radiological Characterization Work

Mr. Noll has worked with Enbridge to complete the characterization of suspect radiological materials. Specifically, Mr. Noll has worked with Enbridge to complete

the necessary sampling of natural gas equipment that has been removed from service. LaBella coordinated/ completed radiological surveys (alpha, beta and gamma) in order to preliminarily characterize the material. LaBella also coordinated/ completed the collection of samples and analysis (through a 3rd party laboratory) for waste characterization purposes of materials that warranted such testing. This sampling included gamma spec analysis and other parameters as needed for the disposal facility.

LMC Industrial Contractors: Gas Pipeline Reclamation Facility

Mr. Noll has partnered with LMC Industrial Contractors in order to design and permit a facility that specializes in the recycling of natural gas piping that contains an asbestos coating. LaBella worked with LMC to design the facility and obtained the necessary New York State permits (air permit) and local permits (wastewater discharge). LaBella also oversees the program that completes the waste characterization of the piping for PCBs (exterior coating and interior) and asbestos (exterior coating). The facility has led to the reclamation of steel that may otherwise have been disposed of in landfills or transported at significant expense to facilities in Texas or elsewhere. The facility has taken piping from project sites in New York, Massachusetts, Connecticut, Rhode Island and Pennsylvania.

Confidential Utility Client: SPCC Program

Mr. Noll worked with a private utility client in order to develop a program to complete Spill Prevention, Control and

Countermeasure (SPCC) Plans for approximately 600 electrical substations in New York State. Mr. Noll organized the program and led a team of over forty staff members to complete the inspection of each facility and develop an SPCC Plan for each facility in order to keep the facilities in compliance with Federal Regulations. The project included making recommendations for identifying areas of compliance issues. Mr. Noll worked with the Client on a second phase to implement recommendations at approximately 200 facilities across New York State to ensure compliance with regulations. The recommendations included modifications to routine monitoring and where necessary additional secondary containment.

Repsol (Talisman Energy) – Groundwater Proection Program

Mr. Noll has managed the assessment of groundwater monitoring to assess for the potential of stray gas issues in the Marcellus Shale area of Northern Pennsylvania. The sampling work is includes completion of pre-drill sampling to establish baseline conditions of groundwater for wells within a certain distance from drilling operations prior to the operations occurring. This information is utilized when there is a complaint subsequent to drilling operations in order to evaluate for the potential for stray gas issues related to gas fracturing projects per the Pennsylvania Department of Environmental Protection (PADEP) regulations and additional requirements by the Client. The work includes assessing the areas around drilling sites to establish potential potable water

sources, contacting of residences to confirm potable water sources and then conducting pre-drill sampling for potential contaminants and gases in the potable water that exist prior to drilling operations to confirm baseline conditions. In the event of a complaint, post-drilling samples are collected and compared to pre-drill sampling to determine differences and potential issues. Mr. Noll also assisted with assessing potable water sampling information in order to evaluate and recommend potential treatment systems to address issues identified.

NYS Department of Transportation: Hazardous Materials Assessment & Remediation Term - DOT Regions 3, 4, 5, & 6

Mr. Noll manages a NYSDOT Term Agreement for Hazardous Materials Assessment & Remediation for Regions 3, 4, 5, & 6. This agreement includes a variety of services to support the NYSDOT for all manner of construction projects and for property acquisition. The work includes Phase I & II Environmental Site Assessments to support property acquisitions and/or to pre-characterize soil and groundwater prior to construction in a NYSDOT corridor. Mr. Noll also has assisted NYSDOT with waste characterization of soil, spent paint, and wastewater. In addition, NYSDOT has utilized LaBella for community air monitoring during construction work at impacted properties and to complete radiological screening for areas where radioactive slag has been a concern.

Stern Family Limited Partnership: Former Manufacturing Facility BCP Site - Rochester, NY

Dan was the Project Engineer for this BCP Site, which underwent a Remedial Investigation, Interim Remedial Measures, and installation of a sub-slab depressurization system. Dan completed and stamped the Final Engineering Report required to obtain the Certificate of Completion for the property owner, allowing them to obtain their tax credits.

Springs Land Company: Carriage Cleaners BCP Site - Rochester, NY

As Project Manager, Dan completed a Brownfield Cleanup Program (BCP) Application & Work Plan to conduct a Remedial Investigation at a former dry cleaning facility. A soil, groundwater, and soil gas study was undertaken to develop remedial costs and assist with redeveloping the property. Subsequently, an Interim Remedial Measure was completed to remove the source area of impacts from the Site. Dan completed a remedial alternatives analysis for selecting a treatment approach for the residual groundwater plume. Dan also attended Town Board Meetings regarding this project.

American Siepman Corporation: Former Manufacturing Facility BCP Site - Henrietta, NY

Dan was the Project Manager for this Brownfield Cleanup Program (BCP) Site and has overseen the installation of a groundwater monitoring well network and subsequent routine sampling as part of a Monitored Natural Attenuation (MNA) program for remediation of chlorinated groundwater impacts at the Site.

RJ Dorschel Corporation: Former Gasoline/Service Station BCP Site - Rochester, NY

Dan was the Project Manager for this BCP Site, which included Remedial Investigations at two adjoining parcels, implementation of Interim Remedial Measures, and development of the Final Engineering Report and Site Management Plan. The project also included implementation of necessary Citizen Participation requirements. The project ultimately obtained the Certificate of Completion and thus the NYS tax credits.

One Flint Street Associates: Vacuum Oil BCP Site - Rochester, NY

Dan was the Project Manager for this Brownfield site that is the oldest oil refinery in the United States. The current project includes developing a remedial investigation plan for two parcels that have had a history of oil refining since the 1800s. The remedial investigation was designed to fill data gaps from previous studies in order to minimize cost to the Client.

Genesee Valley Real Estate: Former Bausch & Lomb Facility BCP Site - Rochester, NY

Dan is Project Manager for this Brownfield site that served as a manufacturing facility from the 1930s to the 1970s. The project includes a Remedial Investigation (RI) of a four-acre parcel with ten areas of concern identified based on historic information. The RI identified four areas requiring remedial actions and Interim Remedial Measures have been completed in three of the locations. The areas of remediation included petroleum impacted soil and groundwater with free floating petroleum

product, and chlorinated solvent contamination including bedrock impacts at depth. A remedial alternatives analysis is being completed to determine a final remedy for the site.

Alternative description below:

Dan was Project Manager for this Brownfield site that served as a manufacturing facility from the 1930s to the 1970s. The project included a Remedial Investigation (RI) a four-acre parcel with ten areas of concern identified. The RI identified four areas requiring remedial actions. The remedial areas included petroleum impacted soil and groundwater, free floating petroleum product, and two areas of chlorinated solvent contamination with one including bedrock impacts at depth. A Feasibility Study was completed that evaluated pros/cons and associated cost of each remedial alternative. The remedial work was agreed to with NYSDEC and Dan led the design of the remedial systems for each area. The remedial approach included in-situ chemical oxidation for one of the chlorinated solvent areas through several subsurface injection manifolds. The remediation approach for the other area of chlorinated solvent impacts included the design and installation of bedrock injection wells and a pump and treat groundwater extraction system. The injection wells were utilized to inject zero-valent iron for treatment of the solvents. The pump and treat system was utilized to pull the injection chemicals across the impacted area for greater distribution. The remedial systems were successful and the site received a Certificate of Completion from NYSDEC in 2018.

Former Corning Hospital - Corning, NY

Dan was the project manager for completion of a Phase II Environmental Site Assessment at the Former Corning Hospital and 8 associated adjacent properties. A soil boring and groundwater monitoring program was implemented to identify subsurface impacts associated with former uses of the site including gasoline filling stations and former railroad.

Bajrangee, Inc.: Comfort Inn – BCP Site - Rochester, NY

Dan was the Project Manager for this Brownfield site that included a design phase investigation to determine the extent of remedial work. The remediation work included excavation of chlorinated solvent impacts to soil and groundwater from the basement of the building. This included proper shoring design to facilitate the removal action. A second phase of the remediation included injection of treatment chemicals to address downgradient groundwater impacts.

Former Emerson Street Landfill Redevelopment

Mr. Noll has assisted the City of Rochester since 2010 with managing environmental legacy issues at this 250-acre former ash and municipal landfill. Mr. Noll has worked with the City to conduct environmental investigations at over 45 different parcels across the landfill and identify properties/buildings that require mitigation measures. Mr. Noll has assisted with redevelopment activities at 9 different properties that consisted of pre-construction soil and waste characterization to assist with planning and cost estimating activities, developing

waste management and environmental monitoring plans, obtaining regulatory approvals and implementing these plans during construction activities. This work has assisted with the development of industrial/commercial developments and a 6-acre solar array. The solar array development also included utilizing a Beneficial Use Determination, site plans, geotechnical assessment and delisting the property from the NYSDEC list of inactive hazardous waste disposal sites.

Alternate description below:

Mr. Noll was the project manager and lead design engineer assisting the City of Rochester since 2010 with managing environmental legacy issues at this 250-acre former ash and municipal landfill. Mr. Noll has worked with the City to conduct environmental investigations at over 45 different parcels across the landfill and identify properties/buildings that require mitigation measures due to soil vapor intrusion. The investigation resulted in the design and installation of sub-slab depressurization systems for two buildings at the Site. Mr. Noll then assisted the City with the delineation of a significant chlorinated solvent plume emanating from a portion of the former landfill. The solvent plume is over 3 acres in size and extends almost 50-ft. below grade. Subsequent to completing the investigation, Mr. Noll completed a Feasibility Study to assess remedial options and associated cost. The selected remedy was agreed to by NYSDEC and a Remedial Action Work Plan was approved and implemented in 2021. The remedy included the design and construction of

a Permeable Reactive Barrier utilizing zero-valent iron. The remedy included drilling 80 pilot holes 15 ft. into bedrock and completing blasting of the bedrock in order to create a highly permeable blast enhanced bedrock zone which was used to uniformly distribute over 430,000 lbs of zero-valent iron. The iron was injected through bedrock wells and direct injection within the shallow bedrock. The final remedial work includes construction of a 12.5-acre site cover system which is planned for construction in 2022.

NYSDEC Petroleum Spill Investigation and Remediation Projects

Alexander Associates: Former Genesee Hospital - Rochester, NY

Dan was Project Manager for a Phase II ESA of a former hospital campus and adjoining parking garage. This assessment included evaluating potential impacts from the hospital chemical storage area, backup generators and associated fuel tanks, and historical site uses which included a former car dealership and service center. The Phase II ESA progressed in to the remediation of a NYSDEC Spill prior to redevelopment of the property. The investigation and remediation work obtained closure of a 20+ year old spill in less than 6-months.

DeCarolis Truck Rental: Petroleum Spill Site Remediation - Rochester, NY

Dan was Project Engineer for this site, responsible for the coordination of the removal/disposal of approximately 800 tons of petroleum impacted soil and development of a

confirmatory soil sampling program. Dan also coordinated work with NYSDEC and completed post removal monitoring in order to close the spill file.

City of Rochester: Petroleum Soil Removal & Oxygen Injection System - Rochester, NY

As Project Engineer, Dan developed a soil and groundwater study to investigate former underground storage tanks at a former gasoline/auto repair facility. A remedial alternatives analysis was conducted to evaluate several options for remediating soil and groundwater at the site including light non-aqueous phase liquid. Dan followed this project through remediation which consisted of removing about 1,500 cy of soil and designing/installing an oxygen injection system to remediate groundwater over time.

Hoselton: Petroleum Spill Remediation - Rochester, NY

Dan was project manager for this project which included the removal and disposal of approximately 900 tons of petroleum impacted soil. Dan negotiated closure of the spill file with NYSDEC by addressing off-site contaminant migration by injection of treatment chemicals at the property line.

Permitting & Land Application Sites Mizkan Americas: Lagoon Design/Construction and SPDES Permitting - Lyndonville, NY

Dan served as the Project Manager and Engineer for the design and construction assistance for a 700,000 gallon lagoon to store food-grade wastewater. The objective was to reduce facility costs by discharge of food-grade wastewater to local sprayfields. The lagoon

was designed and installed in accordance with NYSDEC requirements in order to store wastewater during the non-spraying season. This is a 20+ year old client who built their existing lagoon with LaBella's assistance in 1987. Project also includes permitting through NYSDEC SPDES (State Pollution Discharge Elimination System) Program.

Leo Dickson and Sons, Inc.: Land Application and Composting Permits - Bath, NY

Dan managed a project to permit a facility for composting of wastewater biosolids. The project included developing a report for NYSDEC to document design details for the facility, facility operations, and proposed monitoring. The facility received a NYSDEC Part 360 Composting Permit. In addition, Dan continues to provide annual reporting services for ensuring the facility operates within the permit conditions. He also assists this client with the annual reporting and permit renewals of a 2,000+ acre land application project under NYSDEC Part 360 solid waste regulations. The land application work includes permitting approximately 16 municipal facilities for land application.

City of Hornell: Land Application Reporting, Permit Renewals and Modifications - Hornell, NY

Project Manager and Engineer responsible for assisting the City of Hornell with their annual Land Application Reporting, permit renewals and modifications to their permit for over 20 years. In addition to completing each annual report in the past five years, LaBella also recently assisted the City of Hornell with their Permit Renewal (May 2010) and a Permit Modification

(July 2011). LaBella has assisted the City of Hornell for the past 20 years with permitting approximately 498 acres of land for their biosolids application work. Hornell conducts land applications via subsurface injection and typically applies 700,000 to 1 Million gallons annually. In 2011, LaBella assisted Hornell with permitting approximately 204 acres of land. LaBella assisted with all aspects of the process including coordinating with agencies, wetland issues, test pitting, soil sampling, etc. LaBella's work with the City of Hornell has provided us with significant experience in quickly determining issues that require resolution/clarification as a first step prior to completing the application process.

Miscellaneous Projects

Former Emerson Power Transmission Facility - Ithaca, NY

Dan completed a detailed review of this 100-acre site with 800,000 sq. ft. of manufacturing space. The site is in the NYSDEC Inactive Hazardous Waste Disposal Site registry and was a heavy industrial facility for over 100 years. The facility closed in 2009 and Dan is the project manager for environmental due diligence activities for a potential buyer. The facility has known issues with chlorinated solvents in bedrock and with significant off-site impacts. The overall project will include a detailed and in-depth environmental site assessment with sampling for soil, bedrock, groundwater, soil gas, sediments, and surface waters in order to document any impacts above NYSDEC criteria and thus limit liability for the purchaser.

City of Rochester: Genesee River Dredging Project - Rochester, NY

Dan managed a project to permit three areas for dredging near the mouth of the Genesee River. The project included evaluating the previous dredging operations in the area, the existing sediment sampling data, sediment levels, discharge points in the area to be dredged and 3-D modeling of the sediments for accurate volume calculations. This information was summarized in a presentation to NYSDEC and the Army Corp of Engineers in order to streamline the permitting process and determine any additional requirements for obtaining a permit. Subsequent to the presentation, Dan developed the permit and submitted them to the Client for signature, and then approval by regulatory agencies.

MRB Group: Sediment Sampling Project - Erie Canal, NY

Dan managed a project to pre-characterize sediment in the Erie Canal in order to determine the depth and volume of sediment in the work area, as well as the waste disposal requirements. This work was conducted prior to a utility line installation project in order to determine the feasibility of the project and the associated costs.

Dansville Properties, Inc.: Former Foster Wheeler Facility - Dansville, NY

Dan managed the effort to close out existing NYSDEC and EPA permits for the former facility and subsequently obtained permits for the new facility, which included multiple industrial companies operating throughout the campus. The permitting effort included obtaining: a sewer use permit from the local municipality,

a SPDES Multi-Sector General Permit for 5 outfalls, RCRA Generator ID, Title V Air Permit, and PBS Registration. Dan has managed this client's permits for more than 10 years, including permit modifications, renewals, and routine sampling.

Buckingham Properties: Manufacturing Facility - Rochester, NY

Dan assisted a developer that purchased a former Bausch & Lomb manufacturing facility to obtain a SPDES Permit for Industrial Discharges. This project included assessing the new operations and discussion of the Site with NYSDEC to determine the appropriate permits for the facility, since multiple tenants with various operations were in operation at the Site.

City of Rochester: Port Marina - Rochester, NY

Dan assisted with the environmental investigation of the City of Rochester Port Marina. This project included evaluating the extent of slag fill materials that would require proper management during any redevelopment work. The extent of slag was evaluated by implementing a grid pattern of soil borings and using the resulting data to develop a 3-dimensional model of the subsurface at the Site. This model was used to generate volumes of material to be disturbed during redevelopment and estimate the cost burden of the environmental portion of the project. This project also included evaluating the magnitude and permitting of a massive dewatering program to allow the mass excavation to be completed.

City of Rochester: Former Forestry Building - Rochester, NY

Dan managed a project to evaluate the extent of mercury impacts at a former City of Rochester Forestry operations building. The project included multiple rounds of sampling at various depths in order to determine the extent of mercury impacted soils that required removal prior to redevelopment of the Site by a local manufacturing company.

Valeo North America: Former Valeo Facility - Rochester, NY

Dan managed Remedial Investigations of two areas of potential contamination at this former manufacturing facility. These assessments included evaluating bedrock groundwater for plating waste impacts (metals and chlorinated solvents). These evaluations were complicated by the fact that multiple industrial companies were in operation at the Site in the past and thus requiring LaBella to provide a focused assessment to only evaluate potential Valeo responsibilities.

City of Rochester: NYSDEC Legacy Site Soil Vapor Intrusion Project - Rochester, NY

Dan is Project Manager for this project which includes evaluating soil vapor intrusion from a former 230-acre municipal landfill with methane gas and chlorinated solvent impacts. The landfill was converted into an industrial park after closure in 1971 and is now developed with 45 separate parcels and over 2,000,000 square feet of building space. This challenging project included obtaining access from 27 different property owners and conducting site assessments at each facility and separately evaluating

groundwater impacts over approximately 20-acre area. The results of this work determined the cost burden and liability of the City for addressing soil vapor intrusion. LaBella utilized all of the following mitigation approaches for minimizing this significant cost burden to the City: sealing of floors, vapor barriers, sub-slab depressurization systems and building pressurization depending on building conditions/uses.

City of Rochester: Vacuum Oil Brownfield Opportunity Area - Rochester, NY

Dan was Project Engineer for this project and his role was to develop a Pre-Nomination Study Report to facilitate entering the area into the NYSDEC Brownfield Opportunity Area program. The pre-nomination study included evaluating demographics of the area, current and past property uses, property ownership, area-wide utilities, etc. The pre-nomination report was approved by NYS Department of State and a grant was approved for the next phase of the BOA program.

Yates County: Environmental Restoration Program - Penn Yan, NY

Dan was project manager for this Environmental Restoration Program site that included completing a Remedial Investigation at the site and developing a Site Management Plan to guide future redevelopment in-conjunction with remediation. This project turned a liability into an asset for the Count

Monroe County: Crime Lab Property Acquisition - Rochester, NY

Dan was project manager for this project which included conducting Phase I ESAs

and Phase II ESAs at three properties being considered for development by the County for a new crime lab facility. The project included investigation and remedial cost estimates for the County to use in property acquisition negotiations. After property selection, Dan assisted with implementation of a remedial program that included removal of over 3,000 tons of NYSDEC Regulated Solid Waste. In addition, he designed and oversaw installation of a sub-slab depressurization system for addressing soil vapor intrusion concerns at the approximate 11,000 square foot new building.

City of Rochester: Fill Relocation and Sub-Slab Mitigation System - Rochester, NY

Dan was project manager for this project which relocated approximately 3,000 cubic yards of fill material from a development site that is located on a former landfill operated by the City of Rochester. This work was conducted for the City but on private property. The fill was relocated and placed in a soil berm on City property with NYSDEC approval. In addition, Dan designed and oversaw construction of a sub-slab depressurization system for the new 8,000 square foot building.

City of Rochester: Bureau of Water, Lighting, and Parking Meter Operations - Rochester, NY

As Environmental Engineer, Dan worked on the redevelopment of the current site for reuse as a new facility for the operations center, which included the following tasks: delineate the extent of soil and groundwater contamination, evaluate potential remediation options, develop a Comprehensive Action Plan

(CAP), assist in the development of remediation specifications, and identify the scope of potential Interim Remedial Measures (IRMs) at the site.

935 West Broad Street Petroleum Spill Site Characterization and Corrective Action - Rochester, NY

As Project Engineer, Dan developed a soil and groundwater study to investigate former underground storage tanks at a former gasoline/ auto repair facility. A remedial alternatives analysis was conducted to evaluate several options for remediating soil and groundwater at the site including light non-aqueous phase liquid. Dan followed this project through remediation which consisted of removing about 1,500 cy of soil and installing an oxygen injection system to remediate groundwater over time.

Petroleum Spill Investigation & Remediation - 300 Scajaquada Expressway Buffalo NY

Mr. Noll was project manager for a Phase II Environmental Site Assessment that was completed to assess a former manufacturing facility that also included a reported underground storage tank (UST). The Phase II ESA identified an orphan UST with associated petroleum related impacts to soil and groundwater. In addition, the Phase II ESA identified fill material including industrial byproducts consisting of ash, cinders, slag, etc. Based on the petroleum impacts identified the NYSDEC was contacted a Spill File was opened for the parcel. Subsequent to completing the Phase II ESA, LaBella assisted the client with estimating the cost of remediating the Site in order to facilitate the real estate transaction that was pending

for the property. LaBella was also retained to complete the remedial work which consisted of excavation and disposal of petroleum impacted soils and removal of the orphan UST. The work was completed on-time and within budget, which allowed the NYSDEC Spill File to be closed and the real estate transaction to be completed.

Brownfield Redevelopment Project, Covanta Rail-to-Truck Intermodal Facility - Niagara Falls, NY

Mr. Noll was the remedial engineer for the investigation, remediation and redevelopment of a 15-acre former industrial site for use as a Rail-to-Truck Intermodal Facility (RTIF). The project was completed through the Brownfield Cleanup Program (BCP) and involved the completion of a Remedial Investigation (RI); development of a NYSDEC-approved Remedial Action Work Plan to address a range of contamination, including radioactive slag. The project was completed successfully and obtained a Certificate of Completion which allowed redeveloping the property for the proposed use.

USEPA Grant Funded Work

Mr. Noll has worked on numerous EPA funded projects for different clients. This work included conducting investigation and remediation projects at gas stations, dry cleaners, former industrial properties, and railroad yards. Mr. Noll has managed all aspects of these projects including developing Remedial Investigation Work Plans, Quality Assurance Project Plans, Analysis of Brownfield Cleanup Alternatives and Final Engineers Reports. Through this experience, Dan has a firm understanding

of the EPA requirements for planning and implementing investigation and cleanup projects funded by the EPA.

Republic Steel: NPDES & 40 CFR 112.7 Compliance - Lorain & Canton, Ohio

Mr. Noll led a project to assist an industrial client with updating compliance plans for two steel manufacturing facilities in Ohio (Lorain & Canton). The Lorain facility was dormant; however, the facility still had an active NPDES Permit and had a release of oil to a surface water (prior to LaBella being retained). Due to the surface water release the facility was under a Consent Order with USEPA. The USEPA Consent Order (with Ohio EPA involvement) required updating of the SWPP Plan for both the vacant Lorain facility and the active manufacturing facility in Canton Ohio. In addition to numerous outfalls at each facility which necessitated the NPDES Permits, both facilities also had large quantities of oil storage and thus required SPCC Plans. LaBella was retained to update both facilities SWPP Plans and SPCC Plans for review by USEPA and Ohio EPA. LaBella completed a review of existing plans, completed site visits and updated the plans for review by regulatory agencies.

Ebenezer Plaza II – BCP Site Remediation

Mr. Noll was the engineer of record for the design and construction of remedial systems at a Brownfield Cleanup Program Site in Brooklyn NY. The remediation work consisted of a source area soil removal, in-situ chemical injections and a sub-slab depressurization system (SSDS). The soil removal was completed in-conjunction with the site development work in order to minimize excavation and dewatering costs. Subsequent

**Former Taylor's Lane
Composting – Landfill
Monitoring**

LaBella assist the Village of Mamaroneck with annual monitoring of a formal landfill. Mr. Noll is the engineer of record for recent modifications to the Site Management Plan. The Site Management Plan identifies the required institutional and engineering controls for the Site and also the routine monitoring of the Site. The engineering controls at this Site include a low permeability cap over the former landfill, security fencing, and a stormwater/leachate management system. The monitoring includes annual inspections of the engineering controls and annual groundwater monitoring.



KATHERINE TRUONG

Environmental Geologist

Katherine is an Environmental Geologist and is responsible conducting Phase II Environmental Site Assessments (ESAs) of active and abandoned commercial and industrial properties in accordance with ASTM standards and is responsible for the planning and execution of field data collection programs; regulatory and historical records review; data management and evaluation; and technical report preparation within the context of environmental due diligence and environmental monitoring projects.

In Katherine's previous role as a Phase I Analyst, she was responsible for the preparation of Phase I Environmental Site Assessments. The site assessments included evaluation of environmental liability associated with properties, and Katherine provided efficient analysis and completion of environmental reports for financial institutions, attorneys and private developers.

Town of Palmyra, Old Palmyra Landfill Site Characterization (20191764.01): Palmyra, NY

Katherine served as the primary Environmental Geologist for the environmental investigation at the Old Palmyra Landfill Site. The purpose of the Site Characterization work plan had the following goals: to determine the presence of hazardous waste and whether that wastes poses a significant threat to human health or to the environment, to determine if contamination is present at the site and if it is migrating off-Site, to develop a list of contaminants of concern at the Site, and to adequately determine the depth and direction of groundwater flow. Her primary responsibilities included implementing the Site Characterization work plan which had the following tasks: Test pit installation/soil sampling, surface water/sediment sampling, surface soil sampling, and groundwater monitoring well installation. She had performed the following soil, sediment, surface water, and groundwater sampling events.

68 Marsh Road LLC, 68 Marsh Road Site Remediation (2201381), Rochester, NY

Katherine served at the Environmental Geologist for remedial activities conducted at 68 Marsh Road to address the petroleum impacts to the soil and groundwater at the Site. Her responsibilities were to oversee the remedial work plan which included the excavation of the impact soils, managing the impacted water, and performing confirmatory soil sampling during the excavation.

Phase I and Limited Subsurface Evaluations, Repsol

Katherine was responsible for conducting a set of nine Limited Environmental Due Diligence reports and Limited Subsurface evaluation reports for Repsol's Well Reclamation Project in 2019. Duties for this project included creating a report template that best suited the client's needs and provide satisfactory environmental assessment, conducting site visits, recording field data, performing confirmatory sampling, and

INTERN GEOLOGIST New York State

EDUCATION SUNY Geneseo: BA, Geology

University of Connecticut: MS, Geological Science

CERTIFICATIONS/ REGISTRATIONS

**OSHA 40-Hour Certified
Hazardous Waste Site Worker
Training**

**OSHA 8-Hour Certified
Hazardous Waste Site Worker
Refresher Training**



waste characterization sampling. In 2021, she assisted in Repsol's Well Reclamation Project with the setting up eight Limited Environmental Due Diligence reports and writing the reports.

Former Emerson Street Landfill Remedy Implementation, City of Rochester

Katherine is responsible for executing and overseeing the field activities associated with the agreed upon Remedial Work Plan developed by LaBella Associates, GEI Consultants, City of Rochester, and the New York State Department of Environmental Conservation (NYSDEC). Her other duties included, recording field notes and collecting field data, overseeing overburden/bedrock drilling and injection work, groundwater sampling, and waste characterization sampling.

68 Marsh Road Site Remediation, Hoselton Auto Mall

Katherine was responsible for overseeing the removal of petroleum underground storage tanks and the remediation of petroleum impacted water and soils. Her other responsibilities included the following, recording field notes, overseeing construction consultants, confirmatory sampling, and waste characterization sampling.

Brownfields

Norry Management Corp (213131), BCP Application 3750 Monroe Ave: 3750 Monroe Ave Rochester NY

Katherine served as an Environmental Geologist whose responsibilities included groundwater sampling and implementing a pilot test work plan for interim remedial measure. The goal of the pilot test was to evaluate the effectiveness of chemical injections as a

potential source area treatment method. Her role in implementing the pilot test work plan included: oversight of drilling and the chemical injection into the source area.

Jefferson Wollensack LLC (2182207) 872 and 886 Hudson Brownfield: 872 Hudson Ave Rochester, NY

Katherine served as an Environmental Geologist at the 872 and 886 Hudson Ave Brownfield Site. Responsibilities include site wide groundwater sampling.

Getinge Sourcing LLC (2160339), 1777 East Henrietta Road BCP App: 1777 East Henrietta Road Henrietta NY

Katherine served as an Environmental Geologist and was involved in the remedial measures work plan for two areas of concern. Her responsibilities included: collecting groundwater samples, oversight of drilling injections wells, and the in-situ chemical treatment for the areas of concern.

Phase I ESAs

Katherine has conducted numerous Environmental Site Assessments. Site assessments include evaluation of environmental liability associated with properties such as warehouses, gas stations, colleges, commercial properties, agricultural properties and residential homes. Katherine provides efficient analysis and has completed environmental assessments for the following groups, among others:

Financial Institutions

- ESL Federal Credit Union
- Canandaigua National Bank
- Key Bank
- Community Bank, N.A.

- Five Star Bank
- Northwest Bank
- M&T Bank
- Bank of the Finger Lakes
- Pathfinder Bank
- First Heritage Federal Credit Union
- First Citizens Bank
- Reliant Community Federal Credit Union
- S&T Bank
- Steuben Trust Company

Development and Construction Companies

- SSM Properties Holdings, LLC
- Wowe, LLC
- Gold Wynn Residential, LLC
- Kings Harbor View Associates
- Capstone Construction Services, LLC
- Canandaigua Lakefront, LLC
- Shea Homes, LLC
- Royal Oak Realty
- MCA Group, LLC
- Birnbaum Companies
- Ryan Homes
- Grove Street Management
- Village Solars LLC
- The DDS Companies
- CDS Life Transitions
- Amanda Grover Real Estate
- Monsees Group
- Singh Brothers Properties
- Donohoe Management
- Home Leasing LLC

KATHERINE TRUONG

- Pathstone Corporation
- GHRS Foundation Inc
- South Wedge Properties
- Momentum Holdings Group
- Zap Land Holdings

Local Gov'ts

- County of Orleans, IDA
- Geneva Public Library
- City of Rochester
- Town of Palmyra

Not for Profits

- The Community Preservation Corporation
- The Nature Conservancy
- Tabernacle Joyful Praise

Attorneys

- Stephen Hall, Esq.

Other Clients

- Tompkins Bank of Castile
- Mr. Dadvid Pantalone (private individual)
- MRB Group
- Canandaigua Bank
- L3 Harris Technologies
- 68 Marsh Road LLC
- Mandelbaum and Mandelbaum
- Cycle Enterprises
- Sail Energy
- 4022 Tech Park Blvd
- CEA Fresh Farms
- PathFinder Bank ISAOA
- Pennant Ingredients
- NPV, Inc
- Steuben Trust Company
- Stephen Hall Esq
- Harter Secrest
- Woods Oviatt
- Bond, Schoeneck and King PLLC
- J.O Cook Inc

- Conifer Realty
- Sodus Properties

Phase II ESAs

Development and Construction Companies:

- Leonard's Express
- Sodus Properties
- Conifer Realty

Local Gov'ts

- NYS DOT
- NYS DEC

Other Clients

- Repsol Oil and Gas

68-92 Genesee Street Site Investigation, City of Rochester

Katherine was responsible for executing and overseeing the field activities associated with the agreed upon Remedial Work Plan developed by LaBella Associates. Her responsibilities consisted of overseeing the drilling and installation of 11 bedrock wells, overburden soil borings, site-wide soil and groundwater sampling, and groundwater sampling, GPS data collection, GIS Mapping, air sampling, soil vapor intrusion assessment and waste characterization sampling. She also assisted in writing the Site Investigation Report and Construction Completion Report which was submitted to NYSDEC.

Annual Groundwater Sampling, City of Rochester: 1200 East Main Street, Rochester NY

Katherine served as the Environmental Consultant/Geologist on this job. She was responsible for submitting a proposal for the groundwater sampling event, ordering equipment, assisting the City of Rochester with the groundwater sampling, and submitted the samples. Other duties included

data tabulation, getting the data validated, and submitting a data package to the City of Rochester.

Soil Vapor Intrusion Assessment, Fieldstone Private Wealth: 219 East Main Street, Batavia NY

Katherine served as the Environmental Geologist and performed a soil vapor intrusion assessment of a one-story building. The sampling included: indoor air, sub-slab vapor, and outdoor air samples. Other duties included completing the indoor air quality questionnaire and taking inventory of products. She also submitted the samples, analyzed the results, and submitted the report to the client.

Colfax Street BUD, City of Rochester: 1700 Emerson Street, Rochester NY

Katherine served as the Environmental Geologist and oversaw the transportation of the Colfax Street Bud Material to 1700 Emerson Street Landfill. Her duties included: overseeing the trucking contractor, field notes, and ensuring the project was done in a timely matter.

Site Management Plan Written Report, Clinton North Development Corp 113-117 North Clinton Ave Rochester NY

Katherine served as the primary author for the Site Management Plan at 113-117 North Clinton Avenue. She assisted in creating the figures for the report and putting together the appendix.

Project Environmental Management Plan Written Report, CDS Life Transitions: Various Properties on Clifford and Joseph Avenue

Katherine served as the primary author for the Environmental Management Plan at Various Properties on Clifford and Joseph Avenue. She assisted in creating the figures for the report and putting together the appendix.



ALEXANDER DASILVA

Environmental Analyst

INTERN GEOLOGIST New York State

EDUCATION
University at Buffalo: MA,
Geology with a concentration in
Hydrogeochemistry

SUNY Geneseo: BA,
GeoChemistry

CERTIFICATIONS/ REGISTRATIONS OSHA 40-Hour HAZWOPER

OSHA 40-Hour Certified
Hazardous Waste Site Worker
Training

OSHA 8-Hour Certified
Hazardous Waste Site Worker
Refresher Training

Alex's experience includes completing Phase II Environmental Site Assessments (ESAs) as well as performing investigations and remediation under New York State Programs. He is proficient in ArcGIS, has worked on NYSDEC DER-10 required Site Management Plans. Alex also has project experience in Graduate School focusing on abiotic TCE degradation from natural minerals such as pyrite, and the degradation of ortho-phosphate from sample transportation, as well as in laboratory analytical chemistry. Alex has worked for NYSERDA and the DOE finding sedimentation and erosion rates in glacial materials surrounding West Valley Nuclear Waste Facility, he can decipher different glacial materials. Alex has collected OSL (Optically Stimulated Luminescence) dating and C14 dating methods. Alex took an 8-hour NYSERDA and DOE safety training on how to proceed when encountering nuclear waste materials.

LiDestri Eco-Industrial Park: Former Portion of Kodak Park South – Rochester, NY

Alex conducted a 3-month Site Management Plan (SMP) Implementation including environmental oversight, environmental monitoring, and implementation of an Excavation Work Plan (EWP). He was responsible for tracking all soils relocated on-site by a contractor, including the excavation of over 33,000 cubic yards of soil and cinders. Alex was responsible for collecting soil samples, all data management, surveying, and presentation of data.

Lyons National Bank: Phase II Environmental Site Assessment – Macedon, NY

Alex conducted a Phase II ESA to evaluate subsurface soils and groundwater for a future potential building. Alex was responsible for soil boring logging, installation of groundwater monitoring wells, soil and groundwater sampling, and preparing a Phase II ESA report.

Former Emerson Power Transmission Facility: Interim Remedial Measures Assistance and Groundwater Monitoring Well Decommissioning via NYSDEC CP-43 Policy – Ithaca, NY

Alex was responsible for decommissioning several groundwater monitoring wells on-site in accordance to NYSDEC CP-43 Groundwater Monitoring Well Decommissioning Policy. Alex assisted in monitoring the remedial work on the 100-acre property developed with a 800,000 sq.ft. facility where he oversaw the removal of contaminated soils, and ensured the integrity of the work being performed to protect the interests and limit the liability of the purchaser.

Silver Lakes Brewing Project: Soil Vapor Intrusion Assessment – Perry NY

Alex performed a soil vapor intrusion assessment at a newly opened brewery to ensure air quality in the tasting room was



within the limits of the New York State Department of Health (NYSDOH) guidance values. Alex was responsible for filling out a Department of Health (DOH) questionnaire, locating and documenting any potential chemicals that could skew readings, and setting up stainless steel electropolished (SUMMA) canisters.

WBF Properties XV LLC: Soil and Groundwater Management Plan NYSDEC Spill # 1404095 – Seneca Falls, NY

Alex implemented the Soil and Groundwater Management (SGMP) plan corresponding to a NYSDEC Spill number. Alex was responsible for the screening, sampling and the separation of any petroleum contaminated soils on site. Alex was also responsible for creating a map to show where the areas of contamination were using ArcGIS. Alex discovered additional areas of contamination on site and responded to them according to the SGMP. Alex worked with the DEC on site in order to ensure all areas of concern were accounted for.

Lyons National Bank: Phase II Environmental Site Assessment – Lyons, NY

Alex conducted a Phase II ESA to evaluate subsurface soils and groundwater to limit the liability of the purchaser. Alex was responsible for soil boring logging, installation of groundwater monitoring wells, soil and groundwater sampling, and preparing a Phase II ESA report.

City of Rochester: Former Photech Imaging Site, Periodic Monitoring – Rochester NY

Alex is periodically collecting low flow groundwater samples for the City of Rochester in accordance with the SMP at

the Former Photech Imaging site. Alex is responsible for comparing groundwater sample concentrations of analytes dictated by the SMP to previous data to identify any pattern of possible transport or remediation. Alex is also responsible for preparing the periodic monitoring letter for the client.

Canandaigua National Bank and Trust: Phase II Environmental Site Assessment – Rochester, NY

Alex conducted a Phase II ESA to evaluate subsurface soils and groundwater to limit the liability of the purchaser. Alex was responsible for soil boring logging, installation of groundwater monitoring wells, soil and groundwater sampling, and preparing a Phase II ESA report. During this Phase II ESA Alex identified and delineated petroleum based contamination.

Highland Grove, LLC: Site Management Plan Implementation on a BCP Site – Rochester, NY

Alex conducted a portion of the environmental oversight and monitoring during the construction of a new building on a BCP Site. While on site, Alex was responsible for tracking all soils relocated by the excavation contractor as well as any back fill imported onto the site. In addition to environmental monitoring, Alex collected groundwater and soil samples as per DEC request. Alex also decommissioned wells in accordance to NYSDEC CP-43 Groundwater Decommissioning Policy.

Conifer Reality LLC: Soil and Groundwater Management Plan Implementation – Rochester, NY

Alex is currently implementing the site SGMP. This site had previous known petroleum contamination on it and Alex is

responsible for the environmental oversight and monitoring over the 2-month period of soil excavation. Alex segregates soils into individual stockpiles based on the initial Photoionization Detector (PID) readings. Alex participates in meetings with the excavation contractors and the client regarding soil excavation. Alex also prepares monthly reports including analytical data, monitoring data, and a written letter describing what had happened throughout the month.



DAVID ENGERT, CHMM

VP, Environmental Construction Department Lead

Dave has over 24 years of experience as a Geologist and Project Manager in the environmental consulting and contracting industries. He is the lead for LaBella's Environmental Construction Department, which provides remediation services, environmental and geotechnical drilling, remediation system design, installation, and O&M, and petroleum bulk storage compliance, maintenance, closure, and installation services. He has conducted and managed numerous Phase I and Phase II Environmental Site Assessments, soil and groundwater remediation projects, direct push and rotary drilling projects, and groundwater monitoring programs for both public and private sector clients.

EDUCATION

State University of New York at Buffalo: BA, Geology

CERTIFICATIONS/REGISTRATIONS

Certified Hazardous Materials Manager (CHMM)

ASSP Certificate in Safety Management

City of Rochester Bulk Storage Tank Removal Certificate of Competency

OSHA Hazardous Waste Operations & Emergency Response Supervisor Course

OSHA Hazardous Waste Operations & Emergency Response 40-Hour Site Worker Course & Annual Refreshers

OSHA 10-Hour Construction Safety Course

OSHA Excavation Safety Competent Person

FEMA ICS 100 — Introduction to the Incident Command System

FEMA ICS 200—ICS for Single Resources and Initial Action Incidents

PROFESSIONAL AFFILIATIONS

Alliance of Hazardous Materials Professionals (President, Finger Lakes Chapter)

American Society of Safety Professionals

Remedial Construction

USEPA CERCLA Removal Action – Lake Erie Smelting Corp. Site, Buffalo, NY

Remedial Construction Manager for the removal of lead contaminated soil at a former secondary lead smelting site currently occupied by a low income housing complex encompassing 36 buildings in 18-acres of land. The CERCLA removal action was conducted pursuant to an Administrative Settlement Agreement and Order on Consent stipulated by the USEPA and involved the removal of 3,800 tons of lead, cobalt and copper impacted soil from 11 discrete removal areas covering nearly 2-acres. Work was conducted under the direct oversight of USEPA Region 2 Removal Branch. Responsible for managing remedial construction resources (staff and equipment) and subcontracted waste haulers deployed for the project, as well as securing waste stream approval at an USEPA-approved disposal facility and coordinating the transport and disposal of

contaminated soil. Also secured NYSDEC DER-10 compliant backfill and oversaw site restoration activities.

NYSDEC Standby Investigation & Remediation Contract

Dave serves as Contract Manager and Project Manager on a Standby Investigation and Remediation (I&R) Services contract for Region 8 of the NYS Department of Environmental Conservation. Duties include overall contract management, coordination with project managers and field staff and communication with NYSDEC technical and contract staff. Project assignments associated with the contract include remediation services, subsurface investigations, installation, operation and maintenance of remedial treatment systems and routine sampling.

Former Geneva Foundry Off-Site - Geneva, NY

Dave serves as Project Manager for investigation and remedial actions at properties impacted by lead and arsenic resulting from

aerial fallout from an adjacent foundry. The project is funded through the NY State Superfund Program and is being performed under LaBella's Standby I&R contract with NYSDEC. The project involves the investigation and cleanup of up to 220 primarily residential properties surrounding the former foundry. Remediation has been completed on a total of 150 properties to date with 73,750 tons of contaminated soil being excavated and transported for off-site disposal. The project is anticipated to continue for an additional 2 to 3 years.

UST Removal & Petroleum Site Remediation – Belmont, NY

Dave served as Project Manager for the removal of underground storage tanks (USTs) and remediation of petroleum impacted soil at a vacant truck stop and gas station. The work was performed for the Allegany County Industrial Development Agency (IDA) as part of the IDA's effort to remediate blighted properties and make them more attractive to developers. The project consisted of the removal and decommissioning of ten USTs ranging in size from 1,000 to 27,000 gallons and the excavation, transportation and off-site disposal of 1,200 tons of petroleum impacted soil. In addition, 23,000 gallons of petroleum impacted groundwater pumped from excavations was treated on-site through carbon and transported to the local POTW for disposal.

Yates County: Penn Yan Marine Brownfield Cleanup Program - Penn Yan, NY

Dave served as Project Manager for the remedial actions at a former boat manufacturing facility on the Keuka Lake Outlet. The remedial actions included excavation and off-site disposal

of more than 8,500 tons of non-hazardous soil impacted with semi-volatile organic compounds and heavy metals and approximately 125 tons of TSCA hazardous soil impacted with PCBs. Additional activities included removal and disposal of significant quantities of construction & demolition debris, installation of storm water and erosion control measures, and excavation dewatering.

Generator Tank Replacement at Somerset Generating Station - Somerset, NY

Dave served as Project Manager for contractor services on this design-build project. The project was performed inside an active electrical substation and involved the removal of a 1,000 gallon diesel underground storage tank associated with the control house backup generator and replacement with a 500 gallon aboveground tank and day tank. Dave's responsibilities included procurement of materials and overall coordination of tank removal and installation activities.

Brownfield Cleanup Programs**Urban League of Rochester Economic Development Corporation: Former Michelsen Furniture Co. Site Brownfield Cleanup Program - Rochester, NY**

Dave served as the Project Manager for a BCP project at a site with historical operations including furniture manufacturing and a machine shop. Dave managed investigation activities at the site prior to its inclusion

in the BCP. Dave worked with the project team's legal counsel and developer to prepare project budgets and drafted the BCP Application. The site was accepted into the BCP and a Remedial Investigation was performed. Remedial actions at the site included in-situ chemical oxidation using sodium permanganate as an oxidant, excavation and off-site disposal of solvent impacted soil and installation of a sub slab depressurization system. Subsequent to completion of a Site Management Plan and Final Engineering Report the NYSDEC determined that the cleanup requirements were achieved and issued a Certificate of Completion for the Site.

Former Breneman Site Brownfield Cleanup Program - Oswego, NY

Dave is serving as Project Manager for Remedial Investigation and Remedial Actions at a 2.2 acre former manufacturing facility enrolled in the NYSDEC BCP. A Remedial Investigation was performed and a Remedial Alternatives Analysis and Remedial Action Work Plan were prepared for the Site. Remedial Actions included in-situ chemical oxidation (ISCO) consisting of injection of a persulfate based oxidant, installation of injection wells to facilitate future injections and placement of a cover system (to be performed by Owner). A Site Management Plan and Final Engineering report were prepared and submitted to the NYSDEC and the site received a Certificate of Completion.

Former Labelon Corp. - Canandaigua, NY*

Project Manager for Brownfield

**Projects completed under previous employment.*

Remedial Investigation at vacant building that was historically operated by a bicycle factory and manufacturer of heat sensitive labels. Performed Phase I and Phase II Environmental Site Assessments prior to site being accepted into NYS Brownfield Cleanup Program. Contaminants of concern at the site included trichloroethene and associated daughter products, heavy metals and petroleum. Developed Remedial Investigation Work Plan and secured approval from NYSDEC. Provided oversight of Remedial Investigation performed by USEPA contractors under a Brownfield Assessment Grant.

NYSDEC Petroleum Spill Investigation and Remediation

Petroleum & Solvent Remediation at Former Industrial Laundromat and Taxi Cab Company - Rochester, NY

Dave served as Project Manager for the remediation of petroleum and solvent contaminated soil at a site with historical operations that included an industrial Laundromat and automotive repair associated with a taxi cab company. The majority of the soil impacts consisted of gasoline contamination, however, a portion of the impacted soil consisted of commingled petroleum and solvent impacts. Subsequent to additional characterization sampling a Contained-In Demonstration Work Plan was submitted to and approved by the NYSDEC. After implementation of the work plan a Contained-In request was approved by NYSDEC and the solvent containing soil was approved for disposal as non-hazardous soil at a Subtitle D landfill, realizing significant savings for the client. Approximately 275 tons of gasoline impacted soil and 78 tons of solvent and petroleum

impacted soil was excavated, transported and disposed.

6 Oil UST Removal at Former Manufacturing Facility - Rochester, NY

Dave served as Project Manager for the removal of two out of service 30,000 gallon #6 heating oil underground storage tanks at a former railroad signal manufacturing facility. The tanks contained over 25,000 gallons of #6 heating oil and 15,000 gallons of water. The heating elements in the tanks were corroded and not functional. After removal of the water hydraulic hose was placed in the tanks and connected to a 100,000 BTU ground heater to circulate heated glycol to reduce the viscosity of the oil and facilitate pumping. The oil was then pumped and transported to an energy recovery facility. The tanks were then removed from the ground, cleaned and decommissioned as scrap steel.

Petroleum Spill Site Remediation at Former Gasoline Station - Henrietta, NY

Dave served as Project Manager for the remediation of a former gasoline station. Remediation activities include the removal of four underground storage tanks, excavation and off-site disposal of over 3,200 tons of petroleum contaminated soil, and amendment of backfill with oxygen releasing compounds to promote natural degradation of the downgradient groundwater plume. A sub slab depressurization system was installed to mitigate vapor intrusion into an adjacent building. A groundwater monitoring program is ongoing.

Petroleum Spill Site Investigation & Remediation at Apartment Complex - Brighton, NY

Dave served as Project Manager for the investigation and remediation of apartment

complex that is the site of a former gasoline and fuel oil bulk storage terminal. The investigation consisted of a geophysical survey, a direct-push soil boring program, installation and sampling of groundwater monitoring wells and a vapor intrusion assessment of select apartment buildings. Remediation activities include excavation and off-site disposal of petroleum contaminated soil. A Soil and Groundwater Management Plan was developed to address residual contaminants. Secured closure of the site from NYSDEC.

Petroleum Spill Site Investigation and Remediation at Silver Lake Marine - Castile, NY

Dave served as Project Manager for the investigation and remediation of private marina and boat showroom on Silver Lake. Designed and implemented a Phase II Environmental Site Assessment to assess the findings of a lender-required Phase I. Remediation activities included excavation and off-site disposal of petroleum impacted soils adjacent to boat launch and break wall. Secured closure of site from NYSDEC.

Former HEP Sales - Horseheads, NY*

Project Manager for remediation of former hardware store and automobile dealership. Responsibilities included coordination of all contractors working independently. Remedial activities included excavation and off-site disposal of approximately 2,300 tons of petroleum and non-hazardous solvent impacted soil, installation and sampling of groundwater monitoring wells, injection of oxygen releasing compounds to treat residual groundwater impacts and development of a Soil and Groundwater Management Plan.

Secured closure of site from NSYDEC.

Gasoline Station - Watertown, NY*

Project Manager for investigation and remediation at gas station prior to property transfer. Conducted a Phase II Environmental Site Assessment to identify subsurface conditions and develop a Remediation Action Plan for NYSDEC approval. Responsibilities included coordinating removal of underground storage tanks, excavation, transportation and disposal of over 1,100 tons of petroleum impacted soil, contaminated groundwater management and development of a Soil and Groundwater Management Plan. Secured closure of site from NYSDEC.

Elmer's Brighton Garage - Brighton, NY*

Project Manager and Geologist for investigation and remediation at an automobile repair facility. Identified recognized environmental conditions (RECs) during a Phase I Environmental Site Assessment. Performed a Phase II Environmental Investigation to address RECs and acquire data necessary for design of remedial strategy. Site remediation included the excavation, transportation and disposal of approximately 300 tons of petroleum impacted soil, removal of two underground hydraulic lifts, groundwater extraction utilizing a vac truck and installation of six bedrock groundwater monitoring wells. Conducted quarterly groundwater sampling to monitor contaminant degradation until obtaining regulatory closure.

Gasoline Tanker Rollover - Dresden, NY*

Project Manager for cleanup of approximately 5,000 gallon release of gasoline resulting

from a motor vehicle accident. Assigned responsibility for site management after completion initial response activities. Responsibilities included the installation of a high-vacuum extraction system, oil water separator, diffused air stripper and carbon treatment unit for the remediation of groundwater at the site contaminated with dissolved and free-phase gasoline and monthly operations and maintenance activities, quarterly sampling and reporting to regulatory authorities. Secured closure of site from NYSDEC.

Former Service Station - Rochester, NY*

Project Manager for remediation of former service station. Responsibilities included design, installation and operations & maintenance of a high-vacuum extraction system inside the site building. Oversaw O&M and periodic monitoring of system performance and conducted final investigation to determine effectiveness of system on treatment of soil and groundwater contamination. Secured closure of site from NYSDEC.

Artco Industrial Laundries - Rochester, NY*

Project Manager for monitoring and remediation of former dry cleaning site under a Voluntary Cleanup Agreement with the NYSDEC to address soil and groundwater contamination resulting from a release of tetrachloroethene. Responsibilities included oversight of system installation, operation and maintenance, groundwater sampling, report writing and coordination with client, attorneys and NYSDEC officials.

Phase I & Phase II Environmental Site Assessments*

Project Manager and Geologist for numerous Phase I and Phase II Environmental Assessments for private individuals, corporations, law firms and lending institutions. Properties have included bulk storage facilities, gasoline stations, automobile dealerships, light industrial and commercial facilities, cellular tower sites and agricultural properties. Phase II activities have included design and supervision of soil sampling and direct-push boring programs, installation of monitoring wells and groundwater sampling, conducting soil-gas investigations, and interpretation and reporting of acquired data.



Project Team Summary

LDC personnel have hands-on experience in the areas of data validation, laboratory QA/QC, CLP SOWs, and environmental laboratory analyses. As documented in the resumes of our staff, the project team has significant experience with USACE and DoD protocols, current technology, SW-846, and all methods stated in the SOW.

LDC is presenting the following staff to perform key roles for this contract. The key staff of the project team and their experience are as follows:

- **Stella Cuenco, Principal Chemist/Operations Manager**
Project Role: Principal Chemist/Program Manager
Data Validation Experience: 26 years
Overall Laboratory and Data Validation Experience: 32 years
B.S. Chemistry, University of the Philippines, 1991

Ms. Cuenco has over 32 years of environmental laboratory and data validation experience under DoD and EPA guidelines. Her experience includes performance of data validation in gas chromatography/mass spectrometry for volatile and semivolatile organics and extensive Navy and EPA data review and data verification for all organic and inorganic analyses. Her laboratory experience includes hands-on CLP and SW-846 GC/MS methods.

- **Pei Geng, Senior Chemist/Project Manager**
Project Role: Senior Organic Data Validator/Project Manager
Data Validation Experience: 25 years
Overall Laboratory and Data Validation Experience: 32 years
M.S. Chemistry, Sam Houston University, 1989

Ms. Geng will perform the role of day to day Project Manager for this project. She will monitor schedules, compliance of validation to the Required Guidelines, perform routine surveillance activities such as generation of non-conformance reports, validator training and QA reports to management.

Ms. Geng will perform the role of organic data validator for this project. She will perform data validation for GC/MS and gas chromatography analyses and serve as a peer reviewer in the initial validation review process.

Ms. Geng has over 31 years of environmental laboratory and data validation experience. Her experience includes performance of data validation in the gas chromatography area for volatile and semivolatile organics and extensive DoD data review and data verification for all organic analyses. Her laboratory experience includes hands-on CLP and SW-846 GC/MS methods.

- **Michael Giangjordano, Chemist/Project Manager**
Project Role: Project Management Assistance
Data Validation Experience: 8 years
Overall Laboratory and Data Validation Experience: 22 years
B.S. Kinesiology, pending, San Diego State University, San Diego, CA



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Mr. Giangiordano has 8 years of experience at LDC and specializes in supporting the company's custom software products. Mr. Giangiordano has a thorough knowledge and understanding of the company's branded software and has led numerous workshops and training sessions for clients ranging from laboratory personnel to consulting firms to USACE. He has extensive experience in electronic data deliverables and electronic data deliverable review and provides database support and management solutions for clients using LDC's custom environmental database management system (EDMSi).

Mr. Giangiordano has 14 years of environmental laboratory. His experience includes Project Manager at EnviroMatrix Analytical, Inc., an accredited full service environmental analytical chemistry facility, Mr. Giangiordano oversaw projects that provided analytical services and support to clients ranging from environmental consulting firms to marine biology firms, in addition to waste and wastewater treatment and disposal firms and municipalities. Mr. Giangiordano was also the Supervisor of the WET Chemistry and Microbiology Departments at EnviroMatrix Analytical, Inc. where he was responsible for all department functions which included overseeing daily operations, training staff, final reporting of analytical data, compliance with method requirements, as well as introducing and developing new methods for additional accreditation.

An Le, Inorganic Chemist

Project Role: Inorganic Data Validator

Data Validation Experience: 5 years

Overall Laboratory and Data Validation Experience: 23 years

B.S. Biological Science, 2000, University of California, Irvine

Ms. Le has over 23 years combined environmental laboratory and data validation experience. Her experience includes performance of data validation using USEPA National Functional Guidelines, client Quality Assurance Program documents, and the Department of Defense QSM depending on the project requirements for the clients.

Ms. Le was a Wet Chemistry Analyst at TestAmerica Laboratories, Ms. Le performed analysis of an extensive list of wet chemistry analyses. Ms. Le also performed volatile organic compounds analysis according and was also responsible for training new analyst employees and performing second level review of data.

Judy Ecklund, EDD Specialist

Project Role: Electronic Data Entry (EDD)

EDD Experience: 14 years

Ms. Ecklund specializes in Electronic Data Deliverables and is familiar with a variety of deliverable formats, including but not limited to NEDD, EQUIS, and SEDD. Ms. Ecklund is also an expert in submitting data to NIRIS the Navy database.

Ms. Ecklund has over 31 years combined environmental laboratory and validation related experience. Her experience includes working with electronic data deliverables (EDDs) as well as performing database uploads.

- **Tony Rommelfanger, Data Control Manager**
Project Role: Data Custodian



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Mr. Rommelfanger will perform the role of data custodian for this project. He will perform the log-in of all data packages into the LDC tracking system. This system will generate spreadsheets for identifying all samples, their collection date, analysis performed, matrix, and report due date. Upon the completion of each delivery order, he will archive and catalog all reports and data in a secured storage area.

Mr. Rommelfanger has over 31 years of experience in laboratory and data management experience. He has experience in organizing, logging in, and tracking data packages for technical staff.



Resumes of Key Staff

- Stella Cuenco, Senior Chemist
- Pei Geng, Senior Chemist
- Michael Giangiordano, Chemist
- An Le, Inorganic Chemist



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RESUME STELLA S. CUENCO

EDUCATION

B.S. Chemistry, 1991
University of the Philippines (UP)

PROFESSIONAL HISTORY

Laboratory Data Consultants, Inc.
Senior Chemist
1996 to present

Ceimic Corporation
GC/MS Chemist
1996

Analytical Technologies, Inc.
GC/MS VOA Group Leader
1992 to 1996

Analytical Technologies, Inc.
GC/MS Chemist
1991 to 1992

Natural Products Research, UP
Research Assistant
1990 to 1991

REPRESENTATIVE EXPERIENCE

Ms. Cuenco has over 32 years combined environmental laboratory and data validation experience. Her experience includes performance of data validation in the GC and GC/MS areas for major Federal projects. She has performed large validation projects under Boeing, Navy Southwest, Northwest and Pacific Division, EPA Region IX ESAT, USACE and AFCEE/AFCEC programs. Her laboratory experience includes hands-on CLP and EPA analysis of GC and GC/MS volatile organic compounds.

Specifically, Ms. Cuenco has over 26 years organic data validation experience using USEPA (including Region III) functional guidelines and other applicable documents.

- As senior chemist with LDC, Ms. Cuenco specializes in the data validation and contract compliance screening of gas chromatography-mass spectrometry analyses as well as gas chromatography analyses. She has a thorough knowledge and understanding of gas chromatography and gas chromatography-mass spectrometry (GCMS) and high resolution GCMS methods referenced in EPA CLP, SW-846, EPA 500, 600 and 1600 series documents. She has performed large data validation under Boeing, Navy Southwest and Pacific Divisions and EPA Region IX ESAT, USACE and AFCEE/AFCEC projects.



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Ms. Cuenco has over 6 years experience in an environmental laboratory performing the analysis of organic parameters.

- As GC/MS chemist at Ceimic Corporation, a full service environmental analytical chemistry facility, Ms. Cuenco performed GC and GC/MS volatile analyses. She was responsible for the final reporting of analytical data for this section.
- As GC/MS VOA Group Leader at Analytical Technologies Inc., a full service environmental analytical chemistry facility, Ms. Cuenco was responsible for all GC/MS functions which included overseeing daily operations, training staff, final reporting of analytical data, and compliance with method requirements.
- As research assistant at Natural Products Research, UP, Ms. Cuenco researched chemical literature for plants with known medicinal properties as well as performed microbiological and pharmacological tests on plant extracts.



RESUME
PEI GENG

EDUCATION

M.S. Organic Chemistry, 1989
Sam Houston State University

B.S. Environmental Chemistry, 1983
Nankai University

PROFESSIONAL HISTORY

Laboratory Data Consultants, Inc.
Senior Chemist
1997 to present

Ceimic Corporation
GC/MS and GC Chemist
1996 to 1997

PACE Analytical Service Inc.
GC/MS and GC Chemist
1990 to 1996

REPRESENTATIVE EXPERIENCE

Ms. Geng has over 32 years combined environmental laboratory and data validation experience. Her experience includes performance of data validation in the GC and GC/MS areas for major Federal projects. She has performed large validation projects under Boeing, Navy Southwest, Northwest and Pacific Division, EPA Region IX ESAT, USACE and AFCEE/AFCEC programs. Her laboratory experience includes hands-on CLP and EPA analysis of GC and GC/MS volatile organic compounds.

Specifically, Ms. Geng has over 25 years organic data validation experience using USEPA CLP (including Region III) functional guidelines and other applicable documents.

- As chemist with LDC, Ms. Geng specializes in the data validation and contract compliance screening of gas chromatography-mass spectrometry analyses as well as gas chromatography analyses. She has a thorough knowledge and understanding of gas chromatography and gas chromatography-mass spectrometry (GCMS) and high resolution GCMS methods referenced in EPA CLP, SW-846, EPA 500, 600 and 1600 series documents. She has performed large data validation under Boeing, Navy Southwest and Pacific Divisions and EPA Region IX ESAT, USACE and AFCEE/AFCEC projects.



Ms. Geng has over 7 years of experience in an environmental laboratory performing the analysis of organic parameters.

- As both a GC and GC/MS chemist at Ceimic Corporation, a full service environmental analytical chemistry facility, Ms. Geng performed GC and GC/MS volatile and semivolatile analyses.
- As both a GC and GC/MS chemist at PACE Analytical Service Inc., a full service environmental analytical chemistry facility, Ms. Geng performed GC and GC/MS volatile and semivolatile analyses as well as overseeing the final reporting of analytical data, and compliance with method requirements.



RESUME
MICHAEL D. GIANGIORDANO

EDUCATION

B.S. Kinesiology, pending
San Diego State University, San Diego, CA

PROFESSIONAL HISTORY

Laboratory Data Consultants, Inc.
Sr. Environmental Informatics & Software Support Specialist
2016 to present

EnviroMatrix Analytical, Inc.
Project Manager
2005-2015

Laboratory Supervisor
2003 to 2015

Laboratory Technician
2001 to 2003

REPRESENTATIVE EXPERIENCE

Mr. Giangiordano has over 22 years combined environmental laboratory and data management experience and possesses certifications as a Project Management Professional (PMP) and Scrum Master as well as a Laboratory Analyst. Mr. Giangiordano came to Laboratory Data Consultants, Inc. with over 14 years of hands-on environmental laboratory experience at an accredited full service environmental analytical chemistry facility and now specializes in supporting the company's custom software products.

- As Senior Environmental Informatics & Software Support Specialist with LDC, Mr. Giangiordano has a thorough knowledge and understanding of the company's branded software and has led numerous workshops and training sessions for clients ranging from laboratory personnel to consulting firms to USACE. Mr. Giangiordano specializes in tending to client software and electronic data deliverable needs and provides technical support throughout the life of LDC's various custom software products. He has extensive experience in electronic data deliverables and electronic data deliverable review and provides database support and management solutions for clients using LDC's custom environmental database management system (EDMSi).
- As a Project Manager at EnviroMatrix Analytical, Inc., an accredited full service environmental analytical chemistry facility, Mr. Giangiordano oversaw projects that provided analytical services and support to clients ranging from environmental consulting firms to marine biology firms, in addition to waste and wastewater treatment and disposal firms and municipalities. During this time, Mr. Giangiordano also served as liaison to US military in designing a wastewater compliance infrastructure that decreased analytical reporting limits and increased equipment capabilities



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- As Supervisor of the WET Chemistry and Microbiology Departments at EnviroMatrix Analytical, Inc., Mr. Giangiordano was responsible for all department functions which included overseeing daily operations, training staff, final reporting of analytical data, compliance with method requirements, as well as introducing and developing new methods for additional accreditation.
- As an analytical chemist and microbiologist, Mr. Giangiordano performed the analysis of inorganic constituents and bacteriological contamination in drinking water, wastewater, soil, tissue, and sediment and was responsible for the final reporting of analytical data for these sections.



RESUME
AN LE

EDUCATION

B.S. Biological Science, 2000
University of California, Irvine

PROFESSIONAL HISTORY

Laboratory Data Consultants, Inc.
Senior Chemist
Feb 2017 to present

TestAmerica Analytical Inc., Irvine, CA
GCMS Analyst
2007 to 2017

EMSL Analytical Inc.
Industrial Hygiene Analyst
2006 to 2007

TestAmerica Analytical Inc., Irvine, CA
Wet Chemistry Analyst
2000 to 2006

REPRESENTATIVE EXPERIENCE

Ms. Le has 6 years of data validation experience.

- As a chemist at LDC, Ms. Le has performed data validation using USEPA National Functional Guidelines, client Quality Assurance Program documents, and the Department of Defense QSM depending on the project requirements for the clients.

Ms. Le has over 17 years of experience working in the lab and performing secondary data review in environmental testing field.

- As a Wet Chemistry Analyst at TestAmerica Laboratories, Ms. Le performed an extensive list of wet chemistry analyses including but not limited to Total Organic Carbon, pH, Conductivity, Biological Oxygen Demand, Total Dissolved Solids, Total Suspend Solids, Alkalinity, and Carbon Dioxide. Ms. Le has also performed Ion Chromatography analysis for Nitrite, Nitrate, Phosphate, Perchlorate, Chromium VI, and used the Spectrophotometer to analyze for Sulfide, Phenol, Chromium VI, Chemical Oxygen Demand (COD), Sulfactants (MBAS), Phosphorous, and Cyanide. As a Gas Chromatography Mass Spectrometry (GCMS) analyst, Ms. Le performed volatile organic compounds analysis according to methods 8260, 5030, 5035, and 624. Ms. Le was also responsible for training new analyst employees and performing second level review of data.
- At EMSL Analytical Inc., Ms. Le performed sample extraction and analysis of samples for metals using inductively coupled plasma (ICP) and flame atomic absorption (GFAA).



Relevant Project Experience

LDC has performed data validation and Quality Assurance services for contaminated sites overseen by AFCEE/AFCEC, Navy Southwest Division, DoE, DoD, EPA Superfund projects overseen by EPA Regions II, III, IV, IX, X, Brown Fields Cleanup for NY Sites, USACE projects reviewed by the Alaska, Baltimore, Louisville, Albuquerque, Seattle, Philadelphia, and Sacramento Districts, and Navy projects reviewed by NFESC.

LDC is the software developer and expert in the use of the Automated Data Review (ADR) software. LDC has been using the ADR.NET version and has the current Version in full implementation. LDC has performed over 1000 ADR projects in the past 10 years' worth over \$2,000,000 in revenue. ADR clients include, but are not limited to: Tetra Tech EC, Sealaska, AMEC, EPA, California DTSC, MWH, Trevet, Brown & Caldwell, AECOM, Shaw, ITSI, CDM, Weston Solutions and the San Gabriel Watermaster.

LDC has validated over 1,000,000 samples for analyses such as volatile organics (CLP, EPA Method 8240/8260), semivolatile organics (CLP, EPA Method 8270), organochlorine pesticides/PCBs (CLP, EPA Method 8081/8082), chlorinated herbicides (EPA Method 8151), purgeable halocarbons and aromatics (EPA Method 8021), trace metals (CLP, EPA Method 6010/6020/7000), PAHs by EPA 8310 and 8270, TOC analyses, hexavalent chromium, total petroleum hydrocarbons (EPA Method 8015/CDOHS LUFT), radiochemical constituents including gross alpha/beta, alpha spec, gamma spec, tritium, and uranium, and general minerals.

LDC has met their contractual turnaround time and quality requirements on over 99% of the projects completed.



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Project References/Experience

Name and Address, Contact Person, Telephone	Work Description and Location	Requested Deliverables	Number of samples/ Matrix	Value (\$)	Start/Stop
SESI Consulting Engineers 12A Maple Avenue Pine Brook, NJ 07058 ATTN: Mr. Steven Gustems Office: 862-702-5728 Mobile: 973-518-8547 Email: ssg@sesi.org	Huguenot Street Development, 33 Centre Avenue, New Rochelle, NY NYSDEC sites LDC performed Category B equivalent data validation Analyses included: VOC, SVOC, Pesticide, PCBs, PFAs, Metals, Wet Chemistry	Category B data validation, EDD Population, and NYSDEC DUSR reports	>1,000 Soil, Water, and Air	\$38872.24	12/2019-present
AECOM 1001 Bishop Street Suite 1600 Honolulu, HI 96813 ATTN: Ms. Alethea Ramos Office: 1-808-529-7283 Mobile: 1-808-389-5383 Email: alethea.ramos@aecom.com	Red Hill Bulk Storage Facility, CTO 18F0126 Data validation per Stage 2B and Stage 4 guidelines for volatile organic, semivolatile organic, pesticides/PCBs, herbicides, phenols, phosphorus pesticides, dioxin, trace metal, and wet chemistry analyses in soil, water, and tissue matrices. (Navy CLEAN IV, Honolulu, HI)	Stage 2B and 4 data validation reports, EDD, and DQAR reports	>2,000 samples Soil and Water	\$72,283.89	9/2018-present
EA Engineering 225 Schilling Circle Hunt Valley, MD 21031 ATTN: Ms. Tara Office: 410-584-7000 ext. 5172 Direct Dial: 410-329-5172 Email: Lamondtlamond@eaest.com	6332103 Off-Base Drinking Water Site Inspection, USACE Omaha PFAS Mitigation Various AFBs Data validation per Stage 2B and Stage 4 guidelines for PFAs analyses in soil and water, and tissue matrices.	Stage 2B and 4 data validation reports and EDD	>500 samples Soil and Water	\$10,787.60	4/2020-present



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Name and Address, Contact Person, Telephone	Work Description and Location	Requested Deliverables	Number of samples/ Matrix	Value (\$)	Start/Stop
<p>EA Engineering 225 Schilling Circle Hunt Valley, MD 21031</p> <p>Project Manager ATTN: Ms. Brenda 615 Piikoi Street, Honolulu, HI 96814 Office: (808) 589-1455, x102 Fax: (808) 589-1575 Mobile: (808) 256-8268 E-mail: bnuding@eaest.com Nudingbnuding@eaest.com</p>	<p>Fairchild AFB LDC performed Stage2B and 4 data validation for PFAs and Wet Chemistry analyses and EDD population.</p>	<p>Stage 2B and 4 data validation reports. Work conducted under Working Copy Quality Assurance Project Plan for Environmental Remediation Services FAFB and PFAS Fairchild Air Force Base, Spokane County, Washington (July 2019) and the USEPA Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFAS) Analyzed Using EPA Method 537 (November 2018).</p>	<p>>400 Water</p>	<p>\$6,727</p>	<p>08/2019-present</p>
<p>Cape Inc. 500 Pinnacle Court, Suite 100 Atlanta, GA 30071 ATTN: Mr. Wayne Vermeychuk wvermeychuk@cape-inc.com Office: 727.940.4713 Mobile: 678.492.9384</p>	<p>Mather AFB LDC performed Level II and IV data validation for a full suite of analyses including GCMS, GC, Metals, and Wet Chemistry analyses and ERPIMS EDD Upload.</p>	<p>Level II and IV data validation reports. Work conducted under Draft Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) for Environmental Services for Western Region Base Realignment and Closure (BRAC) Bases, Mather Air Force Base (AFB), California (February 2017), the DoD QSM 5.2 (2018), and a modified outline of the USEPA NFG.</p>	<p>>1700 Water and Air</p>	<p>\$55,142</p>	<p>02/2017-present</p>



LABORATORY DATA CONSULTANTS, INC.

2701 Loker Ave. West, Suite 220, Carlsbad, CA 92010 Bus: 760/827-1100 Fax: 760/827-1099

Name and Address, Contact Person, Telephone	Work Description and Location	Requested Deliverables	Number of samples/ Matrix	Value (\$)	Start/Stop
Ayuda Companies 410 Acoma Street Denver, CO 80204 ATTN: Susan Royse Office: 303.999.2146 Fax: 303.999.2099 sroyse@ayudacompanies.com	Various Omaha AFB Sites LDC performed Level III and IV data validation for a PFAs analyses and ADR and SEDD population, and ERPIMS EDD Upload.	Level III and IV data validation reports. Work conducted under Addendum 3 to the Final Uniform Federal Policy – Quality Assurance Project Plan for Site Inspection of Aqueous Film-Forming Foam Areas, United State Air Force Academy (July 2018), the DoD QSM) 5.1 (2017), and the DoD General Validation Guidelines (February 2018).	>360 Water and Soil	\$8434	02/2017-present
Washington State Department of Transportation Environmental Services Office P.O. Box 47332 Olympia, WA 98504 ATTN: Mr. Brad Archbold ArchboB@wsdot.wa.gov 360-570-6636	WSDOT NPDES Stormwater Monitoring LDC performed Stage2A, 2B and 4 data validation for a full suite of analyses including GCMS, GC, Metals, and Wet Chemistry analyses.	Stage 2A, 2B, and 4 data validation reports. Work conducted under Washington State Department of Transportation Stormwater Monitoring	>3,800 Soil and Water	\$48,332	04/2013-07/2016
Leighton Consulting, Inc. 17781 Cowan Irvine, CA 92614 ATTN: Mr. Mark Withrow mwithrow@leightongroup.com cell: 949-394-2194 office: 949-681-4211	San Onofre Nuclear Generating Station (SONGS) Mesa Facility LDC performed EPA Level III and IV equivalent data validation for a full suite of analyses. Analyses included GCMS, GC, Metals, and Wet Chemistry analyses.	EPA Level III and IV data validation reports. Work conducted under USEPA Contract Laboratory Program National Functional Guidelines (CLPNFG).	>3,600 Soil, Water, and Air	\$149,714	09/2015-present
Leighton Consulting, Inc. 17781 Cowan Irvine, CA 92614 ATTN: Ms. Julie Harriman jharriman@leightongroup.com Direct : (949) 681-4264 Cell: (949) 572-8129	Aliso Canyon LDC performed EPA Level II equivalent data validation. Analyses included VOA, SVOA, Total Hydrocarbons, Isopropyl Alcohol, Total Dust, and Sulfur Compounds.	EPA Level II data validation reports and PARCC summary report. Work conducted under USEPA Contract Laboratory Program National Functional Guidelines (CLPNFG).	>1,200 Air, Wipe, and Disk	\$15,749	07/2016-08/2016



LABORATORY DATA CONSULTANTS, INC.

2701 Loker Ave. West, Suite 220, Carlsbad, CA 92010 Bus: 760/827-1100 Fax: 760/827-1099

Name and Address, Contact Person, Telephone	Work Description and Location	Requested Deliverables	Number of samples/ Matrix	Value (\$)	Start/Stop
Tetra Tech, EM Inc. 1999 Harrison Street, Suite 500 Oakland, CA 94612 ATTN: Ms. Sara Woolley Sara.Woolley@tetratech.com Direct: 510.302.6311 Main: 510.302.6300	Subcontract 161408 For Various project sites including: EAGLE NEST INVESTIGATION FORT IRWIN GOLD BEACH MILL HPNS MARE ISLAND MOTCO LITIGATION NAF EL CENTRO NWS CONCORD LDC performed cursory and Full data validation for a full suite of analyses using specified EPA Guidelines, DoD QSM Version 4.2, and Tetra Tech EMI, Inc. validation documents.	TTEMI Format data validation reports and EDD using Tetra Tech's validate program.	>3000 Soil and Water	\$39,785	10/2011 – 10/2013
GEI Consultants, Inc. 455 Winding Brook Drive Glastonbury, CT 06033 (860) 368-5342 direct (860) 368-5300 main Jaimie Wargo JWargo@geiconsultants.com	Various NYSDEC sites LDC performed Category B equivalent data validation Analyses included: VOC, SVOC, Pesticide, PCB, Herbicide, Steroids, Metals, Wet Chemistry	Category B data validation and NYSDEC DUSR reports	>1,700 Soil and Water	\$72,000	2010-present
TetraTech EC 17885 Von Karman Ave, Suite 500 Irvine, CA 92614 Attn: Lisa Bienkowski (949) 809-5028 Lisa.Bienkowski@tetratech.com	Tetra Tech Hunter's Point CA LDC performed EPA Level III and IV equivalent data validation for a full suite of analyses on more than 50,000 soil and water samples. Analyses included tritium, isotopic thorium, uranium and plutonium, and gross alpha/beta. Expedited turnaround times were included (5 day TAT)	EPA Level III and IV data validation reports. Work conducted under US Navy RAC program, Southwest Div.	>50,000 Soil and Water	\$645,733	02/2001-present
AECOM (Earth Tech) 700 Bishop Street Honolulu, HI 96813 Contact: Scott Lewis (808) 523-8874 Scott.Lewis@aecom.com	Data validation per EPA level "3/C" and "4/D" guidelines for volatile organic, semivolatile organic, pesticides/PCBs, herbicides, phenols, phosphorus pesticides, dioxin, radiochemical, and trace metal analyses in soil, water, and tissue matrices. (Navy PACDIV CLEAN, Honolulu, HI)	LDC worksheets and validation reports	>10,000 samples Water/Soil/Air	\$750,000	4/98-present



LABORATORY DATA CONSULTANTS, INC.

2701 Loker Ave. West, Suite 220, Carlsbad, CA 92010 Bus: 760/827-1100 Fax: 760/827-1099

Name and Address, Contact Person, Telephone	Work Description and Location	Requested Deliverables	Number of samples/ Matrix	Value (\$)	Start/Stop
CBI (formerly Shaw E&I) 3347 Michelson Drive, Ste 200 Irvine, CA 92612 Contact: Mr. Dwayne Ishida Phone: (949) 660-7561 Dwayne.Ishida@CBIFederalServices.com	Data validation per EPA level "3" and "4" and AFCEE/AFCEC guidelines for volatile organic, semivolatile organic, pesticides/PCBs, herbicides, phenols, phosphorus pesticides, dioxin, radiochemical, and trace metal analyses in soil, water, and tissue matrices. (Navy Southwest Division RAC, San Diego, CA and various AFCEE/AFCEC projects)	LDC worksheets and validation reports	>5000 samples Water/Soil/Air	\$350,000	6/06-present
Santa Clara Pueblo Office of Environmental Affairs 578 Kee Street Española, New Mexico, 87532 Ms. Ernestine Naranjo 505-692-6270 phone 505-747-2728 fax enaranjo@santaclarapueblo.org	Data validation per EPA level "III" SCP-OEA-DEPO, Data Validation using ADR For full suite of Organic, Inorganic, and Radiochemical analyses. Radiochemical analyses including Gross alpha & beta, Gamma Spectroscopy, Iodine, Radium-226/228, Strontium-90, Isotopic Pu, Th, and U, Tritium, and Americium by various EPA and GA methods.	Level III validation using ADR	>2000 Soil, Water, and Air	\$78621	12/2015 - present
Anchor Environmental, LLC 720 Olive Way, Suite 1900 Seattle, WA 98101 Ms. Joy Dunay 206.287.9130, jdunay@anchorage.com	Data validation per Level "C" Newtown Creek Phase 2: Third Party Data Validation of laboratory results, EDD population, and Data Quality Assessment Reports (DQAR) for various methods Subcontractor	LDC worksheets and validation reports	>63,000 Soil and Water	\$743,793.88	6/14-1/16
P.W.Grosser Consulting2015 630 Johnson Ave, Suite 7 Bohemia, NY 11716 Attn: Mr. Derek Ersbak w. 631.589.6353 f. 631.589.8705 dereke@pwgrosser.com	Former Arkansas Chemical Co.Site and Former Ronkonkoma Wallpaper Site 203 Jay St. LDC performed Category B equivalent data validation Analyses included: VOC, SVOC, Pesticide, PCB, Metals, Wet Chemistry	Category B data validation and NYSDEC DUSR reports	>200 Soil and Water	\$3,024.00	11/2014-present
Amec Foster Wheeler Environment and Infrastructure, Inc. 9210 Sky Park Court, Suite 200 San Diego, CA 92123 Attn: Mr. Rolf Schottle rolf.schottle@amecfw.com Tel +1 (858) 300 4300, Fax +1 (858) 300 4301, Direct +1 (858) 300 4323	Regional Harbor Monitoring Program (RHMP), San Diego, California Third party validation of LDC performed EPA Level III and IV equivalent data validation for a full suite of analyses.	LDC worksheets and validation reports	>200 Water	\$9,011.40	3/15-6/16

Note: All above projects were 100% self-performed by LDC



APPENDIX B

Field Logs

GENERAL			
Date		Start Time / End Time:	/
AM Weather (Temp/Wind):		PM Weather (Temp/Wind):	
LaBella field representative completing form		Names of team members on Site:	
Vistors on Site:			
General summary of tasks completed			
Deviations from CAP			
CAMP			
Upwind location		Upwind dust meter/PID equipment ID	
Downwind location		Downwind dust meter/PID equipment ID	
Exceedances for dust (Y/N), explain		Exceedances for VOCs (Y/N), explain	
Description of dust/ VOC suppression used			
EXCAVATION/ BACKFILL			
Description of area excavated			
Daily # Trucks Contaminated Soil/ Bedrock Removed		Daily # Trucks Backfill Delivered	
Total # Trucks Contaminated Soil/Bedrock Removed		Total # Trucks Backfill Delivered	
Estimated Total Tonnage Soil/ Bedrock Removed		Estimated Total Tonnage Backfill Delivered	
CONFIRMATORY/ DOCUMENTATION SOIL SAMPLING			
Summary of sampling locations/ types			
Sample IDs			
MS/MSD/Duplicates (1 per 20)			
DEWATERING			
Approx. daily volume water removed during Site work:		Water Samples for discharge/disposal:	
Removed water from Sitework container description (type/size/quantity/location):		Water discharge/disposal location and amount (if discharged):	

Site Sketch

Field Notes



BORING LOG

Boring No. GP-

Sheet 1 of 1

Project No.:

CHKD BY:

Project Name:

Location:

Client:

Drilling Firm:

Driller:

Start Date:

Finish Date:

Key:

- Geologic Strata Change
- - - - - Gradation Change Within Strata
- End of Boring or Overpacked

Drill Rig:

Casing:

Sampler: Macro-Core - 5ft length, 2 in diameter

Sampling Method: Direct Push

Hammer:

Core Type:

Time Start:

Time End:

LaBella Rep.

Other

Boring Location:

Depth (ft)	Sample ID	Depth of Change (ft)	VISUAL-MANUAL MATERIAL DESCRIPTION	PID <small>(parts per million)</small>	COMMENTS <small>(e.g., Native, core run, RQD, % recovered)</small>
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

DEPTH (FT)			ADDITIONAL NOTES:		
WATER LEVEL DATA			BOTTOM OF CASING	BOTTOM OF BORING	GROUNDWATER ENCOUNTERED
Date	Time	Elapsed Time			

GENERAL NOTES

1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL.

2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER

BGS = Below Ground Surface and = 35 - 50% C = Coarse R = Rounded

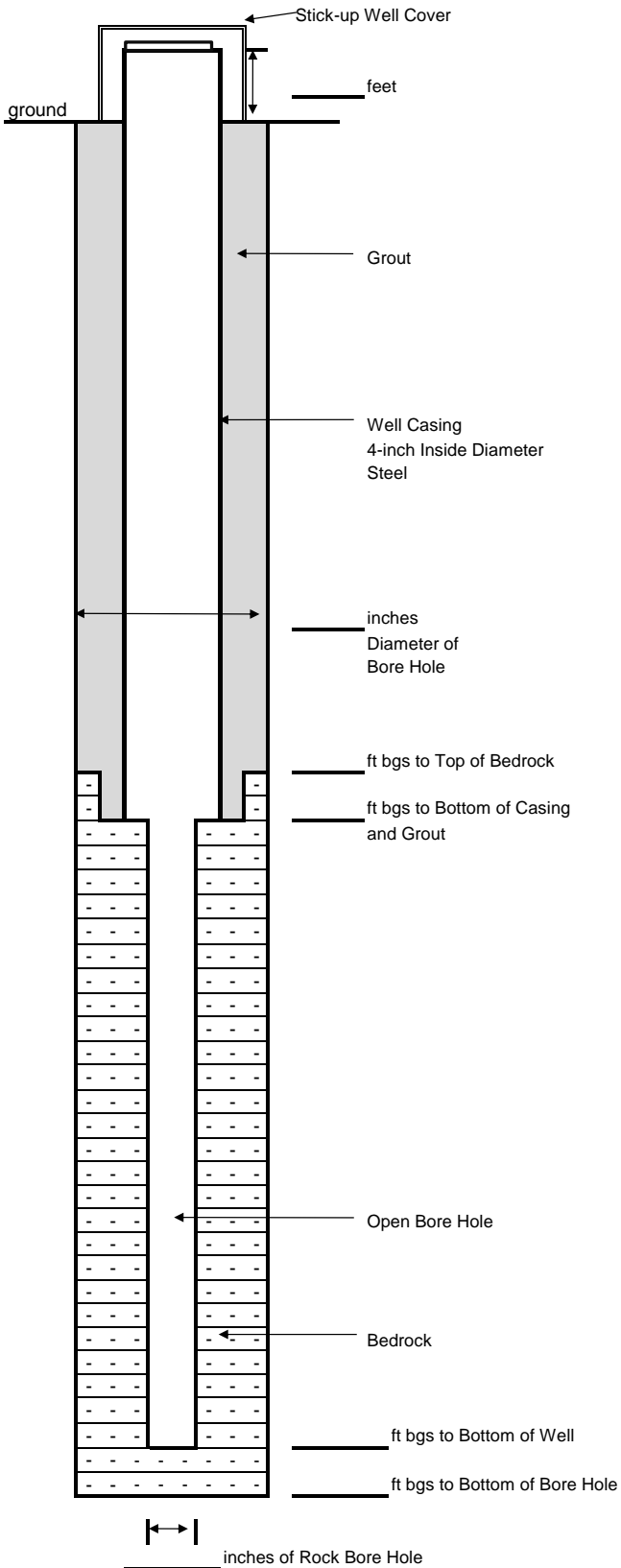
NA = Not Applicable some = 20 - 35% M = Medium A = Angular

BC = Blow counts per 6" sampler little = 10 - 20% F = Fine SR = Subrounded

NR = No Sample Recovery trace = 1 - 10% VF = Very Fine SA = Subangular

BORING: GP-

WELL CONSTRUCTION LOG



Well No.:

Project: _____
 Address: _____
 Town/City: _____ State: _____
 Project No. _____ County: _____
 Installation Date(s): _____
 Drilling Method: _____
 Drilling Contractor: _____ Driller: _____
 Drill Rig: _____
 Drilling Fluid: _____
 Datum: _____ Elevation: _____ ft

Well Development Information

finished with protective stick-up well cover, j-plug

Static Water Level: _____ feet from top of casing/ground/other
 Fluid Lost During Drilling: _____ gallons
 Water Removed During Development: _____ gallons
 Date(s) of Development: _____
 Purging Method: _____ Sampling Method: _____
 Well Cover Size/Tools Needed to Open: _____

Notes: ft = feet, bgs = below the ground surface

Prepared By: _____



APPENDIX C

Laboratory Quality Assurance Manual

Quality Systems Manual

Alpha Analytical, Inc.

D/B/A

Alpha Analytical
Eight Walkup Drive
Westborough, MA 01581-1019
Telephone: (508) 898-9220
Facsimile: (508) 898-9193

Alpha Analytical
Woods Hole Labs Facility
320 Forbes Blvd
Mansfield, MA 02048
Telephone: (508) 822-9300
Facsimile: (508) 822-3288

Email: info@alphalab.com
Web site: www.alphalab.com

Quality Assurance Officer: James Todaro, Ext 508-439-5101
Laboratory Technical Manager (Director) Organics-Westboro: Marco Soares 508-439-5144
Laboratory Technical Manager (Director) Inorganics-Westboro: Elena Dayn 508-439-5131
Laboratory Technical Manager (Director) Mansfield: John Trimble, 508-844-4134
Laboratory Technical Manager (Director) Air-Mansfield: Andy Rezendes, 508-844-4181

Approvers:

Jim Todaro Approved on 11/18/2022 2:13:40 AM, Elena Dayn Approved on 11/17/2022 2:40:13 PM, John Trimble Approved on 11/17/2022 2:58:48 PM, Andrew Rezendes Approved on 11/17/2022 4:46:26 PM, Marco Soares Approved on 11/17/2022 6:40:52 PM

1 Mission Statement

The mission of Alpha Analytical is quite simply to provide our customers with the greatest value in analytical service available. For the 'greatest value' is not only found in the data that is delivered, it is also found in the services provided.

- Data must be of the highest integrity, accuracy and precision.
- Consultation and educational services must be provided to support the customer in establishing data quality objectives and interpretation of the final data package.
- Support services such as sample containers, courier service and electronic data deliverables must be available to the customer.

Alpha's mission continues with an established commitment to our community and environment. We must ensure that we do not produce any additional contamination to our environment or harm our neighbors and community in any way.

The value of Alpha's product is in the honesty and integrity with which each chemist, courier, login staff member, or office staff member performs their tasks. The customer or employee must always feel satisfied that they received the greatest value in their lab experience at Alpha.

Alpha Analytical will vigorously pursue its mission into the next millennium.

Mark Woelfel
President

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3 Introduction

The Quality Systems Manual, referred to as Corporate Quality Systems Manual (CQSM) of Alpha Analytical describes the quality program in use at the laboratory for both Westboro and Mansfield facilities. This Quality Systems Manual provides employees, customers and accrediting agencies with the necessary information to become familiar with how the quality system operates within Alpha Analytical. The quality program includes quality assurance, quality control, and the laboratory systems including feedback mechanisms for the automated continuous improvement of the laboratory operations to meet customer needs.

Implementation of the laboratory operations is by documenting procedures, training personnel and reviewing operations for improvement. Written procedures are maintained as Standard Operating Procedures (SOPs). The SOPs are available to the staff as a controlled, electronic, secure copy. The provisions of the QSM are binding on all temporary and permanent personnel assigned responsibilities. All laboratory personnel must adhere strictly to the QSM and SOPs.

All policies and procedures have been structured in accordance with the NELAC Institute (TNI Standards), DOD QSM 5.4 and applicable EPA requirements and standards.

Twenty-five (25) sections comprise the QSM. Related quality documentation including the listing of SOPs, forms, floor plan, equipment, personnel and laboratory qualifications are available. The QSM sections provide overview descriptions of objectives, policies, services and operations.

3.1 Scope

The QSM describes the requirements of the Laboratory to demonstrate competency in the operations for performing environmental tests for inorganic, organic, air and microbiological testing. The basis for the environmental tests is the methods found in documents published by the United States Environmental Protection Agency (EPA), ASTM, AOAC, APHA/AWWA/WEF, Standard Methods, and other procedures and techniques supplied by customers.

The QSM includes requirements and information for assessing competence and determining compliance by the laboratory to the quality system. When more stringent standards or requirements are included in a mandated test method, by regulation, or specified in a project plan the laboratory demonstrates achievement of the customer specified requirements through its documented processes.

The QSM is for use by Alpha Analytical for developing and implementing the quality system. Accrediting authorities and customers use the QSM for assessing the competence of Alpha Analytical. Alpha Analytical is committed to continually improving the quality system. Meeting customer needs, operating within regulatory requirements and adhering to Alpha's Data Integrity and Ethics policy are several of the mechanism used to continually improve the quality system.

3.2 Policy Statement

This Quality Systems Manual summarizes the policies, responsibilities and operational procedures associated with Alpha Analytical. This manual applies to all associates of the laboratory and is intended for use in the on-going operations at Alpha Analytical. Specific protocols for sample handling and storage, chain-of-custody, laboratory analyses, data reduction, corrective action, and reporting are described. All policies and procedures have been structured in accordance with the NELAC Institute (TNI) Standards, DOD QSM(which includes 17025 standards), applicable EPA requirements, regulations, guidance, and technical standards. This Quality Systems Manual, laboratory Standard Operating Procedures (SOPs), and related documentation describe the quality systems, policies and procedures for Alpha Analytical.

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Alpha Analytical performs chemical analyses for inorganic and organic constituents in water, seawater, soil, sediment, oil, tissue and air matrices. Alpha Analytical's goal is to produce data that is scientifically valid, technically defensible, and of known and documented quality in accordance with standards developed by The NELAC Institute (TNI) Standards and any applicable state or EPA regulations or requirements. It is the commitment of the President, Operations Director, Laboratory Technical Manager and Quality Assurance Officer to work towards continuous improvement of the operation, and towards meeting our customer's needs, requirements, and intended data usage. This continued commitment is built into every activity of the laboratory. It is the responsibility of Senior Management and the Department Managers to ensure that all associates familiarize themselves with, and comply at all times with, the quality systems, procedures and policies set forth in this manual, laboratory SOPs, and related documentation.

Alpha Analytical analyzes Proficiency Test (PT) samples, in accordance with the NELAC Institute (TNI) Standards and other regulatory programs, from a National Institute of Standards and Technology (NIST)-approved PT provider for the analytes established by EPA for water samples, and for other analytes and matrices. The specific analytes and matrices analyzed are based on the current scope of the laboratory services as documented in the laboratory SOPs and state certifications.

The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. This includes a review of facilities and instrumentation, staffing, and any special QC or reporting requirements to ensure that analyses can be performed correctly and within the expected schedule. All measurements are made using published reference methods or methods developed by Alpha Analytical. Competence with all methods is demonstrated according to the procedure described in SOP/1739 prior to use.

Alpha Analytical has developed a proactive program for prevention and detection of improper, unethical or illegal actions. Components of this program include: internal proficiency testing, electronic data audits and post-analysis data review by the QA Officer; a program to improve employee vigilance and co-monitoring; and Ethics Training program identifying appropriate and inappropriate laboratory practices, instrument manipulation practices and consequences. Additionally, all associates are required to sign the Alpha Analytical *Ethics Agreement* form upon commencement of employment and complete annual refresher Ethics Training thereafter. This form clearly outlines the possible consequences of unethical or improper behavior, or data misrepresentation. All staff are required to report any suspected unethical conduct to management. Management will then investigate and determine if the situation was considered unethical and will take appropriate action as described in the Alpha Ethics policy.

It is the policy of the laboratory to discourage and reject all influence or inducements (whether commercial, financial or personal) offered either by customers or suppliers, which might adversely affect results or otherwise compromise the judgment or impartiality of the staff. It is the responsibility of the Operations Director and Laboratory Technical Manager to inform customers and suppliers of this policy when necessary.

In the event that any such influences or inducements are encountered, the staff is instructed to inform management immediately. It is the responsibility of the Operations Director and the Laboratory Technical Manager to take appropriate action to prevent recurrence.

3.3 References

External reference documents are available electronically in the Qualtrax system for staff to access the latest edition or version of the reference methods, regulations or national standards. The Quality Assurance Department maintains the electronic files in the Qualtrax system. Management purchases automated update services, where available, to provide the laboratory with the latest hardcopy edition, where electronic means is not available.

3.4 Definitions

Appendix A lists the definitions as adopted by the laboratory. The definitions are mostly from the 2016 TNI standards and other sources.

4 Organization and Management

4.1 Legal Definition of Laboratory

Alpha Analytical is a full service analytical laboratory. Testing services include Drinking Water, Waste Water, Ground Water, Waste material and Air. Alpha Analytical is a privately held corporation incorporated in the state of Massachusetts. Alpha Analytical, Inc. does business as (D/B/A) Alpha Analytical.

Alpha Analytical has been in business since 1985. The types of businesses served include:

- Consulting firms,
- Engineering firms,
- Waste Management Companies,
- Industrial sites,
- Municipal agencies
- Department of Defense projects.

4.2 Organization

The laboratory operates a quality system approach to management in order to produce data of known quality. The laboratory organization provides effective communication and lines of authority to produce analytical data meeting customer specifications. The organizational design provides open communication while ensuring that pressures and day to day operating circumstances do not compromise the integrity of the reporting of the final data. See Appendix B for Organizational Chart.

The President is responsible for directing all areas of the company. The following job functions report to the President:

- Operations Manager
- Quality Assurance Officer
- Marketing / Business Development / Sales
- Financial Services
- Human Resources

The Operations Manager is responsible for directing all laboratory operational areas of the company. The following job functions report to the Operations Manager:

- Laboratory Technical Manager(s)
- Customer Services Manager
- Department Managers

The Laboratory Technical Manager(s) is(are) responsible for the laboratory data generated by the organics testing, inorganics testing and metals testing areas and the Air Technical Director is responsible for laboratory data generated by air analyses.

The Departmental Managers (Supervisors) have the following responsibilities:

- The organics managers direct personnel in the organics extraction and instrumental laboratories.

The wet chemistry manager directs personnel and team leaders in the wet chemistry and/or microbiological testing areas.

The metals manager directs personnel and team leaders in the metals sample preparation and instrumental laboratories.

The Quality Assurance Officer is a member of the staff and reports directly to the President and has defined responsibility and authority for ensuring that the quality system is implemented and adhered to at all times. The Quality Assurance (QA) Officer is responsible for interacting and communicating certification requirements, implementing the Quality Systems Manual and reporting to the Laboratory Technical Manager and Senior Management the status of the quality program. The QAO oversees the Quality Systems Specialists and is responsible for oversight and/or review of quality control data and function independently from laboratory operations.

The Customer Services Manager is responsible for customer interactions, project coordination and laboratory personnel notification of project requirements.

The Marketing, Business Development and Sales personnel are responsible for increasing the volume of work from current customers and adding new customers to the base business of Alpha Analytical. The Marketing and Business Development personnel review all new work with the Laboratory Technical Manager, Operations Manager, President and/or Quality Assurance Officer before contractual commitment.

The CFO is responsible for maintaining and reporting on the financial status of the company. The CFO directs financial personnel on proper accounting procedures and maintaining the list of approved suppliers and subcontractors. The CFO reports directly to the President.

The Human Resource Director is responsible for personnel recruitment, hiring, performance reviews.

Personnel job descriptions define the operational function duties and responsibilities. Administration and Laboratory personnel assignments may include cross-functional training and work performance in multiple areas of the operations. Multiple function training ensures laboratory back up personnel during peak workloads.

During the absence of any staff member, assignment of alternative personnel occurs by memo or e-mail. The Manager or Supervisor authorizes the assignment. The naming of alternative personnel assures the continuing performance of critical tasks during the primary person's absence and ensures that lines of communication remain open for continued decision making. The deputy for the Laboratory Technical Manager is the Quality Assurance (QA) Officer. The deputies for the Quality Assurance (QA) Officer are the Quality Systems Specialists.

For the purposes of the NELAC Institute (TNI) Standards the Lead Laboratory Technical Manager is the Laboratory Technical Manager. The deputies for the Lead Technical Manager are the Quality Assurance (QA) Officer, and the Departmental Managers. The Laboratory Technical Manager meets the requirements specified in the Section 4.1.7.2 Volume 1, Module 2 of the 2016 TNI standards. If the Laboratory Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, a full-time staff member meeting the qualifications of Laboratory Technical Manager will be designated to temporarily perform this function. The primary Accrediting Body shall be notified in writing if the Technical Manager's absence exceeds 35 consecutive calendar days.

4.3 Business Practices

Alpha maintains certification for the programs and analytes required by regulatory programs. The listing of qualifications from the various certifications, registrations and accreditation programs are available upon request. Alpha Analytical operates Monday to Friday from 7:30 a.m. to 5:30 p.m. Management prepares and posts the holiday schedule for the year indicating closed operations. Sample delivery occurs during normal operating hours unless arranged in advance.

Alpha's reputation depends upon timely reporting and quality data. The standard turnaround time for engineering and consulting firms is five business days from time of sample receipt. Standard turnaround for all other customers is ten business days from time of sample receipt. The time of sample receipt is when the verification of the chain of custody and samples meets the laboratory sample acceptance policy. Laboratory management must approve any special arrangements for rush or expedited turnaround time. The basis for data quality depends on customer, regulation and method performance criteria. Accuracy, precision, sensitivity and comparability are expressions of method performance criteria.

All work is performed in the strictest confidence. New and contract employees must review corporate policy and practice requirements for protecting customer confidentiality and proprietary rights. The review occurs during orientation and ethics training. It is the policy of the laboratory to release data to the customer authorized contact. Personnel assigned the duties of interacting with customers review project files and discuss data related only to the project. Personnel whose duties do not include routine customer contact must check with the customer service manager before discussing data with regulators or third parties

5 Quality System

Establishment, Audits, Essential Quality Controls and Data Verification

5.1 Establishment

The Mission Statement presents the policy and objectives for Alpha Analytical. The Quality Systems Manual provides the framework for the processes and operations to implement the Mission. The Quality Systems Manual and documentation controlled by the laboratory system detail the management authorized operations for achieving the objectives of the company.

The laboratory operates a quality system approach to management in order to produce data of known quality. Alpha Analytical is a full service laboratory designed to provide its customers with accurate, precise and reliable data within the best turn-around time and at the most reasonable prices. Alpha employs chemists of the highest training, ethics and caliber in the field of analytical chemistry. This and state-of-the-art instrumentation and automation combine to insure data of known and documented quality.

5.2 Quality Systems Manual

The QA Officer is responsible for the publication and distribution of the Quality Systems Manual and annual review. Management reviews and authorizes the manual. Implementation of major changes in the quality system occurs after revision of the appropriate Quality Systems Manual section and authorization by management.

The authorization of the Quality Systems Manual is documented electronically in Qualtrax. Updates of this manual occur at any time throughout the year. Document control procedures (SOP1729) apply to the distribution of the Quality Systems Manual. Controlled copies of the manual are maintained electronically within Qualtrax. Persons or organizations outside of Alpha Analytical may receive uncontrolled copies. Copies are distinctly indicated "Uncontrolled Documents" within the footer of each page.

5.3 Audits

Laboratory audits, both internal and external, review and examine the operations performed in the laboratory. Internal audits are conducted by qualified QA Specialists and external audits are reviews by external organizations to evaluate the ability of the laboratory to meet regulatory or project requirements. Internal audits are conducted on a frequency of annually, or method required.

A QA designee schedules internal process audits to ensure the completion of the annual audit of each operational area. The process audits are a more detailed review of the operations. Personnel from areas other than the one audited perform process audits.

The internal system audit is a review of the implementation of the documented quality system. The system audit includes sample tracking from receipt to disposal, a data audit of a completed report, and all operations not audited during the process audit.

The purpose of the internal system audit is:

- Verification that adequate written instructions are available for use;
- Analytical practices performed in the laboratory are consistent with SOPs;
- The quality control practices are applied during production;
- Corrective actions are applied as necessary;

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Deviations from approved protocols are occurring only with proper authorization and documentation;
Reported data is correct and acceptable for reporting;
SOPs, quality records, analytical records, electronic data files are maintained properly; and
Personnel training files and records are satisfactory and current.

Before a scheduled internal audit, the assigned auditor reviews checklists, if used, and/or the SOP specific to the area. The checklist may be from an external source or prepared by the auditor. After the audit, the auditor submits a summary or notes from the audit to the Laboratory Technical Manager or QAO as part of the audit report. The summary identifies discrepancies found during the audit. Technical personnel are responsible for the inspection and monitoring of in-process and final data. Personnel independent of those having direct responsibility for the work performed audit the quality system and processes.

Representatives sent by customers and government or accrediting agencies often perform external audits. These audits are most often announced inspections, but sometimes are not announced. The Quality Assurance Officer, Laboratory Technical Manager or assigned deputy, and/or appropriate Department Manager accompany the external audit team through the laboratory. The auditors receive a brief overview of company objectives, activities, and facilities. Interviews with essential supervisory staff and technical staff are arranged, along with retrieval of any documentation pertinent to the audit. Auditors usually provide a report on their findings shortly after the audit. The QA Officer receives the audit report and copies are provided to laboratory personnel for review. Corrective actions are identified and distributed to responsible parties for implementation in response to any cited deficiencies.

5.4 Audit Review

Management reviews internal and external audit reports to evaluate system effectiveness at the annual management review meeting. Tracking of the audit findings occurs through the nonconformance action process. The management and staff work together to establish a time line for resolving the audit findings. The Quality Assurance team tracks the time line and reports to the Laboratory Technical Manager on any outstanding audit findings. Approved corrective actions for DoD that are not implemented or avoided may result in loss of DoD ELAP accreditation and may result in work being discontinued until implementation is verified by DOD ELAP AB.

5.5 Performance Audits

Alpha Analytical participates in inter-laboratory comparisons and proficiency test programs required by customers and certifying agencies. The performance audits provide information on the data comparability of results generated by the laboratory. Test samples received by the laboratory are handled following routine laboratory procedures. Proficiency test samples are unpacked, checked against the packing slip and examined for damage. Reporting requirements and deviations to routine practices are noted as would be required for any project.

Analysts demonstrate proficiency by analyzing either an external proficiency test sample, an internally prepared blind test sample or Initial Demonstration of Capability (IDC) before independent operation of a test method. The results of performance audits serve several purposes. The QA Officer may use performance audits for evaluating analyst proficiency, laboratory performance in a specified area to facilitate laboratory improvement efforts, and/or to provide information to an accrediting agency on correction of past performance of an external performance audit.

5.6 Corrective Actions/Preventative Actions (CAPA)

The corrective action process at Alpha Analytical is detailed in SOP 1736. The corrective action program at Alpha Analytical uses the Quality Nonconformance workflow in Qualtrax to document and follow through the corrective action/preventative action process for three main areas: nonconformance's within the laboratory, customer complaints and failed PT studies. The process ensures continuous improvement of company performance by preventing the recurrence of quality problems.

Nonconformance reports are tracked for closure date and the type. Reports to management include the listing of open nonconformance reports and the frequency of the type of nonconformance occurring. A QA designee monitors the completeness of the forms, as well as verifies the actions are complete and acceptable.

Customers will be notified within 5 days of any question(s) regarding validity of results.

5.7 Managerial Review

The management review occurs at least once per year as part of the strategic planning process. Documentation of the management review meeting is by recording the meeting minutes and listing the attendees. The focus of the quality management review is the frequency of the type of nonconformance, closure status, audit progress and other quality assurance actions. Meetings include discussion and progress on quality system initiatives since the last meeting.

Prior to the meeting, an agenda is distributed to all personnel expected to be in attendance. The meeting is chaired by the President. Minutes are taken and distributed at the conclusion of the meeting by a QA designee. If action is necessary on any issue, a Summary Report is generated and distributed to responsible parties for implementation. Actions are monitored by the QAO or designee until completion.

5.8 Essential Quality Control Procedures

The following general quality control principles apply to all tests. The manner implemented is dependent on the type of test performed. The laboratory SOP presents the specific quality control checks undertaken to ensure precision, accuracy and sensitivity of each test method. Deviations from the existing SOP are allowed only upon approval of the deviation by the department manager and Quality Assurance Officer. This documentation must be either in form of written notice or email.

Alpha Analytical uses quality control samples to evaluate the following:

1. Adequate positive and negative controls to monitor blanks, spikes, reference toxicants, zero blanks;
2. Adequate tests to define the variability and/or reproducibility of laboratory results;
3. Measures to ensure the accuracy of the test data including sufficient calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples;
4. Measures to evaluate test performance, such as detection limits and quantitation limits or range of applicability such as linearity;
5. Selection of appropriate formulae to reduce raw data to final results such as linear regression, internal standards, or statistical packages;
6. Selection and use of reagents and standards of appropriate quality;

7. Measures to assure the selectivity of the test for its intended purpose;
8. Measures to assure constant and consistent test conditions for the method such as temperature, humidity, light, or specific instrument conditions.

Note: All quality control samples are treated in the same manner as field samples.

All quality control measures are assessed and evaluated on an on-going basis, and quality control acceptance limits are used to determine the usability of the data. Control charts and/or calculated control limits monitor the long-term method performance by analyte, by instrument for water matrices. Routine evaluation and reporting of the control chart performance provides supervisors and management with additional performance measures to ensure data comparability. Control limits are recalculated when trends are observed.

Where no reference method or regulatory criteria exist, the laboratory specifies the acceptance/rejection criteria in the SOP. The test SOP specifies the QC samples performed per batch of samples. The quality control samples are categorized into the following, as appropriate to the method

- Method Blank
- Laboratory Duplicate
- Laboratory Control Sample (LCS)
- Laboratory Control Sample Duplicate (LCSD)
- Matrix Spike (MS)
- Matrix Spike Duplicate (MSD)

Selection of samples for Duplicate, Matrix Spike (MS) & Matrix Spike Duplicate (MSD)

2. Duplicate samples

- a. Samples will be selected if identified and requested by customer
- b. If no samples are identified by the customer then random samples will be analyzed within the batch as defined by the method, program or at a minimum batch of 20 samples.

3. Matrix Spike (MS) / Matrix Spike Duplicate (MSD) samples

- a. Samples will be selected if identified and requested by customer
- b. If no samples are identified by the customer then random samples will be selected and analyzed within the batch as defined by the method, program or at a minimum batch of 20 samples.
- c. If MS/MSD is not required, LCS/LCSD may be substituted for

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precision and accuracy evaluation.
All DOD projects require MS/MSD.

The frequency is dependent on the reference method and test protocol. The following is the default requirement for quality control checks in lieu of any other guidance. The frequency for each quality control sample is generally one (1) per every 20 samples.

5.9 Data Reduction

After completion of the test procedure, the data reduction process begins.

Chromatography data may require the manual integration of peak areas or heights before reporting of results. The analyst must perform manual integration when software does not properly integrate or identify the peak. Manual integration must not occur for the purpose of achieving acceptable quality control or calibration. Signatures of analyst performing manual integrations can be found by electronic entry of analysts initials that can be traced to original signatures in the "Employee Signature Register". The analyst notes the rationale for performing the manual integration using the M-Codes listed in the manual integration SOP 1731 and ensures the "TIC" marks from the software represent the integration area used for reporting the results. The analyst must minimize and avoid manual integration. The establishment of the proper integration parameters in the software reduces the number of manual integration occurrences.

The SOP for each test presents the formulas used for the specific test method. The formulas for the data calculations used throughout the laboratory are the following:

% Recovery (LCS)

$$\frac{MV}{TV} * 100 = \%R_{LCS}$$

where: MV = Measured Value
 TV = True Value

% Recovery (MS or MSD)

$$\frac{MV - SV}{TV} * 100 = \%R_{MS}$$

where: MV = Measured Value
 TV = True Value
 SV = Amount found in sample

Average (\bar{X})

$$\frac{\sum_{i=1}^n X_i}{n} = \bar{X}$$

where: \bar{X} = Average of all values
 X = Result of each measurement
 n = Number of values

Relative Percent Difference (% RPD)

$$\frac{R_1 - R_2}{\frac{(R_1 + R_2)}{2}} * 100 = \%RPD$$

where: R_1 = Larger of two observed values
 R_2 = Smaller of two observed values

% Difference (%D)

$$\frac{X - \bar{X}}{\bar{X}} * 100 = \%D$$

where: \bar{X} = Average of all values
 X = Result of measurement

Standard Deviation of the sample (S_x)

$$\sqrt{\frac{\sum (X - \bar{X})^2}{n - 1}} = S_x$$

where: \bar{X} = Average of all values
 X = Result of each measurement
 n = Number of values

Relative Standard Deviation (%RSD)

$$\frac{S_x}{\bar{X}} * 100 = \%RSD$$

where: \bar{X} = Average of all values
 S_x = Standard Deviation ($n - 1$)

Range of Logs (for microbiological enumeration analysis)

10% of routine samples are analyzed in duplicate and the range of logs is determined.

MDL (See 40CFR Part 136 for details)

where: MDL = The method detection limit

$$\left[\sqrt{\frac{\sum_{i=1}^n x_i^2 - \left(\sum_{i=1}^n x_i\right)^2 / n}{n-1}} \right] * t_{0.99} = MDL$$

X = Result of each measurement

n = Number of values

t(n-1, 1 = .99) = The students' T value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. (See Students t Test Table)

Reporting Limit (RL)

Lowest calibration standard or greater

Control Limits

Upper Control Limit: $\bar{X} + 3 * S_x = UCL$
 Lower Control Limit: $\bar{X} - 3 * S_x = LCL$

Warning Limits

Upper Warning Limit: $\bar{X} + 2 * S_x = UWL$
 Lower Warning Limit: $\bar{X} - 2 * S_x = UWL$

Method of Standard Additions (MSA): (See EPA 7000A for details)

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_s of a standard analyte solution of concentration C_s. To the second aliquot (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration C_x is calculated:

$$C_x = \frac{SB V_s C_s}{(SA - SB) V_x}$$

where SA and SB are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that SA is roughly twice SB on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume.

For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance.

The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. A linear regression program may be used to obtain the intercept concentration.

5.10 Document Control

The Document Control Procedure (SOP/1729) describes the process for controlled and uncontrolled documents. The use of the revision number allows for the retention of a previous document for historical information purposes.

Every document is assigned a unique identification number, which is present on each page of the document. A master list of documents includes the unique identification. Each controlled copy includes the revision number, published date and page number.

Full document control includes the status of each document: active, inactive or superseded/archived. Inactive documents are procedures not currently requested, but may be in the future. Archived documents are procedures replaced with a later revision. Authorized personnel must review and approve each document and any subsequent revisions before use in the laboratory. Personnel authorized to review and approve a document have access to all necessary information on which to base their review and approval. The history section of the document in Qualtrax includes a description of the nature of the document change.

Standard Operating Procedures (SOPs) are instructions for repetitive or standard operations performed by the laboratory. The SOP author is the person familiar with the topic. The standard format for writing SOPs is set-up as a template for administration and technical SOPs. Each SOP is peer reviewed, authorized by management, and QA before final publication and implementation. Authorized signatories for controlled documentation include one or more of the following personnel: Company President, Quality Assurance Officer, Laboratory Technical Manager, Department Manager, Department Team Leader. Personnel acknowledges approved documents as read, understood and agreed to through electronic attestation forms associated with each document as SOP Attestation Tests which reside in Qualtrax.

SOPs must receive evaluation and input by laboratory supervisors and key technical personnel. The content of each SOP must conform to applicable requirements of analytical methods and certification agencies. Within these constraints, the content of a SOP meets the needs of a particular area of the laboratory. A new or revised SOP is needed when regulatory programs update or add methods, the scope of the existing method is extended, or when activities are being performed without adequate documentation.

Updating, modifying and changing SOPs, forms and the contents of this QSM are prompt and part of the routine practices. The prompt modification of these documents ensures the documents reflect the current practices and operations of the laboratory. During annual review of a document, (including but not limited to: SOPs, Ethics Policy, Quality Systems Manual), requested changes are reviewed and the document reissued using the information and a new revision number is assigned and published in Qualtrax.

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The laboratory maintains control over the possession and distribution of all documents that directly affect the quality of data. This includes, but is not limited to, documents such as the Quality Systems Manual, Standard Operating Procedures, customer instructions, Laboratory Work Instructions, data sheets, check lists and forms.

5.11 Detection Limits

Detection Limits (DLs), previously referred to as Method Detection Limits (MDLs), are determined for all analytes as specified in the Institute (TNI) Standards. DLs are determined for all new instrumentation, whenever there is a change in the test method or instrumentation that affects performance or sensitivity of the analysis. From these, detection limits, Reporting Limits (RLs), are established. The RL is the minimum concentration of an analyte that can be identified and quantified within specified limits of precision and bias during routine and analytical operating conditions.

Method Blanks are evaluated to determine an MDLb when performing an initial MDL study and annually thereafter.

Laboratory reporting limits lie within the calibration range, at or above the RL. For methods that require only one standard, the reporting limit is no lower than the low-level check standard, which is designed to verify the integrity of the curve at lower levels. If reporting limits are required below the lower level of the calibration curve, RL, or low-level check standard, method modifications are required. Refer to DL/LOQ SOP/1732. Note: "J" Estimated value: Upon customer request, the Target analyte concentration can be reported below the quantitation limit (RL), but above the Detection Limit (DL) with a "J" qualifier.

5.12 LOD/LOQ Studies

A. LOD (Limit of Detection) Verification - DOD only

1. LOD is required quarterly for all DOD projects. If there are no DOD projects for a particular quarter than LOD is not required for that quarter.
2. All sample-processing steps of the analytical method shall be included in the determination of the LOD.
3. The validity of the LOD shall be confirmed by **qualitative** identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 2-3X the LOD for single analyte tests, and > 1X up to 4X the LOD for multiple analyte tests. This verification must be performed on every instrument that is to be used for analysis of samples and reporting of data.
4. An LOD study is not required for any component for which spiking solutions or quality control samples are not available such as temperature. Where an LOD study is not performed, the laboratory may not report a value below the limit of quantitation.

B. LOQ (Limit of Quantitation) Verification

1. LOQ (Limit of Quantitation) verification is required quarterly for each target analyte. The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix. A successful analysis is one where the recovery of each analyte is within the established test

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method acceptance criteria for accuracy

The LOQ study is not required for any component or property for which spiking solutions or quality control samples are not commercially available or otherwise inappropriate (e.g., pH)..

Refer to DL/LOQ SOP/1732

5.13 Range of Logs – Precision of Quantitative Methods - Microbiology

- A. Precision of duplicate analyses is calculated for samples examined by enumerative microbiological methods according to the following procedure:
 - a. Perform duplicate analyses on first 15 positive samples.
 - b. Record duplicate analyses as D1 and D2 and calculate the logarithm of each result.
 - c. If either of a set of duplicate results is <1, add 1 to both values before calculating the logarithms.
 - d. Calculate the range (R) for each pair of transformed duplicates as the mean of these ranges.

6 Personnel

6.1 Laboratory Management Responsibilities

Management is responsible for communicating the requirements of the quality system, customer specifications and regulatory needs to all personnel. Management job descriptions detail the responsibilities of each position.

The H.R. Director has job descriptions for all positions in the laboratory defining the level of qualifications, training, and experience and laboratory skills. During initial training, management provides access to documented operations procedures, observes personnel performance, and evaluates personnel proficiency. Management documents technical laboratory staff's proficiency initially and on a continuing basis through use of laboratory control samples and purchased proficiency evaluation standards.

Management is responsible for verification of proper sample management and all aspects of data reporting. The communication of the operating practices of the laboratory is through the document control and attestation process.

Either the Quality Assurance Officer, Operations Director and/or Technical Managers have the authority to stop work due to non-conformances and have the authority to resume work after it has been stopped.

6.2 Laboratory Staff Requirements

Recruitment is the responsibility of the Operations Manager and HR Department, with input from other personnel as required. The Training Program procedure SOP/1565 details the process for completing requirements and training to ensure personnel have adequate skills and competence for the job function. Initial training includes ethics training, Qualtrax Training, QA Basics, IT/LIMs including computer security.

A job description details the necessary requirements for each job and includes position title, minimum educational requirements, skills, responsibilities and reporting relationships and any supervisory responsibility.

Initial training of new employees and contract staff includes laboratory ethics and quality policies, signing the Employee Signature Log, as well as execution of an Ethics Agreement. Any employee found to knowingly violate the Ethics Policy Agreement, report data values, that are not actual values obtained or improperly manipulated, or intentionally report dates and times of data analyses that are not the actual dates and times of analysis, will lead to disciplinary action, including termination, as outlined in Section V.K of the Employee Handbook. Each employee must report personally or anonymously to the Laboratory Technical Manager, QA Officer and/or Ethics Team Member any accidental or suspected intentional reporting of non-authentic data by others for follow up action. The review of the laboratory ethics and ethics training occurs annually with all personnel.

(DOD) All inappropriate and prohibited laboratory practices, as detailed in the DOD QSM 5.4, will be reported to the appropriate accrediting body within 15 business days of discovery. Records of corrective actions or proposed will be submitted within 30 business days. Failure to notify the AB within 15 business days will result in suspension of the DOD ELAP accreditation.

The Ethics program consists of the following key components:

- Ethics Policy /Agreement (Appendix E)
- Initial and annual ethics training

- Internal audits conducted annually
- Adherence to Manual Integration SOP/1731
- Ethical or Data Integrity issues reported to Lab Managers, QAO or HR Director
- Anonymous reporting to HR Director - This is accomplished by writing a detailed description of the suspected ethics breach and submitting the information, anonymously, to the Human Resource Director.
- “No-fault” policy encouraging reporting of incidences without fear of retribution
- Electronic tracking and audit trails through LIMs and instruments enabled where available.

6.3 Training

The Quality Systems Manual and related documentation is available to all employees. Cross training, supervisory training and other related training takes place on a scheduled and as-needed basis. Training ensures the communication and understanding of all personnel in the laboratory-documented procedures and practices.

All personnel undertake orientation-training sessions upon initial employment. Orientation training includes laboratory business practices, employment specifications, Ethics Policy, Quality Systems Manual, Chemical Hygiene Plan, and all SOPs required for the job function.

Managers ensure the training for new employees and review the continuing training for current employees. Training includes on-site and off-site programs presented by staff members, contractors, equipment manufacturers, and institutions of higher learning.

Training of new personnel to any job assignment takes place on-site according to the Training Program procedure. Laboratory personnel may perform their assigned methods/protocols without supervision only after documentation of acceptable proficiency. Training records lists the current training status.

On-the-job training includes demonstration of skills during job performance, initial demonstration of proficiency, and review of SOPs. Health and Safety training takes place on an annual basis with careful introduction to new principles. Personnel have access to the Chemical Hygiene Plan and Safety Data Sheets. On-site training includes side-by-side hands-on training, formal classroom type instruction on the SOP or a meeting to discuss procedural changes or to address questions related to the laboratory operation. All training is documented via the Training Attestation Form, which is signed by all in attendance that they understood and will implement what was presented to them.

Training is an on-going opportunity to evaluate the laboratory operations. The updating of SOPs, Quality Systems Manual and other related information documents all changes to the quality system. Training is documented via the Training Attestation Form or in Qualtrax with training test records.

Off-site training takes place on an as-needed basis. Recommendations and suggestions regarding educational programs come from all levels of staff. It is the employee’s responsibility to present a copy of any certificates or attendance information to the HR Director. The information is added to the individual’s training record.

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6.4 Records

The QA Department is responsible for maintaining training records. Credentials including certificates, transcripts, diplomas, resumes, and other records of training are placed in the individual's training file. Demonstrations of Capabilities are kept either in Qualtrax or LIMS.

Appropriate personnel are notified through email and/or Qualtrax or by the QA department when a revision is complete for the controlled version of a document. The manager of the area determines when a change is significant to require training.

Job descriptions are included in the training record files. The Human Resources Department reviews the job descriptions, Resumes and/or biosketches are kept on file with the Human Resources Department and the QA Department.

7 Physical Facilities – Accommodation and Environment

This laboratory facility has a total area of 25,000 square feet for each of the Westboro and Mansfield Facilities

The laboratory functional areas include:

- Administration and offices
- Sample receiving
- Sample management
- Air analysis (Mansfield Facility only)
- Microbiological (Westboro Facility only)
- General analytical chemistry
- Metals sample preparation (Mansfield Facility only)
- Organic sample preparation
- Metals analysis (Mansfield Facility only)
- Volatiles gas chromatography (GC)
- Volatiles gas chromatography/mass spectrometry (GC/MS)
- Volatiles air analysis (Mansfield Facility only)
- Semivolatiles gas chromatography/mass spectrometry (GC/MS)
- Semivolatiles gas chromatography (GC)
- Emerging Contaminants (Mansfield Facility only)
- Miscellaneous facility mechanical and storage areas.

All chemicals are stored in appropriate cabinets and properly disposed of as required. All flammable solvents are stored in OSHA and NFPA approved cabinets. Acids are stored in OSHA acid cabinets. Separate waste areas houses the sample and chemical waste before pickup by a licensed waste hauler.

7.1 Environment

Lighting, noise, humidity, heating, ventilation and air conditioning satisfy the needs of the testing performed on the premises. The laboratory building design ensures regulated temperature control for analytical equipment. Air-handling systems minimize airborne contaminants that may jeopardize sample integrity or analytical performance.

The analytical instrumentation is in separate rooms from laboratory activities that involve the use of large quantities of organic solvents or inorganic acids. A separate room, in the Westboro facility, provides the facilities for the microbiological testing.

Standards and other materials requiring below 0°C storage temperatures are placed in freezers and separated from samples or potential contaminating materials. Refrigerators provide cooling needs for samples and materials with temperature requirements of below room temperature and greater than freezing. Sample and standard storage areas are monitored and controlled for temperature and recorded in the data logger system. Sample storage areas for volatiles are separated from other samples and monitored for any effects due to cross contamination.

Bulk hazardous waste containers are located away from the testing activities. Waste disposal uses lab pack procedures and those designated by the regulatory authorities. The Chemical Hygiene Plan and the Waste Management and Disposal SOPs (Westboro: SOP/1728 and Mansfield SOP/1797)) include the procedures for handling and disposing of chemicals used in the laboratory.

The working and storage environments are maintained in a safe and appropriate manner. A Chemical Hygiene Plan details the requirements for safety and chemical handling. Safety measures that protect property and personnel from injury or illness include: fume hoods, fire extinguishers, fire blankets, alarm systems, safety training, protective clothing, emergency showers, eyewashes, and spill control kits.

7.2 Work Areas

Good housekeeping is the responsibility of all personnel. Each person is responsible for assuring clean and uncluttered work areas. The job descriptions list specific housekeeping duties. Records, samples and waste materials are the common cause for clutter in the laboratory.

. Removal of administration and laboratory records to the record storage area occurs to reduce clutter and ensure traceability. The individual filling the laboratory record box, labels the box with a number, the contents, date and laboratory area. Authorized personnel assign and record into a permanent record the box number, discard date and box contents. Authorized personnel review the box label for number, discard date and contents. Boxes are stored onsite and off-site for the record retention period identified in the NELAC Institute (TNI) Standards and EPA regulations, whichever is more stringent.

Sample management personnel remove samples to the sample storage area after all data is correct and complete. Sample coolers are removed to a designated storage area for recycling. Samples are stored in the designated process storage areas until testing is complete. Sample removal from the process storage occurs after mailing of the final report. The sample management staff places the samples in the archive storage area for thirty days after report release. The archive sample storage area is not controlled or monitored. Based on customer specifications, samples are properly disposed or returned to the customer.

Waste materials, expired reagents, expired standards and materials are disposed of and not stored in the laboratory. Hazardous waste labeled accumulation containers in the laboratory collect designated waste streams for later bulk disposal. Laboratory personnel remove the less than five-gallon accumulation containers when full from the laboratory and place the containers in the bulk hazardous waste area. Refer to the Waste Management and Disposal SOPs for Westboro: SOP/1728 and Mansfield SOP/1797. Personnel identifying out of date reagents and standards remove the materials to the proper disposal area.

7.3 Security

Alpha Analytical provides a secure environment for our employees, guests, customers, samples and analytical data. Security procedures require that all exterior doors remain locked unless manned. Access to the laboratory is limited to employees and contractors. Visitors not under signed contract are required to sign the Visitors Log and must be accompanied by a laboratory employee at all times within the testing areas.

The defined high security area is the sample management area. Identification card locks on the internal doors control entry into the laboratory area.

All doors are locked after hours and require a key for entry. The security alarm continuously monitors for smoke and fire related heat. When the alarm is activated, the appropriate emergency response officers are notified. The local emergency offices have the emergency contact list for the laboratory.

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8 Equipment and Reference Materials

8.1 Maintenance

The laboratory has a proactive equipment maintenance program. The laboratory maintains service contracts for most major equipment, which include routine preventative maintenance visits by the service provider. Technical personnel perform manufacturer's specified maintenance on a routine basis to ensure equipment operates at peak performance.

A brief summary of some common preventive maintenance procedures is provided in Appendix D. All instrument preventative and corrective maintenance is recorded in the maintenance logbook assigned to the equipment. After maintenance or repair, the instrument must successfully calibrate following the method SOP. Laboratory personnel must demonstrate quality control performance before sample analysis.

The laboratory maintains a stock of spare parts and consumables for analytical equipment. Backup instrumentation for some analytical equipment is available on site for use in case of major equipment failure. The person discovering or suspecting an equipment maintenance problem or failure tags the equipment with 'out of service' tag. If routine maintenance measures do not eliminate the problem, the Laboratory Technical Manager or Operations Director is notified and the appropriate equipment service provider is contacted.

All major laboratory equipment has individual and traceable maintenance logbooks in which to document manufacturer's recommended maintenance procedures, specific cleaning procedures, comments on calibration, replacement of small worn or damaged parts, and any work by outside contractors. The person performing routine or non-routine maintenance signs and dates the maintenance logbook. If an instrument is down for maintenance, a complete record of all steps taken to put it back into service is recorded including reference to the new calibration and quality control checks. Any equipment service providers working on the equipment are recorded in the logbook.

Record repetitive or on-going equipment problems other than normal maintenance requirements on nonconformance action forms. The nonconformance action form notifies management and the Quality Assurance Officer of a problem affecting the performance and data quality.

The laboratory groups some equipment into a single laboratory equipment maintenance logbook. Examples include: autopipets, thermometer calibration. The identity of each item is by serial number or a laboratory-designated item number. The same data recorded for major equipment applies to this documentation.

The maintenance records shall include:

- Equipment name;
- Manufacturer's name, type identification, serial number or other unique identification;
- Date received, date put into service, condition when received;
- Current location;
- Details of past maintenance and future schedule;
- A history of any damage, malfunction, modification or repair;
- Dates and results of calibration or verification.

The maintenance logbook may include the reference to the location of the equipment operational and maintenance manuals. The logbook may include the reference to laboratory run logbook or data files for the calibration and quality checks of daily or frequent calibrations.

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The Courier Supervisor ensures that maintenance and records for transportation vehicles are complete. The purchasing process is used for ordering garage maintenance, the garage work order is reviewed, and the vehicle checked for condition. The Controller receives all paperwork for completion of the maintenance process.

8.1.1 Microbiology General Equipment Maintenance

Optics of the Quebec colony counter and microscope are cleaned prior to each use. The stage of the microscope is also cleaned and the microscope is kept covered when not in use.

Glassware is checked for residual alkaline or acid residue utilizing bromothymol blue (BTB) on each day of media preparation.

8.2 Equipment Listing

A listing of the major equipment used for testing is available upon request. The equipment list details the unique identification number, equipment location, serial number, model number, and purchase date. The unique identification number is attached to the piece of equipment.

The laboratory performs analyses using state of the art equipment. In addition to the major equipment, the most common equipment used in the laboratory are: thermometers, balances, autopipets, water baths, hot plates, autoclaves, pH meters, conductivity meters and a variety of labware. The SOPs list the calibration and verification requirements for all laboratory equipment used in measurements.

8.3 Laboratory Water

Laboratory water is purified from central DI and RO water systems and piped to all laboratory areas. The QA Department samples the laboratory grade water and submits the samples for analysis by the lab to document the water meets the drinking water certification criteria. The Laboratory Water Logbook lists the daily conductivity checks and acceptance criteria for the laboratory water. The laboratory documents the daily, monthly and annual water quality checks. Please refer to Table 8-1 for tested parameters, monitoring frequency and control limits for each parameter (SOP/1738). Additional parameters may be tested for at the laboratory's discretion.

When additional treatment occurs in the test area, that test area records the water quality checks from the most frequently used tap. At a minimum the quality of the laboratory grade water is monitored daily by conductivity measurements. Records of the daily checks are found in the Laboratory Water Logbook. If out of specification results occur, a nonconformance action form is submitted.

TABLE 8-1

<u>Parameter</u>	<u>Monitoring Frequency</u>	<u>Control Limits</u>
Conductivity	Daily	<2 µmhos/cm @ 25°C
pH	Daily	5.5 - 7.5
Total Organic Carbon	Monthly	< 1.0 mg/L
Total Residual Chlorine	Monthly	< detection limit
Ammonia	Monthly	< 0.1 mg/L
Metals: Cd, Cr, Cu, Pb, Ni and Zn	Monthly (Required Annually)	< 0.05 mg/L
Total Metals	Monthly (Required Annually)	< 0.1 mg/L

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Heterotrophic Plate Count (Westboro only)	Monthly	< 500 CFU/mL
Water Quality Test (Biosuitability) (Westboro only)	Annually	0.8 – 3.0 ratio

8.4 Reference Materials

Reference materials include: Class 1 weights, NIST thermometers and reference standards. Timers used for DOD projects are NIST-certified. Logbooks record the reference materials used for calibration and verification. The Department Manager or QA Department maintains any certificates received with the reference materials. Laboratory personnel record in the standards logbook the reference standards date received, unique identification number, expiration date and number of containers. Each laboratory area records the unique identifier on the reference standard certificate and the Department Manager maintains the certificate. The identifier allows traceability from the certificate to the analytical data.

9 Measurement Traceability and Calibration

9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before put into service and on a continuing basis. The results are recorded in the instrument specific logbook. The laboratory has a program for the calibration and verification of its measuring and test equipment. The program includes all major equipment and minor equipment such as balances, thermometers and control standards. The Quality Systems Manual and method SOP describe the calibration records, frequency and personnel responsibilities.

9.2 Traceability of Calibration

The program of calibration and/or verification and validation of equipment is such that measurements are traceable to national standards, where available. Calibration certificates indicate the traceability to national standards, provide the results, and associated uncertainty of measurement and/or a statement of compliance with identified metrological specifications. A body that provides traceability to a national standard calibrates reference standards. The laboratory maintains a permanent file of all such certifications.

9.3 Reference Standards and Materials

Alpha Analytical has a program for calibration and verification of reference standards. The results and program are recorded in the appropriate instrument logbook. Required in-service checks between calibrations and verifications are described in method SOPs and are recorded in the appropriate instrument logbook.

Calibration standards are maintained within the area of consumption. A logbook of use is maintained and use is limited strictly to method required calibrations. Each calibration standard is identified as to test method used, date received, date opened, and expiration date. Calibrations are verified by using a second source or lot number of the calibration standard. Calibration check procedures are stated in applicable test method SOPs.

Preparation of standards must be performed using Class A glassware. Class A glassware must be used for all processes involving quantitative analyses. The only exception to this is when the method specifically requires or recommends plastic (ie. EPA 537.1).

Reference standards of measurement in the laboratory's possession (such as calibration weights or traceable thermometers) are used for calibration only and no other purpose.

Standards and reagents are uniquely identified as outlined in Westboro SOP 1745 and Mansfield SOP 1816.

9.4 Calibration General Requirements

Each calibration record is dated and labeled with method, instrument, analysis date, analyst(s) and each analyte name, concentration and response. For electronic processing systems that compute the calibration curve, the equation for the curve and the correlation coefficient are recorded in the appropriate instrument logbook. This is also true for manually prepared curves. Calibrations are tagged to the specific instrument through use of the instrument logbook and or sequence file documentation.

Initial calibration requires a standard curve that brackets the expected sample concentration. Initial calibration generally uses three to five standards depending on the equipment and

reference method specifications. Before the start of each analytical sequence, initial calibration is verified by using a continuing calibration standard. Calibration verification or continuing calibration uses the same standard as the ICAL unless method specifies otherwise. The ICV is from a second source or lot number than that used for initial calibration. The acceptance criteria for the continuing calibration standard must meet acceptance criteria before analysis of any samples. When the acceptance criteria is not within limits, review maintenance protocols and perform any necessary maintenance before starting the initial calibration sequence.

9.5 Equipment Calibration

The SOP used for the analysis defines the instrument and equipment calibration required. The following defines the general practices for equipment calibration of selected equipment.

9.5.1 Gas Chromatography/Mass Spectrometry (GC/MS)

The GC/MS is hardware tuned before performing the initial and continuing calibrations. Results must meet the peak ratio specifications of the analytical methods. For volatiles analyses, bromofluorobenzene (BFB) is used, and for semivolatiles analyses, decafluorotriphenylphosphine (DFTPP) is used for instrument tuning.

The mass spectrometer response is calibrated by analyzing a set of five or more initial calibration solutions, as appropriate, for each GC/MS method. Each solution is analyzed once, unless the method or the customer requires multiple analyses. The relative response factor for each analyte is calculated for internal standard calibration. The calibration factor for external standard calibration is calculated using the expressions found in the laboratory method SOP. Calibration is acceptable when all acceptance criteria are within method criteria.

The initial calibration is verified through the analysis of a continuing calibration standard every 12 hours. The concentration of the continuing calibration standard is dependent on the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated and must be less than the acceptance criteria stated in the method.

An acceptable continuing calibration run must have measured percent differences for the analytes within method specified ranges. If any criteria for an acceptable calibration are not met, either instrument maintenance must be performed until the continuing calibration analysis meets all criteria or a new initial calibration is established before any samples are analyzed. No samples may be analyzed unless the acceptance criteria are met for the initial and continuing calibration.

Additional quality control samples are part of the GC/MS analysis. These include internal standards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SOP.

9.5.2 Gas Chromatography (GC)

Internal standard calibration or external standard calibration is utilized for analysis by GC. The method-specified number of calibration standards is used. Each solution is analyzed once and the analyte relative response factors or calibration factors are calculated. The mean relative response factor for each analyte is then obtained by using the expression in the formula listed in the SOP. Integrated areas are utilized for these expressions.

For multiple response pesticides, PCBs or hydrocarbons the quantitation consists of the average of selected peaks or the integration of the area defined by a reference standard. The SOP details the integration criteria for each compound.

The initial calibration is verified through the analysis of a continuing calibration standard every 12 hours or 20 samples. The concentration of the continuing calibration standard is dependent on the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated. The percent drift (%d) may be calculated when calibration factors are used for quantitation.

An acceptable continuing calibration must have measured percent differences or percent drift for the analytes within method specified ranges. Should any criteria for an acceptable calibration not be met, either instrument maintenance is performed until the continuing calibration analysis meets all criteria, or a new calibration is established before any samples are analyzed. No samples may be analyzed unless the acceptance criteria are met for the initial and continuing calibration.

Other standard checks may be required for a specified reference method. Instrument performance checks specified in the reference method must be performed and be within the acceptance limits stated in the reference method. Additional quality control samples are part of the GC analysis. These include internal standards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SOP.

9.5.3 Cold Vapor Atomic Absorption Spectrophotometry (CVAA)

An initial calibration is performed daily with freshly prepared working standards that bracket the expected concentration range of the sample. A minimum of a five-point calibration curve is acquired which must have a correlation coefficient of 0.995 or better. The initial calibration is verified at the beginning of the sequence and every 10 samples. The continuing calibration is required to be within method-defined criteria, depending on the analytical method employed. Continuing calibration blanks are run at the same frequency. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within $\pm 5\%$ of the true value for EPA Method 245.1 or $\pm 10\%$ for EPA 7470A and EPA 7471B.

9.5.4 Inductively Coupled Plasma Emission Spectrophotometry-Mass Spectrometry (ICP-MS)

Initial calibration and instrument tune is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of a minimum of three standards and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within method-defined criteria. The continuing calibration is required to be within method-defined criteria. Interference check standards are performed at the beginning of the sequence. Acceptance criteria are stated in the SOP.

9.5.5 Inductively Coupled Plasma Emission Spectrophotometry (ICP)

Initial calibration is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of one standard and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within 5% of the true value for EPA Method 200.7 and 10% for SW846 6010 methods. The continuing calibration is required to be within 10% of the true value. Interference check standards are performed at the beginning and end of the sequence. Acceptance criteria are stated in the SOP.

9.5.6 Thermometers

Laboratory thermometers are checked annually for accuracy against certified, NIST traceable thermometers. Correction factors derived from the annual calibrations are applied to temperature readings where applicable. The analyst records the corrected temperature for all observations.

NIST traceable thermometers are calibrated professionally and re-certified every year. Records of thermometer calibrations are retained by the QA Department. All thermometers are tagged with the ID number, correction factor to be applied and the expiration of the calibration check.

NOTE: Electronic-based thermometers are calibrated on an annual basis. Thermometers are tagged with calibration information by the vendor, including the ID number, correction factor to be applied and the expiration of the calibration check. Certificates are kept on file in the QA Department.

Thermometers are not used past the calibration expiration date or if the thermometer is not reading properly. Replacement thermometers are calibrated and the maintenance logbook is updated when a change in the thermometer is required due to breakage, damage or expired calibration.

9.5.7 Balances

Calibration checks are performed for each day of use, for each balance. The calibration consists of a minimum of two weights, which bracket the weight to be measured. Additional calibration check procedures are performed on balances utilized in Microbiology laboratory. This additional procedure consists of a deflection test, which is performed to ensure that 100mg is detectable at a weight of 150 grams.

The balance logbook lists the acceptance criteria and performance criteria for the various balances used in the laboratory. Calibration weight measurements must meet the acceptance criteria listed on the record form.

Each balance is serviced and calibrated by a professional semi-annually. Balances are labeled with the balance number, date of service and the expiration date for the annual service check. The balance number used for any measurements requiring traceability is recorded with measurement data. Balances are not used past the expiration date or when the weight check is not within acceptable criteria. The accuracy of the calibration weights used by Alpha Analytical is verified annually by an accredited calibration service.

9.5.8 Mechanical volumetric pipettes

Delivery volumes for the mechanical volumetric pipettes (i.e. Eppendorf) are checked and recorded gravimetrically before use and on a quarterly basis. The verification is performed at the volume of use or bracketing the volume range of use. The check must be within the criteria stated in the laboratory logbook. Pipettes failing acceptance criteria are tagged and removed from service until repaired and the criteria are met, or discarded and replaced. Automatic pipettes are labeled with a unique ID number, volumes verified and expiration date.

9.5.9 Ion Chromatography

The ion chromatograph calibration is by analyzing a set of five or more initial calibration solutions, with concentrations of analytes appropriate to the analytical methods. The concentrations must bracket the expected concentration range of the samples analyzed. Procedures for verifying the calibration curve are method specific. The initial calibration is performed at the start of each day. The calibration curve is verified at least after every 20 samples.

9.5.10 pH Meters

pH meters are calibrated prior to use for each day of use. The meter is calibrated following the procedure for pH analysis. The records of the calibration are recorded in an instrument logbook or in the raw data for the analysis being performed. At least two buffer solutions that bracket the measurement range for the analysis are used for calibration. A second source check standard is used at the end of a run to verify meter stability. Buffer solutions used for calibration are NIST certified. Standard buffer solutions are not retained or re-used. The lot number of the buffer solutions is recorded in the data record to ensure traceability of the measurement to NIST.

9.5.11 Conductivity Meters

Three calibration standards of potassium chloride (KCL) solutions are analyzed annually on each instrument range. The calibration standards are used to verify instrument performance. The acceptance criteria are defined in the test SOP. If unacceptable performance is found, the cell is cleaned and rechecked. The cell is not used until satisfactory performance is achieved.

A single KCL standard solution is used to calibrate each range of the instrument. A second standard is used to check the calibration each day the meter is used. The check standard is near the measurement range for the samples to be analyzed. The acceptance criterion is $\pm 20\%$ of the true value. The meter is labeled with expiration date for the annual calibration. A check standard that is NIST traceable is used to allow traceability. The check standard is performed at the end of the analysis run or at least after every 20 samples.

9.5.12 Autoclave

The date, contents, sterilization time and temperature, total cycle time and analyst's initials are recorded each time the autoclave is used. Autoclave cycles must be completed within 45 minutes when a 15 minute sterilization time is used. Autoclave timing mechanisms are checked quarterly with a stopwatch to verify timing controls. A maximum temperature thermometer is used with each cycle to ensure the sterilization temperature is reached.

Spore strips or ampoules are used weekly to confirm sterilization. BTSure ampoules are utilized as follows: An indicator ampoule is placed in most challenging area of sterilizer. Load is processed according to standard operating instructions. Remove from sterilizer and allow to cool for a minimum of 10 minutes. (Chemical indicator on label changes from green to black when processed.) Place the autoclaved indicator and un-autoclaved control indicator in an upright position in the plastic crusher provided. Gently squeeze crusher to break glass ampoules. Incubate both indicators at 55-60°C for 24 hours. Examine appearance for color change. Yellow color indicates bacterial growth. No color change indicates adequate sterilization.

Calibration is conducted and certified annually by an outside service provider and recorded. Certificates are kept on file. Routine maintenance includes cleaning the autoclave seal to ensure freedom of caramelized media and cleaning drain screens to remove any debris buildup. For the efficient operation of the unit, overcrowding is avoided.

10 Test Methods and Standard Operating Procedures

10.1 Methods Documentation

Analysis consists of setting up proper instrument operating conditions, executing acceptable calibrations, monitoring instrument performance tests, analyzing prepared samples, and collecting data from the analyses. The test method SOP describes the instrumental analysis procedures, quality control frequencies and acceptance criteria. EPA accepted methods, national recognized methods or customer-specified methods are the basis for performance criteria, instrument conditions and the steps of the procedure. The method performance requirements of the published methods are followed unless otherwise specified by the customer.

The reference methods define the instrument operating conditions. In many of the reference methods, a range or general guidance on the operating conditions is defined. Documented modifications to the operating conditions clarify the reference methods or improve the quality of the results. In all cases where the method modifications are adopted, the performance criteria from the reference method must be met. Modifications to the operating conditions are stated in the SOP. Changes in the operating conditions made at the time of the analysis are documented in the appropriate laboratory or sequence log. A revision to the SOP takes place, when a day to day change in the operating condition improves performance for all matrices.

The laboratory SOPs include the operation of measurement equipment. The SOPs contain the following information, as applicable:

- The equipment used in the procedure, including equipment type
- Equipment calibration and process for obtaining the measurement from the calibration
- The step by step instructions to perform the measurement
- Acceptance criteria for the calibrations
- Corrective action for failed acceptance criteria, including assessment of previous calibration results
- The basis used for the calibration standards such as traceability to NIST or EPA or demonstration of comparability
- Frequency at which the equipment will be calibrated, adjusted and checked
- The records maintained to document the calibration and use of measurement equipment
- The calibration status for the equipment
- The environmental conditions necessary before measurement equipment may be calibrated or used for measurement
- Allowed adjustments to measurement equipment, including software, which will not invalidate the laboratory analysis
- Maintenance of the equipment and record keeping to track performance before and after maintenance is completed
- Define the standards, reagents and sample handling, interferences, preservation, and storage in order to assure measurement performance

10.2 Standard Operating Procedures (SOPs)

Alpha Analytical maintains SOPs that accurately reflect all phases of current laboratory activities such as assessing data integrity, nonconformance actions, handling customer complaints, sample receipt and storage, purchasing of all materials, and all test methods. These documents include equipment manuals provided by the manufacturer, internally written documents, and published methods with documented changes or modifications.

Copies of all SOPs are accessible to all personnel in electronic form through Qualtrax. Each SOP clearly indicates the published date of the document and the revision number.

10.3 Laboratory Method Manual (s)

All SOPs are posted as secure documents in the Alpha Qualtrax system. Directories are available for each laboratory area and administrative area in appropriate subfolders. Each SOP includes or references where applicable:

- 1) identification of the test method and where applicable;
- 2) applicable matrix or matrices;
- 3) method detection limit;
- 4) scope and application;
- 5) summary of method;
- 6) definitions;
- 7) interferences;
- 8) safety;
- 9) equipment and supplies
- 10) reagents and standards
- 11) sample collection, preservation, shipment and storage;
- 12) quality control;
- 13) calibration and standardization;
- 14) procedure;
- 15) calculations;
- 16) method performance;
- 17) pollution prevention;
- 18) data assessment and acceptance criteria for quality control measurements;
- 19) corrective actions for out-of-control data;
- 20) contingencies for handling out-of-control or unacceptable data;
- 21) waste management;
- 22) references; and
- 23) any tables, diagrams, flowcharts and validation data.

In cases where modifications to the published method have been made by the laboratory or where the referenced method is ambiguous or provides insufficient detail, these changes or clarifications are clearly described in the SOP.

10.4 Test Methods

The laboratory uses appropriate methods and procedures for all tests and related activities within its responsibility (including sampling, handling, transport and storage, preparation of items, estimation of uncertainty of measurement and analysis of test data). The method and procedures are consistent with the accuracy required, and with any standard specification relevant to the calibrations or tests concerned. When the use of mandated methods for a sample matrix is required, only those methods are used. Where methods are employed that are not required, the methods are fully documented and validated and are available to the customer and other recipients of the relevant reports.

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The customer requests the reference method for sample analysis usually based on the regulatory program. The customer services staff may assist the customer with method selection when the customer specifies the regulatory program, but is unsure of the correct method required. The Laboratory Technical Manager or Quality Assurance Officer recommends methods for non-regulatory programs. In all cases, recommendation of methods is based on customer-defined method performance criteria. Customer services may recommend a procedure that meets the customer method performance criteria.

10.5 Method Validation/Initial Demonstration of Method Performance

Before acceptance and use of any method, satisfactory initial demonstration of method performance is required. In all cases, appropriate forms are completed and retained by the laboratory and made available upon request. All associated supporting data necessary to reproduce the analytical results is retained. Initial demonstration of method performance is completed each time there is a significant change in instrument type, personnel or method.

10.6 Sample Aliquots

The aliquot sampling process from a submitted sample is part of a test method. The laboratory uses documented and appropriate procedures and techniques to obtain representative sub-samples. Sample aliquots removed for analysis are homogenized and representative portions removed from the sample container. Personnel record observations made during aliquot sampling in the test method logbooks.

10.7 Data Verification

Calculations and data transfers are subject to appropriate checks which is a 3 tier approach. The initial analyst verifies all of his work, a secondary review of 100% of the initial is conducted by an independent qualified analyst. A Customer Services representative reviews data for project and method performance requirements where applicable. A QA representative reviews data for project and method performance requirements when requested by a Customer. Final report review is performed by an authorized company signatory.

For drinking water suppliers, every effort is made to notify the Customer within 24-hours of obtaining valid data of any results that exceed any established maximum contaminant level or reportable concentration. Analyst or Department Supervisor notifies the Customer Services Department of the sample number(s), Customer name, analysis and sample results (preliminary or confirmed). The Customer Services Department notifies the customer.

The laboratory Report Generation and Approval SOP describes the practices to ensure that the reported data is free of transcription errors and calculation errors. Manually entered data into the LIMS is dual entered and checked by the LIMS to minimize transcription errors. The laboratory test method SOP describes the quality control measures used to assure method performance before reporting data.

10.8 Labeling of Standards and Reagents

The purchase, receipt and storage of consumable materials used for the technical operations of the laboratory include the following:

- a) The laboratory retains records of manufacturer's statement of purity, of the origin, purity and traceability of all chemical and physical standards.
- b) Original reagent containers are labeled with the date opened and the expiration date.
- c) Detailed records are maintained on reagent and standards preparation. These records indicate traceability to purchased stocks or neat compounds and include the date of preparation and preparer's initials.

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- d) Where calibrations do not include the generation of a calibration curve, records show the calibration date and type of calibration standard used.
- e) All prepared reagents and standards are uniquely identified and the contents are clearly identified with preparation date, concentration and preparer's initials. These procedures are outlined in Westboro SOP/1745 and Mansfield SOP/1816.

10.9 Computers and Electronic Data Related Requirements

Computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data. The laboratory ensures that computer software and firmware is documented and adequate. The goals of the software development methodology, existing system validations and the change control system are to ensure that:

- the software systems perform the required functions accurately,
- the users understand how to use the system, and
- auditors can assure themselves of the validity of the analytical data.

The computer systems used at Alpha Analytical are purchased. A coordinated effort is made with the supplier to assure the computer operations meet the laboratory requirements for data integrity. Alpha Analytical has a formal validation program of its computer systems. The validation program is a comprehensive program to ensure data transmitted, reported or manipulated by electronic means is correct and free of errors. The validation and verification approach is separated into three areas.

1. New software is developed and validated using test data. Records of validation include the test data report, date and initials. Where formulas are part of the program, documentation includes manual verification of the final calculated values. New software includes the development of macros for spreadsheets and other tools using commercial software packages.
2. Reasons for changes to software are identified through flaws in existing documentation or the need to improve system processes and are documented on the Nonconformance Report. Final implementation of the change is documented on the nonconformance action form. The tracking and timelines of making the change is readily available. This process also provides the complete documentation of all software and electronic data reporting problems. All nonconformance identified with electronic data process result in corrective action that are reported to management before or at the bi-weekly executive meeting. Customers will be notified prior to any changes to software or hardware that will adversely affect customer electronic data. This information is provided by IT department to QA and Project Managers to be communicated to appropriate customers.

Verification of system integrity is through routine maintenance, protection from unauthorized access and electronic verification programs. Routine maintenance including system backups are performed on a scheduled basis. The backup process and password and access protections are defined in the Computer System Backup Control SOP/1562 and Computer Security SOP/1563. Electronic verification may be used to assure the commercially purchased software is performing at its original specifications. This includes virus checking of all network operation at least once per week. Documentation of all verification and maintenance operations is retained.

11 Sample Handling, Sample Acceptance Policy and Sample Receipt

The Sample Login and Custody procedures define the process for sample management from sample receipt through analysis and to disposal. These procedures detail the process for sample receipt, records and storage pending analysis.

Customers or Alpha's Couriers deliver samples to the laboratory during normal business hours. Sample receiving occurs in the sample management area.

Customer service personnel place bottle orders. The orders are filled following the bottle order instruction form. Blanks are prepared as needed with minimal storage. All glass containers are packed to minimize or prevent breakage. The containers are placed in plastic coolers or shipping packages and Chain-of Custody forms, seals (if requested) and labels enclosed. The bottle order is shipped by third party, picked up by the customer or customer representative or delivered by Alpha courier to the customer.

11.1 Sampling Supplies

11.1.1 Sample Containers

Sample containers provided by Alpha Analytical include labels, preservatives and a blank chain of custody form. Preservatives and containers are lot controlled and verified as appropriate for the indicated type of analysis.

Each lot of containers used for the collection of samples for microbiological analysis is checked for sterility prior to distribution. Sterility checks are performed by Microbiology staff and results recorded in Microbiology Sample Container Sterility Log.

Sample Containers for collecting Air samples (TO-15) are cleaned and prepared according to SOP 2190 "Cleaning and Preparation Procedures for Equipment used to collect Air sample for analysis of Volatile Organic Compounds".

11.1.2 Chain of Custody

Chain of custody forms must accompany all samples received by Alpha personnel. The chain of custody form indicates the sample origin and arrival at the laboratory and identifies the analyses requested.

11.1.3 Reagent Water

Alpha Analytical supplies laboratory pure water for field QC blanks. Water used for volatile organics must be free of volatile compounds below the method detection limit. The quality of the laboratory water is monitored for conductivity once per day. Additional water quality criteria may be monitored based on customer specific requests. The water quality in the laboratory is monitored for chemical parameters as required by the EPA certification manual for drinking water (Water Quality Monitoring SOP/1738).

11.2 Sample Tracking

Alpha Analytical uses an internal chain-of-custody in LIMs for sample tracking control purposes. When requested or required by regulation a legal custody program is used in addition to the routine laboratory practices. Legal custody practices must be arranged at the time of contractual commitment.

For legal custody the process must include complete and continuous records of the physical possession, storage, and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For legal custody a sample is in someone's custody if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;
3. It is in one's physical possession and then locked up so that no one can tamper with it;
4. It is kept in a secured area, restricted to authorized personnel only.

The routine sample handling and tracking process includes unique identification of all sample containers, initials of the person removing the sample from the sample management area and documentation of the date of sample removal for disposal.

Samples are assigned a unique identification number from the LIMS program. Each sample container label includes a unique identifier for the container. The person handling the sample is recorded along with the unique identifier in the container tracking records in LIMS.

ALPHA ANALYTICAL utilizes a custom designed Laboratory Information Management System (LIMS) to uniquely identify and track samples and analytical data throughout the facility. The LIMS log-in, is initiated by the Sample Custodian when the following information is entered into the computer:

- Quote number (unique to the project if requested)
- Project name or description
- Analyses requested (per matrices received)
- Sample number (unique to this sample)
- Sample descriptions (customer ID, including number of received containers)
- Date received
- Date(s) and time(s) collected
- Date analytical results are due

11.2.1 Chain of Custody

Chain of custody forms must accompany all samples received by Alpha personnel. The chain of custody form indicates the sample origin and arrival at the laboratory and identifies the analyses requested.

- Customer's name and address
- Notation of special handling instructions
- Additional comments or instruction for the laboratory
- Purchase order number(s), if applicable

Alpha Job Numbers (Process for assigning numbers)

Alpha Job Numbers are unique #'s automatically designated by our LIMS computer system for every individual customer project.

There are 3 parts to this number:

- All numbers start with the letter "L"
- The next two numbers are the last two numbers of the current year.
- The last five numbers are pulled sequentially by the LIMS as each Login personnel requests a new number for a job.

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For example.... L0904165 ---- Year 2009 and 4,165th job to be logged in this year.

The Alpha Job Number then may contain as many extensions as there are individual samples in a job. L0904165-01 is the first sample, L0904165-02 is the second and so on. Each sample may contain as many as 26 containers as the containers are designated with the letters of the Alphabet, and each container receives its own bar-coded label. For example, L0904165-09A is the first container of the 9th sample listed on a customer's Chain of Custody.

Each container is labeled with a unique identifier, a label with a unique identifier number is placed on each sample container. Once labeled, the sample containers are placed in the appropriate storage area.

11.3 Sample Acceptance Policy

The sample management personnel check for proper sample labeling, preservation and handling at the time of arrival at the laboratory. The customer and customer services manager specifies the proper sample preservation, containers, cooling and other criteria on the project review form and in the LIMS. Sample management staff record all observations and immediately notify customer services of any discrepancies or questions arising during sample receipt.

It is possible for samples or sample containers to be lost, damaged, or determined to be unsuitable, for whatever reason, after initial receipt at Alpha Analytical. The problem is brought to the attention of a customer services manager who reports it to the customer. Plans for disposition of the affected samples or container are agreed upon with the customer, carried out, and recorded in the project records. Sample hold times and preservations are listed on the Alpha website (www.alphalab.com) under Support Services "Sampling Reference Guide".

11.4 Sample Receipt Protocols

The sample management staff receives all samples. A unique job number is assigned to each shipment of samples received from a customer. The in-house records for the incoming job, including the internal Chain-of-Custody, are initiated with a Sample Delivery Group (SDG) form. The customer, and Alpha courier and/or the sample management personnel sign the sample custody form at the time of receipt at the laboratory. Samples received via overnight courier are signed on the bill of lading. The bill of lading, SDG form and the sample custody form are completed for external courier delivered samples.

The sample management staff examines the shipping containers, their contents, and accompanying customer documentation. Information about the sample identification, the location, date and time of collection, collector's name, preservation type, sample type, presence and condition of custody seals, the state of preservation of the samples and other required information is noted on the SDG form. Any discrepancies in documentation or problems with sample condition such as appropriate sample containers, thermal preservation variation, holding times and adequate sample volumes are noted and brought to the attention of the customer via the nonconformance action form. The login staff or project manager contacts the client via email or or by phone. The Customer Services Manager provides clarification or further instruction to the sample management staff on the processing of the samples that are incomplete or missing required information.

The sample management staff logs the samples in the LIMs and a durable label for each container is printed. The custodian attaches each label to the appropriate sample container. The following information is recorded for tracking internal custody: laboratory sample ID, customer sample ID, sample matrix and storage location. Sample receipt and log-in specifically requires: date and time of laboratory receipt of sample(s); sample collection date; unique laboratory ID code; field ID code supplied by sample submitter; requested analyses; signature or initials of data logger; comments from inspection for sample acceptance or rejection and in some cases, sample bottle codes.

11.5 Storage Conditions

Alpha Analytical stores samples under proper environmental conditions to ensure their integrity and security. Samples are stored at temperatures that meet specifications of the methodology, regulatory agencies and customer directives. Refrigerators are monitored and controlled to be within $4 \pm 2^{\circ}\text{C}$. Chemical, temperature, holding times and container storage requirements are listed in the LIMS project database.

Customer Quality Assurance Project Plans may list preservation requirements differing from the laboratory. The sample management staff reviews project information for projects specific handling. Addition of chemical preservative to sample containers normally is done in the field at the time of sampling. Chemical preservation and temperature preservation checks at the time of receipt are recorded except for volatile organic compounds, bacteria, sulfite, and dissolved oxygen preservation. Any differences from laboratory or customer specific requirements are recorded on nonconformance action forms and contact made with the customer by the Customer Services Manager or designee.

Sample storage facilities are located within the sample management area, walk-in custody refrigerator or in designated sample storage areas within the analytical departments. Internal chain-of-custody procedures and documentation pertaining to sample possession, removal from storage, and transfer are outlined in the sample custody procedure. Samples are returned to the sample storage area after the sample portion is removed for analysis. Extracts and digestates are tracked and follow the same internal custody operation. Extracts and digestates are removed to the waste disposal area after analysis for proper disposal.

Sample storage precautions are used to ensure that cross contamination does not occur during sample storage. Refrigerator storage blanks are monitored bi-weekly for volatile compounds. The storage blank information allows the assessment of potential cross contamination in the sample storage refrigerator.

Temperatures of cold storage areas are recorded continuously in the data logger system. Corrective action is done as necessary when temperatures are not within the control criteria. In both the Westboro and Mansfield facilities, Automated Data loggers are linked to thermocouples in custody refrigerators and freezers in the Sample Storage areas as well as department standards/storage refrigerators and freezers. The Data logger is calibrated and certified by an outside vendor annually and on a quarterly basis for DOD standards/storage refrigerators and freezers. If there is a catastrophic failure of custody refrigerators, a record of all samples affected and customers associated with such samples are notified of any samples affected by the failure. Refrigerators and/or freezers not connected to the Data Logger system have temperatures measured with NIST traceable thermometers. Temperature records indicate the thermometer or sensor (Data logger) used for obtaining the measurement.

11.6 Sample Disposal

Samples are held for 21 calendar days after the report is released to the customer. Upon written customer request samples may be held longer in an uncontrolled area. Requests for controlled

sample storage must be arranged at the time of contractual commitment. Air canister samples are held for 3 days after the report is released to the customer.

An authorized waste carrier is contracted to pick up waste as needed and dispose of it, in accordance with all regulatory requirements. Post-analysis disposition of samples is dependent upon project specific requests. Remaining sample material may be returned to the customer, safely discarded, or archived for a specific time prior to disposal. The waste disposal SOP 1797 defines the specific requirements for sample disposal and other waste disposal operations.

The sample management staff are responsible for the archival and disposal of raw samples, extracts and digestates. Raw and prepared samples may not be archived or disposed until all of the designated analyses are complete and resultant analytical data is sent to customers. Samples in storage are retained a minimum of 21 calendar days after reporting the results to the customer. Any samples requiring more than 21 calendar days are archived. Air canister samples requiring storage more than 3 business days require prior approval.

When a customer has requested the return of samples, the sample management staff prepares and ships the samples according to the same custody procedures in which the samples were received and following any customer specified requirements. Protection of the samples during delivery is ensured by the implementation of special packaging procedures. Packages are delivered by a commercial carrier whose procedures for protecting the samples are not within the control of this laboratory. Customers are informed that a commercial carrier will deliver their samples if required.

12 Records

Alpha Analytical has a record system that produces accurate records, which document all laboratory activities. The laboratory retains records of all original observations, calculations and derived data, calibration records and a copy of the test for ten years minimum. The system retains records longer than the minimum upon the request of authorized customers, agencies or another regulator. Note: Ohio VAP requires notification before disposal of any VAP records.

12.1 Record Keeping System and Design

The record keeping system allows reconstruction of laboratory processes that produced the analytical data of the sample.

- a) The records include the names of personnel involved in sampling, preparation, calibration or testing.
- b) Information relating to laboratory facilities equipment, analytical methods, and activities such as sample receipt, preparation, or data verification are documented.
- c) The record keeping system provides retrieval of working files and archived records for inspection and verification purposes.
- d) Documentation entries are signed or initialed by responsible staff.
- e) Generated data requiring operator logging on appropriate logsheets or logbooks are recorded directly and legibly in permanent ink
- f) Entries in records are not obliterated by any method. Corrections to errors are made by one line marked through the error. The person making the correction signs and dates the correction.
- g) Data entry is minimized by electronic data transfer and ensuring the number of manual data transcriptions is reduced.

12.2 Records Management and Storage

1. Records including calibration and test equipment, certificates and reports are safely stored, held secure and in confidence to the customer.
2. The laboratory maintains hardware and software necessary for reconstruction of data.
3. Records that are stored or generated by computers have hard copy or write-protected backup copies.
4. Alpha Analytical has established a record management system, for control of hard copy laboratory notebooks.
5. Access to archived information is carefully controlled. These records are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources. Any access to the archive is documented in the Data Archive Access Logbook which is used strictly by the QA Department.
6. In the event that Alpha Analytical transfers ownership or goes out of business, there is a plan to ensure that the records are maintained or

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transferred according to the customer's instructions. A plan will be developed to maintain continuity of our record keeping systems as requested and/or required by both state and federal laws.

Alpha Analytical retains all original hard copy or electronic raw data for calibrations, samples, and quality control measures for ten years, including:

1. Analysts work sheets and data output records,
2. Reference to the specific method,
3. Calculation steps including definition of symbols to reduce observations to a reportable value,
4. Copies of all final reports
5. Archived SOPs,
6. Correspondence relating to laboratory activities for a specific project,
7. All nonconformance action reports, audits and audit responses,
8. Proficiency test results and raw data,
9. Data review and cross checking.

The basic information to tie together analysis and peripherals such as strip charts, printouts, computer files, analytical notebooks and run logs for Alpha Analytical includes:

1. Unique ID code for each Laboratory sample or QC sample;
2. Date of analysis;
3. Instrument identification and operating conditions;
4. SOP reference and version;
5. Calculations;
6. Analyst or operator's initials/signature.

In addition, Alpha Analytical maintains records of:

1. Personnel qualifications, experience and training
2. Initial and continuing demonstration of proficiency for each analyst
3. A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory records. Use of electronic signatures has been approved by regulatory agencies.

12.3 Laboratory Sample Tracking

A record of all procedures to which a sample is subjected while in the possession of the laboratory is maintained. These include but are not limited to records pertaining to:

- a) Sample preservation including appropriate sample container and compliance with holding time requirement; If the time of the sample collection is not provided, the laboratory must assume the most conservative time of day (i.e., earliest).
- b) Sample identification, receipt, acceptance or rejection and log-in;

- c) Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records; this includes inter-laboratory transfers of samples, extracts and digestates.
- d) Sample preparation including cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- e) Sample analysis;
- f) Standard and reagent origin, receipt, preparation, and use;
- g) Equipment receipt, use, specification, operating conditions and preventative maintenance;
- h) Calibration criteria, frequency and acceptance criteria;
- i) Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- j) Method performance criteria including expected quality control requirements;
- k) Quality control protocols and assessment;
- l) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;
- m) Automated sample handling systems;
- n) Records storage and retention; and
- o) Disposal of hazardous samples including the date of sample or sub-sample disposal and the name of the responsible person.
- p) The COC records account for all time periods associated with the samples.
- q) The COC records include signatures of all individuals who had access to individual samples. Signatures (written or electronic) of all personnel who physically handle the samples. Time of day and calendar date of each transfer or handling procedure.
- r) Common carrier documents.

13 Laboratory Report Format and Contents

The Process Planning and Control Procedure details the recording and reporting of data as required by the customer and in accordance with relevant environmental regulations.

Customers specify the report delivery and deliverables required for the work submitted. Report delivery includes standard turnaround and rush turnaround. Customers specify the delivery address or multiple addresses and method of delivery such as U.S. Mail, facsimile or electronic at the start of the project. Alpha Analytical provides data deliverables in hardcopy or electronic format. At the start of any project, the electronic deliverable formats required must be received before sample arrival. Affidavits are required with each report or series of reports generated for a particular project for Ohio VAP reports.

Reporting packages are available for routine regulatory reporting requirements. Regulatory reporting packages include only the information requested by the regulatory agency. In addition to regulatory report packages, Alpha Analytical prepares a standard report format. The standard report format includes:

1. Title: "Certification of Analysis"
2. Name and address of the laboratory
3. Laboratory Job Number, page number and total number of pages included in the report.
4. Name and address of the customer
5. Alpha sample number, Customer identification, Sample location
6. Samples identified that do not meet the sample acceptance requirements for project.
7. Date of sample receipt, sample collection, preparation or extraction date and time (if applicable), analysis date and time, report date and analyst
8. Identification of data reported by subcontractors
9. Test name and reference method number
10. Delivery method and sampling procedures when collected by lab personnel
11. Deviations or modifications that affect data quality and/or data integrity. These deviations or modifications are included in narrative statements and/or data merger files.
12. Statement that results relate only to the sample tested
13. Statement that report must be copied in full unless the laboratory provides written permission for partial copies
14. Glossary, References and limits of liability
15. Units of measure and reporting detection limit
16. Quality control data for: % Recovery surrogates, % Recovery of LCS, % RPD of LCSD, Blank analysis, % Recovery Matrix Spike, %RPD of Laboratory Duplicates, as applicable
17. Signature, title and date of report

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18. A "Certificate/Approval Program Summary" page is included at the end of the report that identifies analytes for which Alpha Analytical holds certification and for those analytes reported that it does not. This summary also includes the certification numbers for either NELAP certified states, State certifications (e.g. Massachusetts laboratory certification identification number)..
19. Alpha Analytical does not accept samples from private residents for drinking water analysis and therefore maximum contaminant levels are not necessary. If Alpha were to change its policy and report drinking water samples, MCLs would be included with the report.

Results transmitted by facsimile or other electronic means include a statement of confidentiality and return of the materials at the laboratory's expense.

The laboratory notifies the customer in writing of any circumstance that causes doubt on the validity of the results. The amended or modified report lists the change, reason for the change, affected page numbers, date of the amendment and authorized signature. The customer will be notified prior to changes in LIMs software or hardware configurations that will adversely affect customer electronic data.

13.1 Data Qualifiers

The following data qualifiers are used in conjunction with analytical results depending on the definition, state or regulatory program and report type.

Note: "J" Estimated value: Upon customer request, the Target analyte concentration can be reported below the quantitation limit (RL), but above the Method Detection Limit (DL) with a "J" qualifier as long as there is a LOD study on file. (See section 5.11)

<u>Data Qualifier</u>	<u>Qualifier Information</u>	<u>Regulatory Requirement</u>
A	Spectra identified as "Aldol Condensation Product".	CT RCP, NC
B	<p>The analyte was detected above the reporting limit in the associated method blank. Flag only applies to associated field samples that have detectable concentrations of the analyte at <5x the concentration found in the blank. For MCP-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than 10x the concentration found in the blank. For NJ-Air-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte above the reporting limit. For NJ-related projects (excluding Air), flag only applies to associated field samples that have detectable concentrations of the analyte, which was detected above the reporting limit in the associated method blank or above five times the reporting limit for common lab contaminants (Phthalates, Acetone, Methylene Chloride, 2-Butanone) For DOD related projects, flag applies to detectable concentration of target analyte in the blank that exceeds ½ the LOG or is greater than 1/10 the concentration in the field sample</p>	EPA Functional Guidelines 'MassDEP MCP, CT RCP, NJ-TO15/LL-TO15; NJ Tech Guidance 2014, DOD QSM 5.4
C	Co-elution: target analyte co-elutes with a known lab standard (i.e. surrogates, internal standards, etc.) for co-extracted analyses.	
D	Concentration of analyte was quantified from diluted analysis. Flag only applies to field samples that have detectable concentrations of the analyte.	NJ-TO15/LL-TO15 - Air only EPA Functional Guidelines; EPA Region 2,5
DL	Same was re-analyzed at a dilution. Qualifier applied to sample number.	

E		Concentration of analyte exceeds the range of the calibration curve and/or linear range of the instrument.	EPA Region 2,5 CT RCP, NJ-TO15/LL-TO15
G		The concentration may be biased high due to matrix interferences (i.e. co-elution) with non-target compound(s). The result should be considered estimated.	In-house/Forensics.
H		The analysis of pH was performed beyond the regulatory-required holding time of 15 minutes from the time of sample collection.	THE NELAC INSTITUTE (TNI) STANDARDS
I		The lower value for the two columns has been reported due to obvious interference.	In-house.
J		Estimated value. This represents an estimated concentration for Tentatively Identified Compounds (TICs).	CT RCP (for TICs),
JN (NJ)		Presumptive evidence of compound. This represents an estimated concentration for Tentatively Identified Compounds (TICs), where the identification is based on a mass spectral library search.	EPA Functional Guidelines 'NJ-TO15-LL
ND	DU-J	Not detected at the method detection limit (MDL) for the sample, or estimated detection limit (EDL) for same-related analysis	In-house
P	All DU	The RPD between the results for the two columns exceeds the method-specified criteria.	MassDEP MCP, CT RCP
Q	All DU	The quality control sample exceeds the associated acceptance criteria. Note: This flag is not applicable for matrix spike recoveries when the sample concentration is greater than 4x the spike added or for batch duplicate RPD when the sample concentrations are less than 5x the RL. (Metals only.)	
R	All DU	Analytical results are from sample re-analysis	Customer-specific

RE	All DU	Analytical results are from sample re-extraction.	Customer-specific
S		Analytical results are from modified screening analysis	

13.2 Compound Summation for Organic Analyses

In order to be compliant with regulations from certain states, Alpha Analytical has created the following Summation Rules to cover reporting "Total Analytes". The following are an example of several compounds that can be reported as "Totals":

Volatiles:	
1,3-Dichloropropene, Total	cis + trans isomers
Xylenes, Total	m/p + o isomers
1,2-Dichloroethene, Total	cis + trans isomers
Trihalomethanes, Total	Chloroform + Bromoform +
	Dibromochloromethane +
	Dichlorobromomethane
PCBs:	
PCBs, Total	Sum of reportable Aroclors
	(all Aroclors reported for the project)

The following are the summation rules that the LIMs uses to calculate the Total values:

Summation Rules:	
H + H = H	Key:
H + J = J	H = Hit (above RL)
J + J = J	J = J-flagged value
H + ND = H	ND = U-flagged value
J + ND = J	
ND + ND = ND	

The ND values are considered "0" during the calculations.
 The "E" flagged values (over the calibration) are ignored and not utilized during the calculations.
 Any "N" flagged values (do not report) are ignored and not utilized during the calculations.
 For dual-column analysis, the Total is reported as part of column "A" data, unless all individuals are reported from "B" column.

For analytical group summations, the Total is reported based on the associated "Reporting List".
For example, if only 7 Aroclors are requested, then the Total is based on 7 Aroclors, not 9.

The RL and MDL for Totals will always be the lowest of the individual compounds used in the summation.

For each Total summation, two values are calculated: TOTALH (calculated from all associated hits above the R L– used in DU reporting formats) and TOTALJ (calculated from all associated hits and J flagged values – used in DJQL reporting formats). Total concentrations are calculated for all samples and QC samples (however, recoveries are not calculated since they are only calculated for the compounds spiked)

If a Total summation is requested, the individual compounds must also be reported.

14 Outside Support Services and Supplies

When Alpha Analytical purchases outside services and supplies in support of tests, the laboratory uses only those outside services and supplies that are of adequate quality to maintain confidence in the tests. Differences between Request/Tender and Contracts must be resolved before work commences.

The Purchasing SOP/1726 describes approval and monitoring of all suppliers and subcontractors used by the laboratory. Where no independent assurance of the quality of outside support services or supplies is available, the laboratory ensures that purchased equipment, materials, and services comply with specifications by evaluating method performance before routine use.

The laboratory checks shipments upon receipt as complying with purchase specifications. The use of purchased equipment and consumables is only after the evaluation and compliance to the specifications is complete. The Purchasing SOP/1726 describes the details for receipt and inspection of purchased product.

The Purchasing SOP describes the process for raising, review and placement of purchase orders. It is company policy to purchase from third party certified suppliers and subcontractors wherever possible. Purchases must be from suppliers approved by the Laboratory. Laboratory or sampling subcontractors specified by the customer are noted as "Trial" on the purchase order. This identifies the subcontractor as a non-approved subcontractor. All DoD work that is subcontracted must comply with Alpha's management system and must comply with the QSM standard and is subject to DoD customer approval.

The laboratory maintains list of approved vendors (Form 18302) and subcontractors from whom it obtains support services or supplies required for tests.

14.1 Subcontracting Analytical Samples

Customers are advised, verbally and/or in writing, if any analyses will be subcontracted to another laboratory. Any testing covered under the NELAC Institute (TNI) Standards that requires subcontracting, will be subcontracted to another THE NELAC Institute (TNI) Standard accredited laboratory for the tests to be performed. The laboratory approves testing and sampling subcontractors by review of current state, national or other external parties' certifications or approvals. This document must indicate current approval for the subcontracted work. Any sample(s) needing special reports (*i.e.*, MCL exceedance) will be identified on the chain of custody when the laboratory subcontracts with another laboratory. Subcontractor Laboratory Certifications are located in Qualtrax under Customer Services folder

The Sample Receipt and Login Procedure describes the process for sample handling when subcontracting samples. Customer notification of subcontracted work is in writing or verbally before releasing samples to the subcontractor.

The review of subcontractor documents for completeness and meeting the specifications defined for the project follows the laboratory process for reporting and verification of process data. The Reporting Department Designee is responsible for receiving the order reviews the information supplied by the subcontractor instead of the Department Supervisor.

15 Customer Relations

15.1 Customer Service

The majority of the customer services occur from personnel in the administration, sample receiving and sampling areas. Customer service involves inquiries into services offered, technical consulting, placing orders, and receiving orders, providing updates on the status of orders and completing orders. Personnel interacting with customers must document and review customer specific project requirements. Call Tracker is used to document communications with customers (SOP/1723). Personnel must document customer interactions following the appropriate laboratory procedures. Each person must communicate deviations, modifications and customer requests following the laboratory defined procedures.

15.2 Project Management

During staff meetings the laboratory management reviews requests for new work. The Operations Director and/or Laboratory Technical Manager address all capacity and capability issues. Where conflicts in workload arise, customer notification is immediate. The Project Communication Form (PCF) contains the documentation of all project information. Cooperation between laboratory and customer services staff allows direct communication and scheduling. Management arranges complex scheduling and coordination between departmental areas. Documentation of approval for waivers from the DoD QSM requirements must be documented on a project specific waiver. This documentation needs to be in writing and readily available for review.

15.3 Complaint Processing

The laboratory staff documents all customers or other parties' complaints or concerns regarding the data quality or laboratory operations. The Nonconformance Report records complaints, correcting the concern, and resolving the concern with the customer or other party. The process uses the same form and process as the nonconformance action process. Where repetitive corrective actions indicate a problem, an audit of the area, Customer Inquiry and Complaint SOP/1722 is immediate to ensure the corrective action has effectively solved the concern.

16 Appendix A – Definitions/References

The following definitions are from Section 3.0 of the 2016 TNI Standard unless otherwise cited. The laboratory adopts these definitions for all work performed in the laboratory.

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accreditation: the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

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.

Aliquot: A discrete, measured, representative portion of a sample taken for analysis. (EPA QAD glossary)

Analyst: The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analyte:

A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed. (TNI)

The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA Glossary)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation).

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Batch: Environmental samples, which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample

in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates), which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

Blanks include:

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Field Blank: blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

Instrument Blank: a clean sample (e.g. distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Reagent Blank: (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Calibration: set of operations which establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

- 1) In calibration of support equipment the values realized by standards are established through the use of Reference Standards that are traceable to the International System of Units (SI).
- 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the Laboratory with a certificate of analysis or purity, or prepared by the Laboratory using support equipment that has been calibrated verified to meet specifications.

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference material used to calibrate an instrument.

Certified Reference Material (CRM): Reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.

Chain of Custody Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. See also Legal Chain of Custody Protocols.

Clean Air Act: the enabling legislation in 42 U.S.C. 7401 *et seq.*, Public Law 91-604, 84 Stat. 1676 Pub.L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and to enforce them.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: Second column confirmation, Alternate wavelength, Derivatization, Mass spectral interpretation, Alternative detectors, or Additional cleanup procedures

Customer: Any individual or organization for which items or services are furnished or work performed in response to defined requirements and expectations. (ANSI/ASQ E4-2004)

Congener: A member of a class of related chemical compounds (e.g., PCBs, PCDDs)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): the enabling legislation in 42 U.S.C. 9601-9675 *et seq.*, as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 *et seq.*, to eliminate the health and environmental threats posed by hazardous waste sites.

Conformance: an affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Consensus Standard: A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof. (ANSI/ASQ ANSI/ASQ E4-2004)

Continuing calibration verification: The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. (IDQTF)

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Integrity: The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.

Data Quality Objectives (DQO):

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

Definitive Data: Analytical data of known quality, concentration, and level of uncertainty. The levels of quality and uncertainty of the analytical data are consistent with the requirements for the decision to be made. Suitable for final decision-making. (UFP-QAPP)

Demonstration of Capability: a procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Detection Limit: (previously referred to as Method Detection Limit –MDL) the lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit.

Detection Limit (DL) (Clarification): The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Environmental Data: Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology. (ANSI/ASQ E4-2004)

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): the enabling legislation under 7 U.S.C. 135 *et seq.*, as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides.

Federal Water Pollution Control Act (Clean Water Act, CWA): the enabling legislation under 33 U.S.C 1251 *et seq.*, Public Law 92-50086 Stat. 8.16, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance.

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Field of Proficiency Testing (FoPT): Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges, and acceptance criteria have been established by the PTPEC.

Finding: an assessment conclusion, referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement.

Finding (Clarification): An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative and is normally accompanied by specific examples of the observed condition (ANSI/ASQ E4-2004).

Holding Times: The maximum time that can elapse between two (2) specified activities. (TNI)

The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR part 136)

In-depth Data Monitoring: When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.

Inspection: An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Isomer: One of two or more compounds, radicals, or ions that contain the same number of atoms of the same elements but differ in structural arrangement and properties. For example, hexane (C₆H₁₄) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.

Laboratory: Body that calibrates and/or tests. (ISO 25)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Legal Chain of Custody Protocols: procedures employed to record the possession of samples from the time of sampling until analysis and are performed at the special request of the customer. These protocols include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

Limit of Detection (LOD): The minimum result, which can be reliably discriminated from a blank with a predetermined confidence level. Also used is Detection Limit.

Limits of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported with a specified degree of confidence.

For DOD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.

Lot: A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.

Management: Those individuals directly responsible and accountable for planning, implementing, and assessing work. (ANSI/ASQ E4-2004)

Management System: System to establish policy and objectives and to achieve those objectives (ISO 9000).

Matrix: The substrate of a test sample.

Matrix Duplicate: A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.

Matrix Spike (spiked sample, fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Measurement System: A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).

Method: A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.

Method Detection Limit: One way to establish a Limit of Detection.

Method of Standard Additions: A set of procedures adding one or more increments

of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is often called spiking the sample.) (Modified Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Mobile Laboratory: A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans and skid-mounted structures configured to house testing equipment and personnel.

National Institute of Standards and Technology (NIST): A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute. (NMI).

Physical Parameter: A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical or biological components.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis.

Primary Accreditation Body (Primary AB): The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.

Procedure: A specified way to carry out an activity or a process. Procedures can be documented or not.

Proficiency Testing: A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.

Proficiency Testing Provider (PT Provider): A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT program.

Proficiency Testing Provider Accreditor (PTPA): An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.

Proficiency Testing Reporting Limit (PTRL): A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.

Proficiency Testing Sample (PT): sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.

PT Study Closing Date:

- a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider.
- b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.

PT Study Opening Date:

- a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT provider.
- b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed.

Quality Assurance (QA): An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is the type and quality needed and expected by the customer.

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements or quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking intended to demonstrate that a measurement system or activity is in control.

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, the ensure the quality of its product and the utility of its product to the users.

Quality Manual Clarification: Alpha Analytical refers to Quality Manual as Corporate Quality Systems Manual (CQSM). (Alpha)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance (QA) and quality control (QC) activities.

Quality System Matrix: These matrix definitions are to be used for purposes of batch and quality control requirements:

Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.

Reference Material: Material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Method: (To be used to determine the extent of method validation in Modules 3-7.) A reference method is a published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a "standard method", that term is equivalent to "reference method"). When a laboratory is required to analyze an analyte by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is not a regulatory requirement for the

analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or at a given location. (TNI)

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage and disposal.

Revocation: The total or partial withdrawal of a laboratory's accreditation by an accreditation body

Safe Drinking Water Act (SDWA): the enabling legislation, 42 USC 300f *et seq.* (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent.

Sensitivity: The capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Signal to Noise Ratio: The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Signatures, Electronic: A technology that allows a person to electronically affix a signature or its equivalent to an electronic document. The electronic signature links the signature to the signer's identity and to the time the document was signed. Alpha approves the use of electronic signatures for signing and initializing any laboratory record including, by not limited to: analytical reports, controlled documents, workflows and purchasing requests.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.

Standard Operating Procedures (SOPs): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Standard Method: a test method issued by an organization generally recognized as competent to do so.

Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method.

Study (or PT Study): This term refers to a Scheduled PT Study or a Supplemental PT Study.

- a) **Scheduled PT Study:** A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants.
- b) **Supplemental PT Study:** A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard, but that does not have a pre-determined opening date and closing date.

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Suspension: The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed six (6) months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.

Technology: a specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.1, amended)

Tentatively Identified Compound (TIC): A compound that has been identified to be present and is not part of the target compound list (TCL) for the method and/or program. All TICs are qualitatively identified and reported as estimated concentrations. Tentatively Identified Compounds, if requested, are reported for compounds identified to be present and are not part of the method/program Target Compound List, even if only a subset of the TCL are being reported.

Test Method: An adoption of a scientific technique for performing a specific measurement, as documented in a laboratory SOP or as published by a recognized authority.

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 et seq. (1976), the provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.

Tuning: A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e. the air, water and land) upon which human life depends. (US-EPA)

Validation: the confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification: confirmation by examination and provision of evidence that specified requirements have been met.

NOTE - In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring

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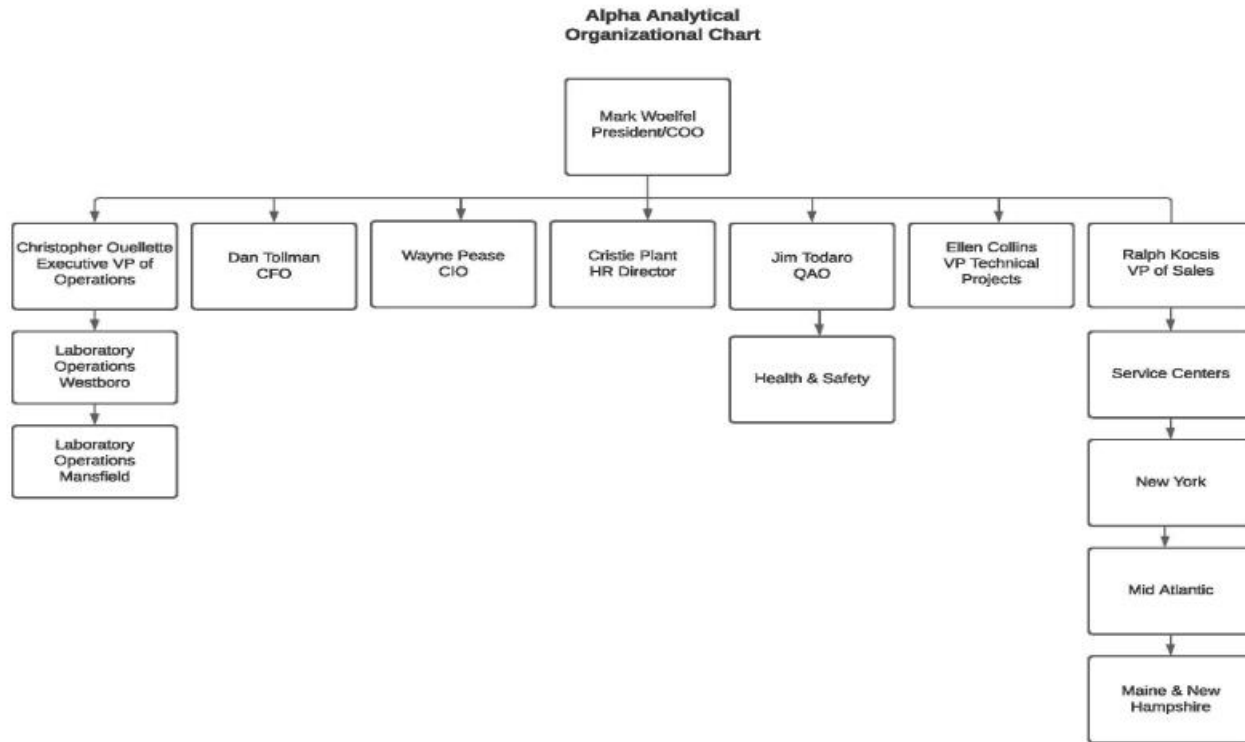
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17 Appendix B – Organization Charts

The following charts provide an overview of the organizational structure of Alpha Analytical. The chart also identifies the key personnel responsible for the listed positions. For the various laboratory areas, the individual departmental supervisors are noted. For a listing of all current key personnel, please refer to Section 18, Appendix C.

Updated 8/3/2022

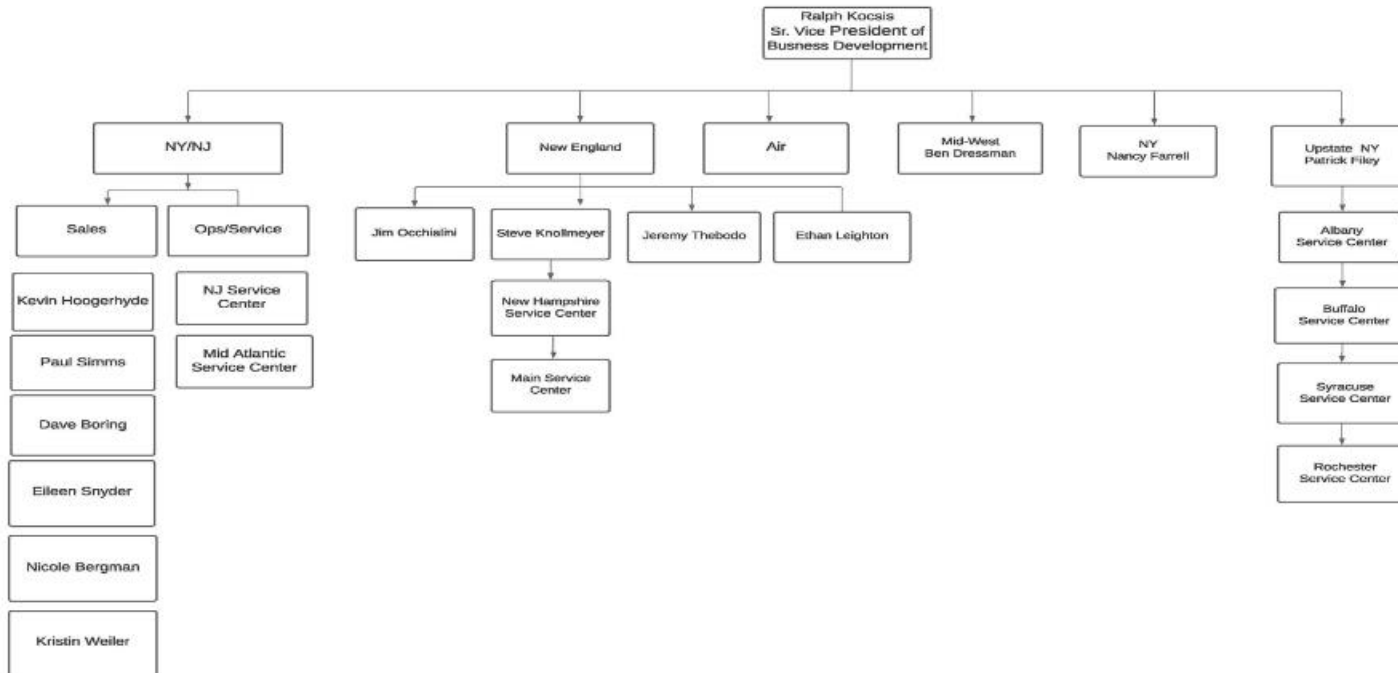


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Updated 11/17/2022
 Alpha Analytical
 Sales Organizational Chart



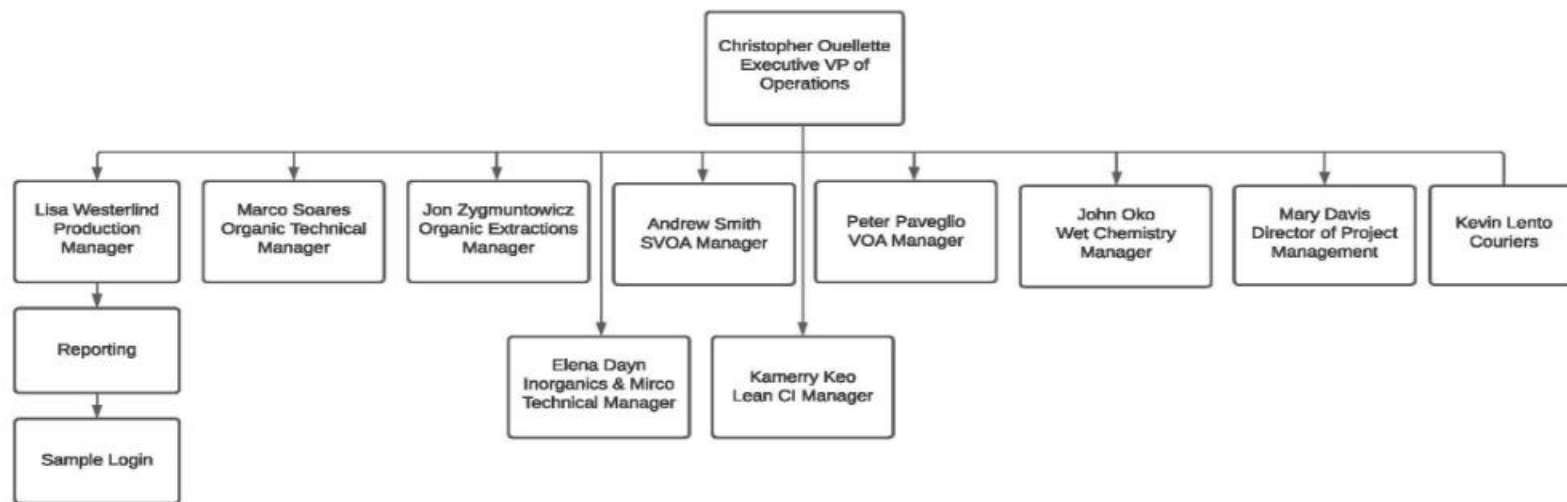
Alpha Analytical
 Sales Organizational Chart



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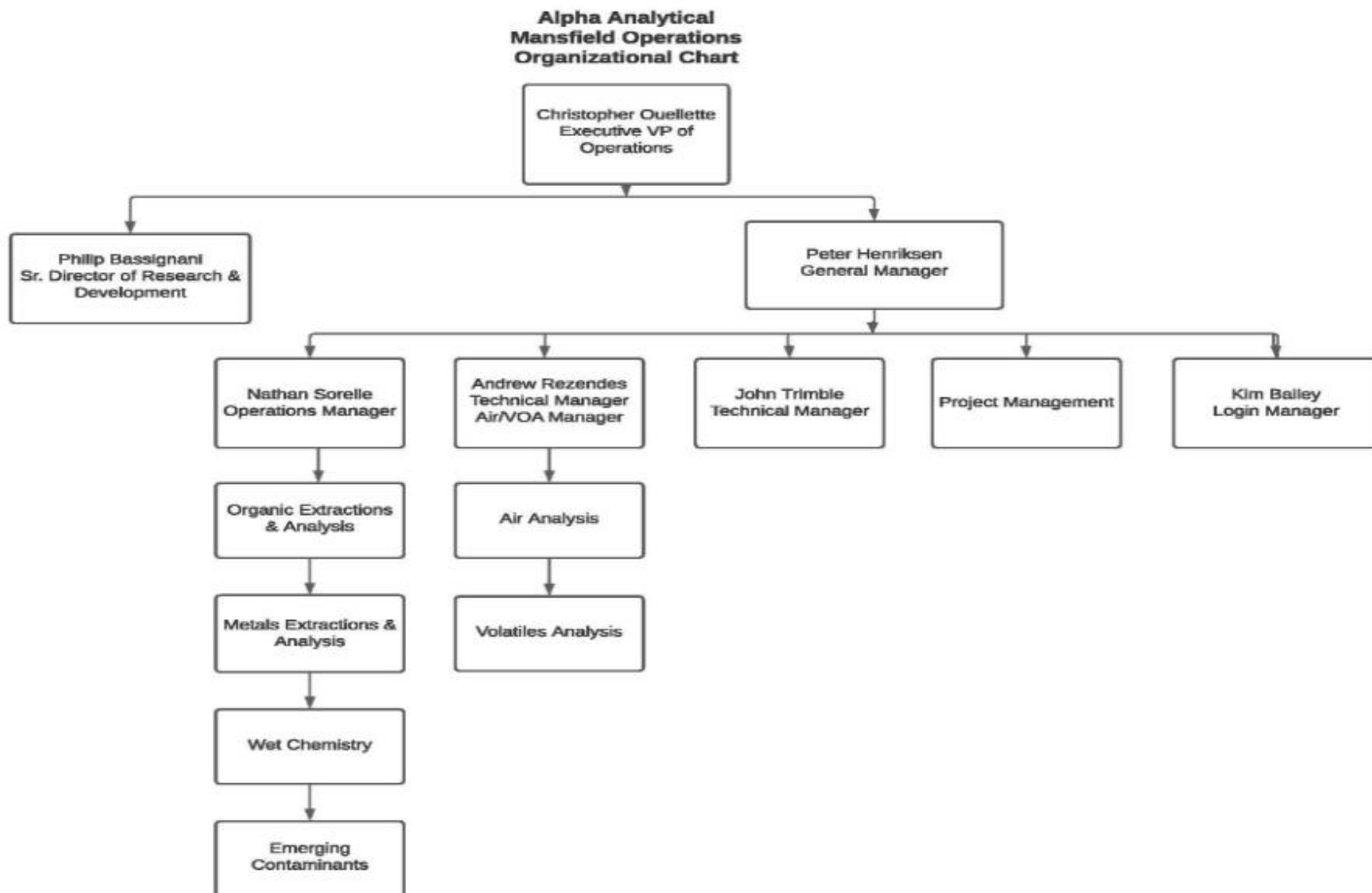
Updated 8/3/2022

Alpha Analytical
Westboro Operations
Organizational Chart



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Updated 8/3/2022



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18 Appendix C – List of Key Personnel

The following is a listing of all current key personnel. If role is specific to a facility it is denoted by either Westboro or Mansfield following the position title. **Updated 11/2022.**

President / COO: Mark Woelfel

Executive VP of Operations: Christopher Ouellette

CFO: Dan Tollman

CIO: Wayne Pease

Laboratory Technical Manager – Organics Westboro: Marco Soares

Laboratory Technical Manager – Inorganics Westboro: Elena Dayn

Laboratory Technical Manager - Mansfield: John Trimble

Laboratory Technical Manager- Air, Volatiles Manager - Mansfield: Andy Rezendes

Quality Assurance Officer/Health & Safety Manager: James C. Todaro

Senior Director of Research & Development: Philip Bassignani

VP, Technical Projects: Ellen Collins

VP of Sales: Ralph Kocsis

VP, Technical Sales: James Occhialini, Patrick Filey, Kevin Hoogerhyde, Stephen Knollmeyer, Nancy Struzenski

Technical Sales Reps: Paul Simms, David Boring, Jeremy Thebodo, Ben Dressman, Ethan Leighton, Kristin Weiler, Nicole Berman

Reginal Technical Coordinator: Eileen Snyder

General Manager, Mansfield: Peter Henriksen

Director of Project Management: Mary Davis

National Air Account Manager: Andy Rezendes

Information Technology Manager: Glenn Fitzgibbons

Service Delivery Manager: Tammy Winter

Human Resources Director: Cristie Plant

Health & Safety Officer: James Todaro

Operations Manager, Mansfield: Nathan Sorelle

SVOA Manager, Westboro: Andrew Smith

Extractions Manager, Westboro: Jon Zygmuntowicz

VOA Department Manager, Westboro: Peter Paveglio

Wet Chemistry Department Manager, Westboro: John Oko

Metals A2 Manager: Cassandra Daley

Metals Department Manager, Mansfield: Grace Deloughery

Metals Prep Manager: Raldi Cabral

Extractions Manager, Mansfield: Cynthia Pimental

Emerging Containments Prep Manager: Ross Lapenta

Login Manager/ Reporting Manager, Westboro Lisa Westerlind

Quality Systems Specialists: Amy Rice, Rene Bennett, Jason Hebert, Michael Selling, Michael Plante, Joseph Fullen

Purchasing: David Peak

Logistics Manager: Kevin Lento

Equipment Technical Specialists: Patrick Sullivan, Szymon Sus, Kimberly Rivera

Continuous Improvement Leader: Kamerry Keo

19 Appendix D – Preventive Maintenance Procedures

Optimized Service-Calibration Intervals		
Equipment	Frequency	Type of Calibration or Maintenance
Balances	semiannually daily	cleaning & operations check by service technician (external) calibration verification using Class S-1 certified weights
COD Reactor	annually	reaction temperature verification
Conductivity Bridge	annually annually each use	verification of cell constant complete operations check by service technician (external) calibration verification
DI Water System	as needed monthly annually daily	complete operations check by service technician (external) Residual Chlorine check Biosuitability testing (external) pH and Conductivity check
DO Meter	annually each use	complete operations check by service technician (external) calibration against air as specified by manufacturer
Emergency/Safety Equipment	annually monthly	fire extinguishers and emergency exit lighting check eye washes, showers, fire blanket and first aid kits checked
Freezers	daily	temperature verification
Gas Chromatographs	as needed as needed beginning and end of batch and 10 to 20 samples as per method	injection port preparation; cleaning of detectors initial multi-point calibration continuing calibration verification (CCV) against initial calibration
ICP	Every other day Daily Annually Annually As needed	Change pump tubing Calibration, profile Complete operations check by service technician (external), Linear Dynamic Range determination Clean torch, clean nebulizer, clean spray chamber
Lachat analyzer	Daily As needed	Calibration, clean lines Change tubing, change O-rings
Mass Spectrometers (GC & ICP)	bi-annually as needed 12 hour or daily	change of mechanical pump oil by service technician (external) cleaning of source BFB, DFTPP or ICP-MS tune analysis followed by ICAL or CCV
Mercury Analyzer	monthly each use	clean cell and change pump windings calibration using multi-point curve
Auto-pipettes	Quarterly Daily	verification of accuracy and precision verification of precision for DOD method auto-pipettes
Microwave	Quarterly Annually	power and temperature verification RPM verification
Ovens	Annually	temperature verification
pH Meters	Annually each use	complete operations check by service technician (external) calibration using certified buffers
Refrigerators (General Use)	daily	temperature verification
Refrigerators (Sample Management)	daily	temperature verification
Spectrophotometer	Semi-annually Semi-annually daily	cleaning & operations check by service technician (external) wavelength verification (external) continuing calibration verification (CCV) against initial calibration
TCLP/ZHE Rotator	Quarterly	RPM verification
Thermometers (Mercury/Alcohol)	Annually	calibration against NIST traceable thermometer (internal)
Thermometers (Bimetal/mechanical)	Quarterly	calibration against NIST traceable thermometer (internal)
Thermometers (digital/IR)	Quarterly	calibration against NIST traceable thermometer (external)
Thermometer (NIST Traceable)	Annually	calibration and certification of conformance (external)
Turbidity meter	Annually each use	cleaning & operations check by service technician (external) calibration using formazin
Weights (Class S-1)	Annually Triennially	Working weights verified against reference weights Reference weights calibrated for conformance (external)

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20 Appendix E – Alpha Code of Ethics Agreement

Alpha Analytical, Inc.
Ethical Conduct and Data Integrity Agreement

A. **Personal Pledge:** I understand that I am charged with meeting the highest degree of ethical standards in performing all of my duties and responsibilities and pledge to only report and/or communicate accurate and precise data and/or information of the highest quality as applicable to my position at Alpha.

B. **Protocol Pledges:** I agree to adhere to the following protocols and principles of ethical conduct in fulfilling my work assignments at Alpha:

1. I will perform all tasks for which I am responsible according to Alpha's Quality System Program and/or the applicable approved documentation.
2. I will not intentionally nor improperly manipulate or falsify data in any manner. I will not modify data values unless the modification can be technically justified through a measurable analytical process or method acceptable to Alpha. All such modifications will be clearly and thoroughly documented per Alpha's Quality System Program.
3. I will not intentionally alter dates and times associated with the collection, custody transfer, analysis and/or reporting of sample data. (Specific to Lab Operations).
4. I will not intentionally represent another individual's work as my own or represent my work as someone else's.
5. I will be honest and not make false statements to, or seek to otherwise deceive Alpha staff, leaders or clients. I will not improperly report and/or communicate measurements, results, data, test results or conclusions.

C. **Guardian Pledge:**

1. I will not condone any accidental or intentional reporting of unauthentic data by other Alpha staff and will immediately report such occurrences to my supervisor, the QA Officer, the Laboratory Technical Manager or corporate leadership. I understand that failure to report such occurrences may subject me to immediate discipline, including termination.
2. If a supervisor or other member of the Alpha leadership group requests me to engage in, or perform an activity that I feel is compromising data validity or quality, I have the right to not comply with the request and appeal this action through Alpha's QA Officer, senior leadership or corporate officers, including the President of the company.
3. I understand that, if my job includes supervisory responsibilities, then I will not instruct, request or direct any subordinate to perform any laboratory practice that is unethical or improper. Also, I will not discourage, intimidate or inhibit a staff member who may choose to appropriately appeal my supervisory instruction, request or directive that may be perceived to be improper, nor retaliate against those who do so.

D. **Agreement Signature:** I have read and fully understand all provisions of the *Alpha Analytical Ethical Conduct and Data Integrity Agreement*. I further realize and acknowledge my responsibility as an Alpha staff member to follow these standards. I clearly understand that adherence to these standards is a requirement of continued employment at Alpha.

Employee Signature

Printed Name

Date

Review Requirements

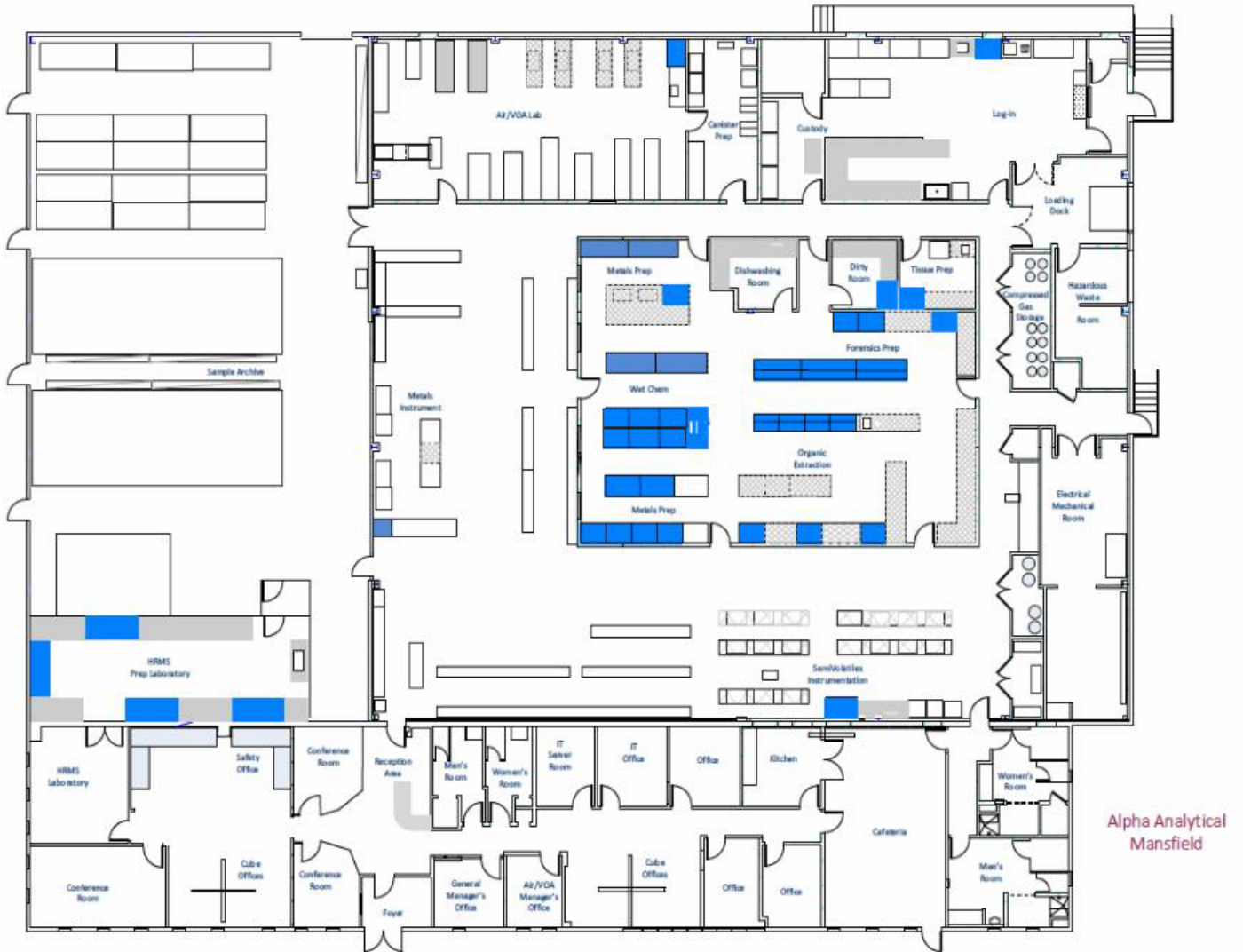
The *Ethical Conduct and Data Integrity Agreement* must be signed at the time of hire (or within 2 weeks of a staff member's receipt of this policy). Such signature is a condition of continued employment at Alpha. Failure to comply with these requirements will result in immediate discharge from Alpha employment. This agreement is not an employment contract and does not modify in any manner the company's *Employment-at-Will Agreement*.

21 Appendix F– Floor Plan Westboro Facility



Alpha Analytical
8 Walkup Drive
Westborough, MA

22 Appendix G– Floor Plan Mansfield Facility



23 Appendix H – Job Titles and Requirements

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Technical Manager (Director) Organic Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 24 credit hours in Chemistry. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of organic analytes in an environmental laboratory	<ol style="list-style-type: none"> 1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Technical Manager (Director) Inorganic Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 16 credit hours in Chemistry. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of inorganic analytes in an environmental laboratory	<ol style="list-style-type: none"> 1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Technical Manager (Director) Microbiology Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 16 credit hours in the Biological Sciences, including at least one course having microbiology as a major component. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of microbiological analytes in an environmental laboratory	<ol style="list-style-type: none"> 1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Quality Assurance Officer	BS/BA in Chemistry, Biology, Environmental or related Science	Two (2) years Environmental Laboratory Experience	<ol style="list-style-type: none"> 1. Advanced technical knowledge of all analytical methods performed by the lab 2. Knowledgeable in Federal, State Programs (THE NELAC INSTITUTE (TNI) STANDARDS, etc.) 3. Able to develop QA/QC policies and certification requirements 4. Able to develop training programs for quality procedures 5. Documented training and/or experience in QA and QA procedures 6. Knowledge of safe laboratory practices and emergency protocols

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TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Laboratory Coordinator	High School Diploma; Associates or BS/BA in Chemistry, Biology or Environmental or related Science preferred	1 year +	<ol style="list-style-type: none"> 1. Knowledge of safe laboratory practices and emergency protocols 2. Proficient in all methods and SOP's within their department 3. Experience with and understanding of LIMS 4. Proven ability to meet TAT (turnaround times)
Quality Systems Specialist	BS/BA Chemistry, Biology, Environmental or related Science	2 years +	<ol style="list-style-type: none"> 1. General knowledge of laboratory methods 2. Experience with and understanding of LIMS 3. Strong attention to detail 4. Strong oral/written communication and organizational skills 5. Knowledge of QA/QC policies and certification requirements
EH&S Coordinator	High School or Equivalent	2 years +	<ol style="list-style-type: none"> 1. General knowledge of lab operations 2. Detailed knowledge of safe lab practices and emergency protocols 3. Hazardous Waste Management and RCRA Regulation Training 4. DOT Hazardous Materials Regulations Training 5. OSHA Compliance Training 6. Able to develop and deliver new hire and ongoing safety training programs
Lab Technician I	HS or Equivalent	0-1 years. 1+ years preferred.	<ol style="list-style-type: none"> 1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Familiarity with standard and reagent preparation 4. Knowledgeable in using volumetric pipettes and glassware 5. Strong oral/written communication and organizational skills
Lab Technician II	HS or Equivalent	2-4 years	<ol style="list-style-type: none"> 1. All skills of Lab Technician I 2. Trained in majority of technician skills relative to department
Lab Technician III	HS or Equivalent	5 years +	<ol style="list-style-type: none"> 1. All skills of Lab Technician II 2. Experienced in training staff
Lab Technician/Chemist I	BS/BA in Chemistry, Biology, Environmental or related Science	0-1 years	<ol style="list-style-type: none"> 1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Familiarity with standard and reagent preparation 4. Knowledgeable in using volumetric pipettes and glassware 5. Strong oral/written communication and organizational skills
Lab Technician/Chemist II	BS/BA in Chemistry, Biology, Environmental or related Science	2-4 years	<ol style="list-style-type: none"> 1. All skills of Chemist I 2. Trained in majority of department methods

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TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Lab Technician/Chemist III	BS/BA in Chemistry, Biology, Environmental or related Science	5 years +	1. All skills of Chemist II 2. Experienced in training staff
Analyst I	HS or Equivalent	0-1 years	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Experienced with sample handling, preparation and/or extraction
Analyst II	HS or Equivalent	2-4 years	1. All skills of Analyst I 2. Experienced in machine operation, maintenance and troubleshooting
Analyst III	HS or Equivalent	5 years +	1. All skills of Analyst II 2. Experienced in data review and reporting 3. Experienced in training staff
Analytical Chemist I	BS/BA in Chemistry, Biology, Environmental or related Science	6 mos-1 year	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Experienced with sample handling, preparation and/or extraction
Analytical Chemist II	BS/BA in Chemistry, Biology, Environmental or related Science	2-4 years	1. All skills of Analytical Chemist I 2. Experienced in machine operation, maintenance and troubleshooting
Analytical Chemist III	BS/BA in Chemistry, Biology, or Environmental or related Science	5 years +	1. All skills of Analytical Chemist II 2. Experienced in data review and reporting 3. Experienced in training staff
Data Deliverable Specialist I	HS Diploma, BS/BA or Associates preferred	0-1 years	1. Introductory knowledge of laboratory methods 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Working knowledge of Adobe Acrobat, Microsoft Word, Excel 4. Good writing and typing skills
Data Deliverable Specialist II	HS Diploma, BS/BA or Associates preferred	2-4 years	1. All skills of Data Deliverable Specialist I 2. General knowledge of laboratory methods 3. Understanding of data review/ data reporting process 4. Experience with and understanding of LIMS and electronic data deliverables

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TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Data Deliverable Specialist III	HS Diploma, BS/BA or Associates preferred	5 years +	1. All skills of Data Deliverable Specialist II 2. Intermediate/advanced knowledge of laboratory methods 3. Able to perform report review 4. Experience with and understanding of LIMS and electronic data deliverables 5. Able to initiate re-work where necessary
Laboratory Intern	2 Semesters of Chemistry, Biology or Environmental Science	None; Lab work study experience preferred	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures

KEY

* Internal terms only. Full title would have "Environmental Laboratory" and specific department preceding it.

** Substitutions: Equivalent knowledge may be substituted for a degree in some instances.

*** Not meant to be an exhaustive list of skill requirements. For full list of skills consult the "Laboratory Skills" list. Actual Job Duties and Responsibilities can be found within job descriptions for each position.

24 Appendix I – Standard Operating Procedures

WESTBORO SOP #	Title
1728	Waste Management and Disposal - Westborough
1730	Balance Calibration Check
1733	Thermometer Calibration
1737	Inorganics Glassware Cleaning and Handling
1738	Water Quality Monitoring
1745	Reagent, Solvent and Standard Control
1948	Separatory Funnel Liquid-Liquid Extraction – EPA 3510C
1953	Organic Extraction Glassware Cleaning & Handling
1954	Soxhlet Extraction – EPA 3540C
1955	Sulfur Cleanup – EPA 3660A
1956	Oil and Waste Dilution – EPA 3580A
1959	Microwave Extraction – EPA 3546
1960	Sulfuric Acid Cleanup – EPA 3665A
1962	Florisil Cleanup
1963	Fractionation Cleanup
1964	Preparation of Samples for Chlorinated Herbicides
2107	Volatile Organic Compounds – EPA 524.2
2108	Volatile Organic Compounds – EPA 8260C
2109	Polynuclear Aromatic Hydrocarbons (PAHs) by SIM – EPA 8270D (modified)
2111	Semivolatile Organics by GC/MS – EPA 8270D
2112	TCLP/SPLP Extraction - Volatile Organics SW-846 Method 1311/1312
2113	EDB, DBCP & TCP in Water by Microextraction & Gas Chromatography – EPA 504.1, 8011
2116	Organochlorine Pesticides by Capillary Column GC – EPA 8081B
2119	Extractable Petroleum Hydrocarbons – MADEP
2120	Volatile Petroleum Hydrocarbons – MADEP
2123	Polychlorinated Biphenyls in Oil – EPA 600/4-81-045
2125	TPH-Diesel Range Organics, Maine 4.1.25, EPA 8015C (Modified)
2127	CT-ETPH
2128	Herbicides by 8151A
2129	PCBs by Capillary Column Gas Chromatography - EPA 8082A
2131	New Jersey EPH Method
2133	TCLP Extraction Metals and Semi-Volatile Organics – SW-846 Method 1311
2135	SPLP Extraction Inorganics and Semivolatile Organics, EPA 1312

WESTBORO SOP #	Title
2161	Fecal Coliform by Membrane Filtration – SM 9222D
2163	Fecal Coliform by Multiple Tube Fermentation – SM 9221E
2191	Heterotrophic Plate Count – SM 9215B
2192	Total Coliform/E.Coli – Presence/Absence (Colilert) – SM 9223B
2194	Total Coliform by Multiple Tube Fermentation – SM 9221B
2195	Chlorophyll A – SM 10200H
2196	E. Coli – Membrane Filtration
2197	Chlorophyll A – EPA 446
2198	Air Density Monitoring
2199	Inhibitory Residue Test
2200	Enterococcus – MF
2201	Total Coliform, E.Coli & Enterococcus by Quantification Methods (Quanti Tray)
2202	pH, Liquid Samples
2203	pH, Soil & Waste Samples
2204	Hexavalent Chromium
2205	Biological Oxygen Demand
2206	Ammonia Nitrogen
2207	Total Kjeldahl Nitrogen
2208	Chemical Oxygen Demand
2209	Oil & Grease by n-Hexane Extraction Method & Gravimetry
2210	Cyanide, Total
2211	Phenol, Total
2212	Sulfate, Turbidimetric Method
2213	Alkalinity, Titration Method –SM 2320B
2214	Determination of Inorganic Anions by Ion Chromatography – EPA 300.0
2215	Total Organic Carbon/Dissolved Organic Carbon
2216	Chloride – SM 4500Cl-E, EPA 9251
2217	Nitrate, Nitrite and Nitrate/Nitrite Nitrogen – EPA 353.2, SM 4500NO ₃ -F
2218	Total Solids (Dried @ 103-105°) and TVS – SM 2540B, SM 2540E
2219	Total Dissolved Solids – SM 2540C
2220	Total Suspended Solids – SM 2540D
2221	Total Sulfide – SM 4500S ₂ -AD, EPA 9030B
2222	MBAS, Anionic Surfactants – SM 5540C
2223	Fluoride, Electrode Method – SM 4500F-BC
2224	Turbidity, Nephelometric Method – EPA 180.1, SM 2130B
2225	Orthophosphate, Colorimetric Single Reagent Method – SM 4500P-E
2226	Total Phosphorous, Colorimetric Combined Reagent Method – SM 4500P-E
2227	Flashpoint – EPA 1010

WESTBORO SOP #	Title
2228	Reactivity – EPA Chapter 7.3
2229	Total Solids (Dried @ 103-105°) – SM 2540G
2230	Specific Conductance and Salinity
2231	True and Apparent Color, Visual Comparison Method
2232	Acidity, Titration Method
2233	Determination of Formaldehyde by HPLC, EPA 8315A
2234	Sulfite, Iodometric
2235	Ferrous Iron
2236	Residual Chlorine
2237	ORP
2238	Ignitability of Solids EPA 1030
2239	Physiologically Available Cyanide (PAC)
2240	Total Settleable Solids SM 2540 F
2241	Fixed and Volatile Solids in Solid and Semisolid Samples – SM 2540G
2242	Tannin & Lignin
2243	Nitrite - Manual Colorimetric Method
2244	Paint Filter Liquids Test
2245	Odor, Threshold Odor Test
2249	Dissolved Oxygen
2251	Perchlorate by IC/MS/MS
3743	Free Cyanide
9177	Total Phenol - SEAL Method
9733	Oil & Grease and TPH in Soil
10807	Percent Organic Matter in Soil
14751	Determination of UV-Absorbing Organic Constituents at 254nm
17972	Extractable Organic Halides (EOX)
18236	Chloropicrin and Carbon Tetrachloride by EPA 8011
19332	DI Water Extraction ASTM D3987
21994	Nonfractionated EPH
23148	Gilson EPH Fractionation
25691	Semivolatile Organic Compounds by GC/MS EPA 625.1
25693	Volatile Organic Compounds by EPA 624.1
26801	TPH - Gasoline Range Organics Maine 4.2.17, EPA 8015D
27634	True and Apparent Color, Single Wavelength Method
28200	PCBs by EPA 608.3
28201	Pesticides by EPA 608.3
32637	Polynuclear Aromatic Hydrocarbons (PAHs) by SIM EPA 8270E (M)
32639	Volatile Organic Compounds EPA 8260D
33262	Extractable Petroleum Hydrocarbons (MA-EPH) 2.1

MANSFIELD SOP #	Title
1753	Glassware Cleaning
1754	Balance Calibration
1755	Pipette Checks
1797	Waste Management and Disposal - Mansfield
1816	Reagent Solvent Standard Control
2134	Hot Block Digestion for Aqueous Samples EPA 3005A
2138	Mercury Aqueous 7470A
2139	Mercury Soil 7471B
2140	AVS SEM
2141	Hydride Generation
2142	Mercury Aqueous 7474
2143	Mercury Soil 7474
2148	Metals Soil Digestion 3050
2150	Metals Microwave 3015
2155	EPA 8270D
2157	PAH by SIM
2158	EPA 8081B
2160	EPA 8082A Aroclors/Congeners by GC and TO-10A
2162	Pesticides/PCB Aroclors/Congeners by GC/MS SIM
2164	1,4-Dioxane GC/MS SIM
2165	Separatory Funnel Extraction EPA 3510C
2166	Tissue Prep
2167	GPC
2168	Sulfur Cleanup 3660
2169	Sulfuric Acid Cleanup 3665
2170	Silica Gel Cleanup
2171	% Lipids
2172	Microscale Solvent Extraction EPA 3570
2173	Soxhlet Extraction EPA 3540C
2174	Soxhlet Extraction of PUFs
2175	% Total Solids
2182	TOC by Lloyd Kahn
2183	Particle Size Determination
2184	Particulates in Air PM-10
2186	EPA TO-15
2187	APH
2188	Air PIANO
2189	Dissolved Gases RSK-175

MANSFIELD SOP #	Title
2190	Cleaning & Preparation of Air Sampling Equipment
2246	TPH and SHC
2247	Alkylated PAH
2248	Organic Lead
2252	Fixed Gases
2255	PIANO Volatiles
2256	Ethanol in Oil
2257	Whole Oil Analysis
2259	Density Determination of Oils
2260	Alumina Cleanup
2261	Shaker Table
2263	Gravimetric Determination
2264	Tissue Extraction
2265	Organic Waste Dilution
2267	Client SOP: SGC - Manual Method
2268	Client SOP: DCM Extractable Method
4246	PAHs by SPME
6438	Mercury in Sorbent Tubes by CVAA
7900	Mercury 1631E Using Cetac-M-8000 Analyzer
9077	Porewater Generation
9480	EPA-TO-12
12863	EPA 8270D GC/MS Full Scan TO-13A
13091	HPAH
13406	Particulate Organic Carbon
14500	Lead in Particulate Matter
17452	TOC by EPA 9060A
17456	Moisture, Ash and Organic Matter
17829	Specific Gravity of Soil
17830	Liquid Limit, Plastic Limit and Plasticity Index of Soils
17940	1,4-Dioxane in Drinking Water by EPA 522
18086	Total Suspended Solids (TSS) SM 2540D
18705	PCB Congeners by GC/MS-SIM EPA 8270D
18710	Trace Elements in Waters and Wastes by ICP-MS EPA 200.8
18711	Metals by ICP EPA 200.7
18715	Mercury in Water (CVAA) EPA 245.1
18716	Hot Block Digestion for Aqueous Samples EPA 3005A
18717	Microwave Assisted Acid Digestion of TCLP Extracts EPA 3015
18718	Microwave Assisted Acid Digestion for Metals EPA 3015A/3051A
18817	Alcohols by FID- Aqueous Direct Injection EPA 8015D

MANSFIELD SOP #	Title
19625	Glycols by GC-FID EPA 8015D
19971	Air Drying Samples for PCBs and Metals Analysis
19978	Density of Soil
22132	Data Review – Ohio VAP
23511	PFAS by LC/MS/MS by EPA 537
23528	PFAS by LC/MS/MS Isotope Dilution by EPA 537(M)
24454	Acetonitrile Extraction for Unknown Compounds via GCFID
25896	EPA 8290A Dioxins and Furans by Hi-Res MS
25900	EPA 1613B Dioxins and Furans by Hi-Res MS
25923	Mercury in Liquid Waste (Automated Cold-Vapor Technique) EPA 7470A
25924	Mercury in Solid/Semisolid Waste (Manual Cold-Vapor Technique) EPA 7471B
26796	Metals by ICP EPA 6010D
26797	Metals by ICP-MS EPA 6020B
27056	HiRes Laboratory Glassware Cleaning
27322	In Vitro Accessibility Assay for Lead in Soil EPA 1340
27360	PFAS in Cranberry Matrix by EPA 537 (M) LC/MS/MS Isotope Dilution
27485	Total Petroleum Hydrocarbons Screen by GC/FID 8015D
27897	PCB Congeners by High Resolution GC/MS
29033	PFAS by LC/MS/MS in Non-Potable Water
29139	Biomimetic Extraction Using SPME
32082	MADEP PFAS by SPE & LC/MS/MS Isotope
31164	Semivolatile Organic Compounds By Gas Chromatography / Mass Spectrometry (GC/MS) 8270E
32324	PAH and PCB Congeners by GCMS with SIM 8270E TO-13A
32200	EPA 533 PFAS LC/MS/MS Isotope Dilution
36216	PFAS LC/MS/MS Isotope Dilution Nonpotable Water
36957	PFAS by EPA 537.1 in Drinking water by LC/MS/MS
45852	Method 1633 Draft PFAS in Aqueous, Solid, Biosolids and Tissue by LCMSMS
40380	Resin Extraction

CORPORATE SOP #	Title
1559	Sample Receipt and Login
1560	Sample Custody and Tracking
1561	Bottle Order Preparation
1562	Computer System Backup/Control
1563	Computer and Network Security
1564	Software Validation and Control

CORPORATE SOP #	Title
1565	Training Program
1566	Report Generation and Approval
1567	Organics Data Deliverable Package Review
1722	Customer Inquiry and Complaint Procedures
1723	Project Management
1724	Quote/Contract Procedure
1725	Project Communication Form Generation
1726	Purchasing Procedure
1727	Accounts Payable Invoice Processing
1729	Document Control
1731	Manual Integration and Compound Rejection
1732	DL LOD LOQ Generation
1734	Control Limit Generation
1735	Analytical Guidelines for Method Validation
1736	Corrective and Preventative Actions
1738	Water Quality Monitoring
1739	Demonstration of Capability (DOC) Generation
1740	Internal Audit Procedure
1741	Data Review – Organics
1742	Calculating Measurement Uncertainty
1743	Annual Management Review
1744	Sample Compositing Procedure
1746	Nonconformance Planning/Procedures
1747	Temperature Datalogger Operation
2274	Data Validation Package
17553	Lab Supply Transfer Procedure
18821	Weights Verification
18909	PT Corrective and Preventive Action Process



APPENDIX D

Laboratory SOP's

Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

References: **Method 8260D**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update VI (Phase II), June 2018.

Method 5035A, SW-846, Closed System Purge & Trap and Extraction for Volatile Organics in Soil and Waste Samples. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Draft Revision I, July 2002.

Method 5030B, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December 1996.

Method 5030C, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, May 2003.

1. Scope and Application

Matrices: Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Definitions: Refer to Alpha Analytical Quality Manual.

The compounds listed in Table 5 may be determined by this method.

There are various techniques by which these components may be introduced into the GC/MS system. Purge-and-trap, by Methods 5030C (aqueous samples) and 5035A (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. One technique is direct injection of an aqueous sample (concentration permitting).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Department Manager, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph/mass spectrometers and in the interpretation of mass spectra and their use as a quantitative tool. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection. The analytes are introduced to a narrow-bore capillary column for analysis. The Gas Chromatograph (GC) is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the GC.

Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard, comparing sample response to the calibration standards.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 1 lists our typical reporting limits.

4. Interferences

- 4.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be free from contamination under the conditions of the analysis. Running laboratory reagent blanks as described in Section 9.1 and 10.3 demonstrates the system is free of contamination. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system must be avoided.
- 4.2 Sample contamination occurs by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A trip blank or a field reagent blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination.
 - 4.2.1 Storage blanks shall be analyzed if contamination is suspect. If contamination is confirmed by positive detections in the sample storage blanks, all data from samples contained in the relative refrigerator or freezer shall be evaluated for possible contamination. If the samples contain suspected contamination, the Client Services department shall be notified in order to contact the necessary clients regarding the contamination. Samples shall be reanalyzed if so desired by the client. If suspected contamination is not confirmed by storage blanks, no further action shall be pursued concerning said blanks. It is recommended that further action be taken to determine the possible cause of suspected contamination.
- 4.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. Whenever a highly concentrated sample is being encountered, it should be followed by an analysis of reagent water (instrument blank) to check for potential contamination. If carry-over is suspected, then numerous instrument blanks may be required; additionally, all affected samples are rerun for confirmation. In case of severe contamination, preventive maintenance of the entire system may be required.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of [safety data sheets \(SDS\)](#) is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

- 5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, standards, or solvents.
- 5.2 All stock solution standard preparation must be performed in the volatiles hood. Initial calibration, continuing calibration, laboratory control sample and client sample dilutions do not need to be performed in the hood.
- 5.3 All expired standards must be placed into the waste bucket in the lab for future disposal. The container must be labeled properly with hazard warning labels indicating the container contents.
- 5.4 Bottles containing Methanol must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Storage, Shipping and Handling

6.1 Sample Collection and Preservation

6.1.1 Aqueous Samples

Grab samples are collected in standard 40mL glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two or more VOA vials should be filled per sample location. EPA Method 8260 requires that samples be acidified to eliminate the possibility of biological degradation. Unless otherwise directed for project-specific reasons, all VOA vials are delivered to the client with approximately 2 – 4 drops of 1:1 HCl added to the vial, which is sufficient to adjust the pH of the sample to < 2. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Fill the sample vial to the point of overflowing so that no headspace is contained within. Samples must be introduced into the vials gently to reduce agitation, which might drive off volatile compounds or cause loss of the HCl preservative.

Seal the bottle so that no air bubbles are in the VOA vial. If preservative has been added, shake vigorously for one minute. Invert the bottle and tap to check for air bubbles. Recollect the samples if any air bubbles are present.

Maintain the hermetic seal on the VOA vial until time of analysis.

6.1.2 Soil Samples

The recommended sampling method for soil samples is EPA 5035A. Method 5035A provides for two distinct sampling procedures, depending on the required reporting limits and suspected or known concentration levels of target analytes. These methods are referred to as the High Level and Low Level methods. Both are listed below but depending on the samples only one of the methods may be required. If concentration levels are unknown, it is recommended that samples be collected using both procedures.

The Lab will analyze the high level sample first, followed by the low level sample if the results from the high level analysis show that the sample is clean or contains analytes at low levels. The typical reporting levels of the two methods are listed in Table 1.

6.1.2.1 High Level Soil Samples

Collect sample in a standard 40mL glass screw-cap vial with Teflon lined silicon septa (VOA vial). The vial is provided containing 15mL of Purge and Trap Grade methanol and is labeled and weighed prior to addition of sample. Record the weight of the vial with methanol on the vial label. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Approximately 15g of soil is added to the vial in the field, making sure that the sample is completely covered by the methanol.

Maintain the hermetic seal on the VOA vial until the time of analysis.

An additional sample of the soil must also be obtained (without methanol) to be used for the determination of soil moisture content to allow for the calculation of the dry weight results, and to calculate the methanol dilution effect. (See Sections 11.2.2.2 and 11.2.2.2.1)

6.1.2.2 Low Level Soil Samples

Collect sample in a standard 40mL glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two samples should be taken per sample location. Vials are provided containing a magnetic stirring bar and 5 mL of either 200g/L sodium bisulfate solution or water, prepared by a certified vendor. These vials are labeled and weighed prior to addition of sample. Record the weight of the vial with the stirring bar and preservative on the vial label.

Approximately 5g of soil is added to the vial in the field, making sure that the sample is completely covered by the sodium bisulfate solution or water.

Maintain the hermetic seal on the VOA until the time of analysis.

6.2 Sample Handling and Storage

Document client specific sample handling, preservation and collection criteria in the project file. The laboratory Log-in staff documents sample temperature at the time of receipt.

Record deviations from this SOP or client specific criteria on the chain of custody form.

Record holding time exceedance, improper preservation and observed sample headspace on the nonconformance report form.

6.2.1 Aqueous Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 1 and 4 °C. Sample receiving personnel must note on the sample delivery group form when samples received at the laboratory are not within the temperature criteria. If more than one vial is received for a sample, the vials are stored in separate refrigerators. Storing the vials apart provides a useful check if laboratory contamination of a sample is suspected. Samples must be analyzed within 14 days of collection. Unpreserved samples must be analyzed within 7 days of collection.

6.2.2 High Level Soil Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Sample receiving personnel note on the

nonconformance report form when samples received at the laboratory are not within the temperature criteria. Samples must be analyzed within 14 days of collection.

6.2.3 Low Level Soil Samples

Ice or refrigerate samples preserved with water or sodium bisulfate from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Samples preserved with water are to be immediately frozen after sampling. Sample receiving personnel note on the nonconformance report form when samples received at the laboratory are not within the temperature criteria. Samples must be analyzed within 14 days of collection.

6.3 Sample Shipping

Samples requiring shipment to the laboratory are shipped in ice-packed coolers via an overnight delivery service in accordance with applicable Department of Transportation regulations.

7. Equipment and Supplies

7.1 Purge and Trap System (For Aqueous samples): The purge-and-trap system consists of two separate pieces of equipment: a purging device (autosampler) (TekmarAtomxXYZ, EST Centurion or equivalents) coupled to the desorber (concentrator) (EST Encon or Encon EV, or equivalents).

- 7.1.1 Purge gas = Helium analytical grade (99.999%) or Nitrogen analytical grade (99.999%).
- 7.1.2 The purging device is configured with 25 mL sample purge tubes, and the purge gas is introduced at the bottom of the water column as finely divided bubbles.
- 7.1.3 The trap used in the desorber is typically a Supelco "K" trap. Different traps may be used if equivalent performance is demonstrated.
- 7.1.4 The desorber is capable of rapidly heating the trap to 260°C. The trap is not heated above manufacturer's specifications.

7.2. Purge and Trap System (For Low Level and High Level Soil Samples): The purge and trap system consists of two separate pieces of equipment: a purging device (autosampler) (Tekmar AtomxXYZ, EST Centurion or equivalents) coupled to the desorber (concentrator) (Tekmar Velocity or Atomx, or EST Encon or Encon EV, or equivalents).

- 7.2.1 Purge gas = Helium analytical grade (99.999%) Nitrogen analytical grade (99.999%).
- 7.2.2 The autosampler purging device is a closed system, designed to accept the 40mL VOA vials. The VOA vial, containing the soil sample, water (or sodium bisulfate), and stirring bar is placed into the autosampler tray. The instrument automatically adds reagent water, internal standards, and surrogates to the unopened VOA vial. The vial is heated to 40 °C, and the purge gas is introduced into the aqueous portion to purge the volatile components onto the trap.
- 7.2.3 The trap used in the desorber is typically a Supelco "K" trap. Different traps may be used if equivalent performance is demonstrated. The desorber is capable of rapidly heating the trap to 260 °C. The trap is not heated above manufacturer specifications.

7.3 Gas Chromatography/Mass Spectrometer/Data System:

- 7.3.1 **Gas Chromatograph, Agilent 6890/7890 or equivalent:** An analytical system complete with a temperature-programmable gas chromatograph with appropriate

interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source of the GC/MS system.

7.3.2 Typical Gas Chromatographic Columns:

7.3.2.1 Column 1: Restek 502.2, 40 meter, 0.18mm ID, 1.0um, or equivalent.

7.3.2.2 Column 2: Restek RTX-VMS, 30 meter, 0.25mm ID, 1.4um, or equivalent.

7.3.2.3 Column 3: Restek RTX-VMS, 20 meter, 0.18mm ID, 1.5um, or equivalent.

7.3.3 **Mass Spectrometer, Agilent 5973/5975/5978 or equivalent:** Capable of acquiring mass spectra from mass/charge (m/z) 35 to 270 at a rate fast enough to acquire at least five (but preferably 10 or more) mass spectra across each chromatographic peak of interest, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 3, when 50ng of the GC/MS tuning standard (BFB) are injected through the GC. For all SIM analysis, the mass spectrometer must also be able to acquire data in a dual acquisition mode (SIM and full scan).

7.3.4 **Data System:** Hewlett-Packard EnviroQuant software is used for data acquisition and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

EnviroQuant E.02.02 (or equivalent) is used for data processing and allows searching of any GC/MS data file for ions of a specified m/z, and plotting such ion abundances versus time or scan-number.

The most recent version of the EPA/NIST Mass Spectral Library is loaded onto the Target / EnviroQuant data system.

7.4 **Wiretrol or Micro syringes:** 10µL - 1,000µL.

7.5 **Syringes:** 5mL, 10mL, or 25mL, glass with Luerlock tip.

7.6 **Balances:** Top-loading, capable of weighing 0.01g.

7.7 **Vials:** 2mL, 4mL.

7.8 **Disposable Pipets.**

7.9 **Volumetric Flasks:** Class A, appropriate sizes, with ground-glass stoppers.

7.10 **Eppendorf pipets.**

7.11 **Ottawa Sand: SiO₂**

8. Reagents and Standards

Reagent grade organic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Great care must be taken to maintain the integrity of all standard solutions. Standards in methanol are stored at -10°C or less, in amber vials with PTFE-lined screw-caps.

8.1 Organic-free Reagent Water:

All references to water in this method refer to organic-free reagent water, which is tap water passed through activated carbon and air bubbled through.

8.2 Methanol:

Purge and Trap Grade or equivalent. Store in flammables cabinet.

8.3 Stock Solutions:

All stock standards are purchased from commercial vendors as certified ampule solutions. When an ampule of stock solution is opened, it is transferred to a labeled amber screw-cap vial with minimal headspace. The expiration date of the stock solution is either the vendor specified expiration date or 6 months from the date the ampule was opened, whichever is sooner. Typical stock standard concentrations are listed in Table 4.

8.4 Intermediate Standards: Intermediate standards are prepared volumetrically by diluting the appropriate stock standard(s) with methanol. Initial Calibration solutions expire 2 months from the date of preparation, or sooner if daily continuing calibration checks do not achieve the method acceptance criteria. If the Intermediate Standards are used as a second source to verify a valid Initial Calibration solution, there is no expiration date.

8.4.1 Internal Standard Solutions:

The internal standards are Fluorobenzene, Chlorobenzene- d_5 , and 1,4-Dichlorobenzene- d_4 . The intermediate IS solution is prepared by diluting the stock solution(s) with methanol to a concentration of $100\ \mu\text{g}/\text{mL}$. The appropriate amount of IS solution is added to the water or soil sample or QC sample to achieve a final concentration of $100\ \text{ng}/\text{sample}$ or standard. Internal standard is added at the same concentration to all standards, samples, and QC samples.

8.4.2 Surrogate Standard Solutions:

The surrogate standards are Dibromofluoromethane, 1,2-Dichloroethane- d_4 , Toluene- d_8 , and 4-Bromofluorobenzene. The intermediate surrogate solution is prepared by diluting the stock solution(s) with methanol to a concentration of $100\ \mu\text{g}/\text{mL}$. The appropriate amount of surrogate solution is added to the water or soil sample or QC sample to achieve a final concentration of $100\ \text{ng}/\text{sample}$.

8.4.3 Target Compound Solutions:

The target analytes routinely reported by this method are listed in Table 1 and 4. The intermediate target compound solutions are prepared by diluting the stock solution(s) with methanol. This set of solutions, at concentrations of $200\ \mu\text{g}/\text{mL}$, is used for preparation of the calibration standards.

8.4.4 4-Bromofluorobenzene (BFB) Tune solution:

8.4.4.1 A solution containing BFB at a concentration of 50 µg/mL is prepared by volumetrically diluting the BFB stock solution. 1 µL of this solution is direct-injected or purged into the GC/MS system to verify system performance prior to any standard or sample analysis.

8.4.4.2 BFB may be analyzed in full scan mode while standards, samples, and QC are analyzed in SIM

8.5 Calibration Standards:

There are two types of calibration standards used for this method – initial calibration standards and calibration verification standards.

8.5.1 Initial Calibration Standards:

Initial calibration standards can be prepared at the levels listed in Table 4 (other/different levels are allowed). The Initial Calibration needs to have a minimum of 5 standards, 6 if a quadratic curve fit is used. Prepare these solutions in organic-free reagent water. The standards correspond to the range of concentrations found in typical samples and do not exceed the working range of the GC/MS system. Initial calibration should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

8.5.2 Initial Calibration Verification Standard (ICV):

The initial calibration verification standard is at the same concentration as the level 3 for liquids and level 4 for soils initial calibration standard. This standard is prepared from source materials from a second manufacturer or from a manufacturer's batch prepared independently from the batch used for calibration. A second lot# from the same manufacturer may be adequate to meet this requirement. The standard must contain all calibrated target analytes that will be reported for the project, if readily available.

8.5.3 Continuing Calibration Verification Standard:

The continuing calibration verification standard, or calibration check standard, should be analyzed near the action level of the project. Since most projects are focused on achieving low reporting limits, the continuing calibration verification standard is at the same concentrations as the level 3 for liquids and level 4 for soils initial calibration standard. This standard is run at the beginning of each analytical sequence, following the BFB tune standard, to verify system performance.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

9.1.1 Before processing any samples, the analyst must demonstrate through the analysis of a method blank (MB) or instrument blank that equipment and reagents are free from contaminants and interferences. The blank must contain the internal standards and surrogates and must be from the same source of water used for preparing the standards, QC samples and sample dilutions.

9.1.2 Analyze a matrix-specific blank, i.e. methanol samples need to have methanol in the blank; sodium bisulfate samples need to have a sodium bisulfate blank analyzed;

TCLP samples need a TCLP blank, after each ICAL and CCV, and prior to any sample analysis to demonstrate that interferences from the analytical system are under control.

- 9.1.3 Low level soil blanks must include 5g of Ottawa sand and be vortexed.
- 9.1.4 Blanks are considered to be acceptable if target analyte concentrations are less than one half the LOQ or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e., target analytes are not present in samples or sample concentrations/responses are >10X the blank).
- 9.1.5 If an analyte of interest is found in a sample in the batch near a concentration detected in the blank, the presence and/or concentration of that analyte should be considered suspect and may require qualification. Reanalysis is not necessary if the analyte concentration falls well below the action or regulatory limit or if the analyte is deemed not important for the project.
- 9.1.6 The laboratory must not subtract the results of the MB (or any blank) from those of any associated samples.

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

An LCS/LCSD pair is analyzed at the beginning of each analytical sequence. Since the LCS contains the same compounds at the same concentrations as the continuing calibration check standard, the same analysis is used to satisfy both QC elements. The LCS/LCSD acceptance criteria are based on in-house control limits, unless specified by project/regulation. Less than 10% of total compounds may be outside of control limits if recoveries are >10%. (Note: this does not apply to difficult analytes listed in Table 7 which may be accepted at recoveries <10.) If >10% of analytes are recovered above control limits, this is deemed acceptable as long as the analytes in question are not detected in associated samples. If these criteria are not met, the entire batch is re-extracted. If re-extraction is not possible, due to insufficient sample or holding time exceedance, the analyst must write up the failure on a narrative sheet for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to 12

9.4 Continuing Calibration Verification (CCV)

Refer to [Section 11.2](#).

9.5 Matrix Spike/ Matrix Spike Duplicate

Upon Client Request or state mandate, a matrix spike/matrix spike duplicate pair may be analyzed with each batch of 20 or less samples. The MS/MSD are sample aliquots spiked with the target compounds at the same concentration as the continuing calibration standard for water analysis and level 6 for soils. The MS/MSD acceptance criteria are based on in-house control limits. If the MS/MSD does not meet the criteria, but the LCSD does, the failure may be attributed to sample matrix. Report the MS/MSD, including a narrative sheet for inclusion with the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Internal Standards

Area counts of the internal standard peaks in all samples and QC samples must be between 50-200% of the areas of the internal standards in the QC check standard.

If any individual percent recovery falls outside the range, that parameter has failed the acceptance criteria. For calibration standards, CCVs, LCS/LCSD or blanks the internal standard must be within the range for data to be reported to the clients. For samples, matrix spikes and duplicates: if the data is not within the range, the sample is rerun to confirm that the failure is due to sample matrix. A nonconformance report form is completed to ensure client notification and reporting if matrix effect is confirmed.

9.7.2 Surrogates

Surrogates are added to each field sample and QC sample. The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The surrogate acceptance criteria are listed in [Table 2](#). Since the SIM analysis is acquired in dual mode, the surrogates from the full scan are used to evaluate the entire sample (SIM and full scan).

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is as follows:

- BFB
- QC Check Standard/Laboratory Control Sample/LCSD
- Method Blank
- Samples
- MS/MSD (upon Client request, may be run any time after the Method Blank)

10. Procedure

10.1 Equipment Set-up

Typical instrument operating conditions are listed below. Alternate conditions are allowed if method performance criteria can be met.

10.1.1 GC Conditions:

Temperature 1:	35°C	Carrier gas:	Helium 99.999% or Nitrogen 99.999%
Hold Time 1:	4 minutes	Carrier mode:	Constant flow
Ramp 1:	6°C/minute	Carrier flow:	1 mL/minute
Temperature 2:	150°C		
Hold Time 2:	0 minutes		
Ramp 2:	8°C/minute		
Temperature 3:	220°C		
Final Time:	1 minute		

10.1.2 MS Conditions:

Mass range: m/z of 35 – 270 amu
Scan time: 0.5 minutes/scan
Source temperature: 230°C or according to manufacturer's specifications.

10.1.3 Velocity Concentrator Purge and Trap Conditions:

Purge time: 11 minutes
Dry purge: 2 minutes

Desorb preheat: 250°C
Desorb temp: 255°C
Desorb time: 2 minutes

Bake temp: 290°C
Bake time: 10 minutes

10.1.4 Encon Concentrator Purge and Trap Conditions:

Purge time: 11 minutes
Dry purge: 1 minute

Desorb preheat: 245°C
Desorb temp: 255°C
Desorb time: 1 minute

Bake temp: 270°C
Bake time: 10 minutes

10.2 Initial Calibration

10.2.1 The initial calibration is performed at a minimum of five (5) concentration levels listed in Table 4, the low level either at or below the reporting limit. The calibration is performed using instrument conditions listed in 10.1.

BFB must be analyzed prior to analysis of the initial calibration standards and must pass the criteria listed in Table 3. The mass spectrum of BFB should be acquired in the following manner:

- (1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
- (2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of BFB.

This is done automatically with the EnviroQuant software.

10.2.1.1 Low Level/High Level Soil Curve on Centurion: To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 50mL volumetric flask using a micro syringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 5mL syringe with standard and transfer to a 40mL VOA vial containing a magnetic stir bar. Load the vial onto autosampler.

- 10.2.1.2 Aqueous/High Level Soil Curve on Solatek or Centurion:** To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 100mL volumetric flask using a micro syringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, and then gently fill a 40mL VOA vial to the top. Load the vial onto the autosampler.
- 10.2.2** Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in Section 10.1. The same operating conditions are used for calibration and sample analyses. Create the analytical sequence using the HP EnviroQuant data acquisition software.
- Relative Response Factors:** The internal standard calibration technique is used. In each calibration standard, calculate the relative response factor for each analyte and the relative standard deviation (RSD) of the response factors using the EnviroQuant data processing software. The response factors are calculated using the areas of the characteristic (quantitation) ion for each target analyte and internal standard. The calculations are performed automatically using the EnviroQuant software, using the formulae listed in Alpha's Quality Manual.
- 10.2.3 Initial Calibration Criteria:** The following sections outline the method acceptance criteria for an initial calibration curve. All criteria must be met for the calibration to be deemed acceptable, and for sample analysis to proceed. For MCP and CT projects see the related project specific addenda for modifications.
- 10.2.3.1 Relative Standard Deviation Criteria:** If the RSD for each target analyte is less than or equal to 20%, then the response for this compound is considered linear over the calibration range and the mean calibration factor can be used to quantitate sample results. If the 20% RSD criterion is not met for an analyte linear regression may be used if $r^2 \geq 0.990$, weighted linear with a weighting factor of $1/SD^2$ and $r > 0.995$, or quadratic fit if $r^2 \geq 0.990$. A minimum of five points are required for linear fit and six points is required for quadratic fit. The calibration must be repeated for any compounds that fail. If more than 10% of the compounds exceed the 20% RSD limit and do not achieve the minimum correlation coefficient for alternative curve fits, sample analysis cannot proceed.
- 10.2.3.1.1 Percent Relative Error (%RE):** For linear and quadratic fit compounds the %RE relative error must be calculated for the standard at or near the midpoint of the initial calibration and at the lowest level. The %RE between the calculated and expected amounts of an analyte should be $\leq 30\%$ for all standards, otherwise recalibration is necessary.
- 10.2.4 Evaluation of Retention Times:** The relative retention times used for identification of target analytes are ± 0.06 RRT (Relative Retention Time) units, based on the most recent standard run. It has been determined that these limits work well, being wide enough to eliminate false-negative results while being tight enough to eliminate false positive results. Due to the selectivity of the mass spectrometer, compound identification is more definitive than when using a less selective detector.
- 10.2.5 Initial Calibration Verification:** After each calibration and before the analysis of samples, an ICV must be analyzed at or near the midpoint of the curve. The ICV must be prepared using a different source than the Initial Calibration and must contain all target analytes. The percent recoveries must be between 70% and 130% for target analytes

except for "difficult" analytes (Table 7), which must exhibit percent recoveries between 40% and 160%. Corrective action is required if greater than 10% of all analytes are outside the prescribed criteria.

- 10.2.5.1** The ICV used for the initial calibration can be used as the continuing calibration verification. See Section 10.4 for criteria.

10.2.6 Additional considerations for SIM analysis

- 10.2.6.1** SIM may be useful for applications requiring quantitation limits below the normal range of electron impact quadrupole mass spectrometry, and both are allowable options for this method. Using the primary m/z for quantitation and at least one secondary m/z (or product ion) for confirmation, set up the descriptor windows based on their retention times. The selected m/z values should include any mass defect noted in the target analyte mass spectra acquired on the instrument, usually less than 0.2 amu. The dwell time for each ion may be automatically calculated by the instrument software or may be calculated based on the peak widths of the analytes of interest, the number of spectra needed to be acquired across each peak, and the number of concurrent ions that need to be acquired in each segment. When fewer m/z values are monitored in each segment, the acquisition time for each m/z can be increased, thereby increasing the sensitivity of the system. The total cycle time for the MS should be short enough that at least five, but preferably ten or more, spectra are acquired per chromatographic peak.

- 10.2.6.2** When compounds are analyzed in SIM mode, the following best practices are recommended:

10.2.6.2.1 Monitor at least two ions for each target analyte and use the mid-point of the calibration curve to establish proper ion ratios for each compound. The ratios of primary and secondary ions are the only qualitative tools available in SIM runs (other than RT), which increases their importance in proper identification. When interferences are expected or observed in a given matrix, acquiring multiple secondary ions may aid in qualitative identification.

10.2.6.2.2 Verify that all monitored ions are correctly integrated in order to achieve proper ion ratios. Update the primary/secondary ion ratios and reference mass spectra after each ICAL using a mid-range ICAL standard.

10.3 Equipment Operation and Sample Processing

The same GC, MS, and Purge and Trap conditions used for the initial calibration must be employed for sample analysis. After verification of system performance by analysis of BFB, the continuing calibration standard and method blank, samples are analyzed and processed as described below.

- 10.3.1** All samples are initially screened using Tekmar 7000 Headspace Autosamplers.
- 10.3.2** Retrieve sample VOA vials from the sample refrigerator just prior to loading onto the purge and trap system.
- 10.3.3 Aqueous samples:**
Load the VOA vial directly on the sampling rack. Dilutions may be prepared volumetrically and poured into VOA vials ensuring there is no headspace left in the vial. The autosampler will then sample 10mL from the VOA vial.

10.3.4 Low level soil samples:

Low level soil sample should be shaken briefly to ensure that the stir bar is loose, and will spin on the autosampler unit. Take the low level VOA vial and place directly into the rack of the autosampler unit. Surrogate and internal standards are added automatically by the autosampler prior to sample purging.

10.3.5 High level soil samples:

Shake or vortex the sample for 15 seconds, ensuring the methanol has completely penetrated the soil in the vial. Let sample settle prior to taking methanol aliquot.

10.3.5.1 Through liquid path

Load a maximum of 860 μ L or appropriate dilution of the methanol into a half-full VOA vial. Fill the VOA vial up to the top with water and cap with no headspace. Allow the auto-sampler to sample 10mL out of the VOA vial which would be equivalent to injecting 100 μ L of the methanol extract. Prepare dilutions accordingly.

10.3.5.2 Through soil path

Into a VOA vial with a stir bar added, load 4.9mL of water plus a maximum of 100 μ L of methanol or appropriate dilution of methanol extract from a 5mL luerlock syringe. Cap the vial and load onto the auto-sampler.

10.3.6 Qualitative Analysis:

10.3.6.1 The qualitative identification of each compound is based on retention time and on comparison of the sample mass spectrum with the reference mass spectrum. The reference mass spectrum must be generated by the laboratory on the same GC/MS system. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.3.6.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. The EnviroQuant data system is configured to make this check.

10.3.6.1.2 The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.3.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

10.3.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is $\leq 50\%$ of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs (i.e., m and p-xylene).

- 10.3.6.1.5** Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.
- 10.3.6.1.6** Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.
- 10.3.6.2** For samples containing non-target analytes, a library search will be performed at client request. Compound identification will be classified as "tentative", and the concentration will be reported as an estimate as no quantitative standards are run for these compounds.
- 10.3.6.2.1** Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) are present in the sample spectrum.
- 10.3.6.2.2** The relative intensities of the major ions should agree within $\pm 20\%$. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
- 10.3.6.2.3** Molecular ions present in the reference spectrum are present in the sample spectrum. If the molecular ion is not present, carefully review library matches in order to avoid misidentification.
- 10.3.6.2.4** Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- 10.3.6.2.5** Ions present in the reference spectrum but not in the sample spectrum are reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks.
- 10.3.6.2.6** Mass spectral library search algorithms typically assign a match factor to the peak identity based on comparison of an unknown mass spectrum to library spectra. For spectra meeting the above conditions, match factors greater than 0.85 (85%) may be considered confirming evidence. Where a known limitation in data collection is identified (e.g., the presence of an incompletely resolved spectral interference), a lower match factor may be considered confirmatory. For multiple library spectra with similar match factors (e.g., for hydrocarbons with low abundance molecular ions, or structural isomers), the tentative identification assigned to the unknown may be better represented as a more generic structure (e.g., unknown hydrocarbon, C4 benzene structural isomer).

10.3.7 Quantitative Analysis:

- 10.3.7.1** Quantitation of a target compound detected in a sample is performed automatically by the EnviroQuant data processing software, using the formulae found in Alpha's Quality Manual. Either the average response factor or calibration curve will be used for sample quantitation, depending on how the particular analyte was processed in the initial calibration curve. If non-target

compounds are to be reported, the quantitation is performed automatically by the EnviroQuant software using the total area of the compound and the nearest internal standard, and assuming a relative response factor of 1.0.

10.4 Continuing Calibration

Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift. For MCP and CT projects see the related project specific addenda for modifications.

10.4.1 Prior to the analysis of samples or calibration standards, inject or purge 1 μL (50 ng) of the 4-Bromofluorobenzene standard (Section 8.4.4) into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 3 before sample analysis begins.

10.4.2 The initial calibration curve for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing the continuing calibration check standard (Section 8.5.3).

10.4.3 A method blank must be analyzed prior to any samples, typically immediately following the continuing calibration check standard, to ensure that the analytical system is free of contaminants. The method blank must not contain any target analytes at or above the required compound reporting limits.

10.4.4 The percent difference or drift for each target analyte must be less than or equal to 20% (30% for all SIM compounds). If greater than 20% of target analytes exceed the %D criteria corrective action must be taken prior to the analysis of samples. If less than or equal to 20% of compounds exceed the criteria, corrective action is not required.

10.4.5 Internal Standard Retention Time:

The absolute retention times of the internal standards in the calibration verification standard are evaluated after data acquisition. If the absolute retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.6 Internal Standard Response:

If the area for any of the internal standards in the calibration verification standard changes by a factor of two (50% to 200%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.5 Preventive Maintenance

Routine preventive maintenance should be performed on the analytical system. This includes replacement of GC septa and periodic rinsing or replacement of purge and trap tubes and sparge needles. The trap should be replaced every six months or sooner if performance criteria

cannot be met. Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

If system performance deteriorates, additional maintenance may be required. This includes replacement of injector ports and seals, clipping several inches off of the front end of the GC column, or in extreme cases the replacement of the GC column. Flushing or replacement of purge and trap lines may be necessary if they become contaminated or develop active sites.

Perform routine preventative maintenance as described throughout this SOP. Record all maintenance in the instrument logbook.

11. Data Evaluation, Calculations and Reporting

11.1 LIMS Data Corrections

Please note that the Laboratory Information Management System (LIMS) automatically adjusts soil sample results to account for the % Total Solids of the sample (as determined per Alpha SOP/07-38) and the methanol preservation dilution effect.

11.2 Data Calculations

11.2.1 Results of Aqueous Sample Analysis:

$$\text{Concentration (ug/L)} = \frac{(\text{Conc.}) (Vp) (DF)}{(Vs)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.

Vp = Volume purged, 10 mL is standard

Vs = Volume of sample purged

DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".

11.2.2 Results of Sediment/Soil, Sludge, and Waste Analysis:

All solids including soils, sediments, and sludges must be reported on a dry-weight basis.

11.2.2.1 Low-Level Samples:

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

11.2.2.2 High-Level Samples:

$$\text{Concentration (ug/Kg)} = \frac{(\text{Conc.}) (V_p) (5000) (DF)}{(W) (V_e) (\%S)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
Ve = Extract volume, mL
Vp = Volume purged, 5 mL is standard
W = Aliquot of sample (wet), g
%S = Sample % solid
5000 = Constant representing the final volume of the methanol extraction.

11.2.2.2.1 High-Level Samples Corrected for Total Water/Solvent Mixture (V_t):

Samples that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the water/solvent mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture calculation.

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

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

The calculated V_t value is now added to the volume of methanol in the sample (typically 5000µL), and the corrected concentration is calculated using the equation below:

$$\text{Corrected concentration (mg/Kg)} = \frac{(\text{Conc.}) (V_t + \text{methanol vol.}) (V_p) (DF)}{(W) (V_e) (\%S)}$$

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

All batch and sample specific QC criteria outlined in section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan
SOP/1732 DL/LOD/LOQ Generation
SOP/1739 IDC/DOC Generation
SOP/1728 Waste Management and Disposal SOP

16. Attachments

Table 1: 8260 REPORTING LIMITS
Table 2: 8260 QC ACCEPTANCE CRITERIA
Table 3: BFB TUNING CRITERIA
Table 4: STANDARD SOLUTIONS and ICAL Levels
Table 5: 8260D Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation
Table 6: 8260D Quantitation Ions
Table 7: Difficult Analytes
Table 8: ICAL Low Point re-quantitation ranges

Table 1
Standard Reported Detection Limits
US EPA METHOD 8260D and 5035A/8260D

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL (µg/KG) ⁽¹⁾	RDL (µg/KG) ⁽²⁾
Acetone ^(3,4,5)	0.01	5.0	25	1250
Acrolein ⁽⁵⁾		5.0	25	1250
Acrylonitrile ^(3,4)		5.0	4	200
Allyl Chloride ⁽⁷⁾		N/A	5	250
Benzene ^(3,4,5)	0.2	0.5	0.5	25
Bromobenzene ^(3,4)		2.5	2	100
Bromochloromethane ^(3,4,5)	0.1	2.5	2	100
Bromodichloromethane ^(3,4,5)	0.300	0.5	0.5	25
Bromoform ^(3,4,5)	0.100	2.0	4	200
Bromomethane ^(3,4,5)	0.01	1.0	2	100
2-Butanone ^(3,4,5)	0.01	5.0	10	500
Butyl acetate ⁽⁷⁾		N/A	5	50
n-Butylbenzene ^(3,4)		0.5	1	50
sec-Butylbenzene ^(3,4)		0.5	1	50
tert-Butylbenzene ^(3,4)		2.5	2	100
Carbon disulfide ^(3,4,5)	0.100	5.0	10	500
Carbon tetrachloride ^(3,4,5)	0.100	0.5	1	50
Chlorobenzene ^(3,4,5)	0.400	0.5	0.5	25
Chlorodifluoromethane ⁽⁷⁾		N/A	5	250
Chloroethane ^(3,4,5)	0.01	1.0	2	100
2-Chloroethylvinyl ether ⁽³⁾		10.0	20	1000
Chloroform ^(3,4,5)	0.300	0.75	1.5	75
Chloromethane ^(3,4,5)	0.01	2.5	4	200
o-Chlorotoluene ^(3,4)		2.5	2	100
Cyclohexane ⁽⁵⁾	0.01	10	10	500
Cyclohexanone		10	20	1000
p-Chlorotoluene ^(3,4)		2.5	2	100
cis-Decahydronaphthalene ⁽⁷⁾		N/A	5	250
trans-Decahydronaphthalene ⁽⁷⁾		N/A	5	250
n-Decane ⁽⁷⁾		N/A	5	250
Dibromochloromethane ^(3,4,5)	0.200	0.5	1	50
1,2-Dibromo-3-chloropropane ^(3,4,5)	0.01	2.5	3	150
1,2-Dibromoethane ^(3,4,5)	0.200	2.0	1	50
Dibromomethane ^(3,4)		5.0	2	100
1,2-Dichlorobenzene ^(3,4,5)	0.600	2.5	2	100
1,3-Dichlorobenzene ^(3,4,5)	0.500	2.5	2	100
1,4-Dichlorobenzene ^(3,4,5)	0.600	2.5	2	100
1,4-Dichlorobutane ^(3,4)		5.0	10	500
trans-1,4-Dichloro-2-butene ^(3,4)		2.5	5	250

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Table 1 (continued)
Standard Reported Detection Limits
US EPA METHOD 8260D and 5035A/8260D

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG) ⁽¹⁾	RDL (µg/KG) ⁽²⁾
Dichlorodifluoromethane ^(3,4,5)	0.01	5.0	10	500
1,1-Dichloroethane ^(3,4,5)	0.300	0.75	1	50
1,2-Dichloroethane ^(3,4,5)	0.07	0.5	1	50
1,1-Dichloroethene ^(3,4,5)	0.06	0.5	1	50
cis-1,2-Dichloroethene ^(3,4,5)	0.200	0.5	1	50
trans-1,2-Dichloroethene ^(3,4,5)	0.100	0.75	1.5	75
1,2-Dichloropropane ^(3,4,5)	0.200	1.75	1	50
1,3-Dichloropropane ^(3,4)		2.5	2	100
2,2-Dichloropropane ^(3,4)		2.5	2	100
1,1-Dichloropropene ^(3,4)		2.5	0.5	25
cis-1,3-Dichloropropene ^(3,4,5)	0.300	0.5	0.5	25
p-Diethylbenzene ⁽⁴⁾		2.0	2	100
Diisopropyl Ether ⁽⁶⁾		2.0	2	100
1,4-Dioxane ⁽⁵⁾ (non-SIM)		250.0	80	4000
trans-1,3-Dichloropropene ^(3,4,5)	0.300	0.5	1	50
Ethanol ⁽⁷⁾		250.0	1000	50000
Ethyl acetate		10.0	10	500
Ethylbenzene ^(3,4,5)	0.400	0.5	1	50
Ethyl ether ^(3,4)		2.5	2	100
4-Ethyltoluene ⁽⁴⁾		2.0	2	100
Ethyl methacrylate ^(3,4)		5.0	10	500
Ethyl-Tert-Butyl-Ether ⁽⁶⁾		2.0	2	100
Freon-113 ⁽⁵⁾	0.05	10.0	4	200
n-Heptane ⁽⁷⁾		N/A	5	250
Hexachlorobutadiene ^(3,4)		0.5	4	200
Hexachloroethane ⁽⁷⁾		N/A	5	250
Hexane		1.0	5	250
2-Hexanone ^(3,4,5)	0.01	5.0	10	500
Iodomethane		5.0	10	500
Isopropyl Alcohol (IPA)		100.0	100	5000
Isopropylbenzene ^(3,4,5)	0.400	0.5	1	50
p-Isopropyltoluene ^(3,4)		0.5	1	50
Limonene ⁽⁷⁾		N/A	5	250
Methyl Acetate ⁽⁵⁾	0.01	10.0	4	200
Methylene chloride ^(3,4,5)	0.01	3.0	5	250
Methyl Cyclohexane ⁽⁵⁾	0.05	10.0	4	200
Methyl isothiocyanate ⁽⁷⁾		N/A	5	250
Methyl Methacrylate		2.5	5	250
4-Methyl-2-pentanone ^(3,4,5)	0.03	5.0	10	500

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Methyl-tert-butyl-ether ^(3,4,5)	0.100	1.0	2	100
Naphthalene ^(3,4)		2.5	4	200
Nitrobenzene ⁽⁷⁾		N/A	5	250
2-Nitropropane ⁽⁷⁾		N/A	5	250
n-Nonane ⁽⁷⁾		N/A	5	250
n-Octane ⁽⁷⁾		N/A	5	250
n-Butanol ⁽⁵⁾		100.0	300	15000
n-Propylbenzene ^(3,4)		0.5	1	50
n-Propyl bromide		5.0	N/A	N/A
Pentachloroethane		2.0	N/A	N/A
Styrene ^(3,4,5)	0.200	1.0	1	50
Tert-Butyl Alcohol ⁽⁵⁾		10.0	20	1000
Tertiary-Amyl Methyl Ether ⁽⁶⁾		2.0	2	100
1,1,1,2-Tetrachloroethane ^(3,4)		0.5	0.5	25
1,2,4,5-Tetramethylbenzene ⁽⁴⁾		2.0	2	100
1,1,2,2-Tetrachloroethane ^(3,4,5)	0.200	0.5	0.5	25
Tetrachloroethene ^(3,4,5)	0.100	0.5	0.5	25
Tetrahydrofuran ⁽³⁾		5.0	4	200
Toluene ^(3,4,5)	0.300	0.75	1	50
1,2,3-Trichlorobenzene ^(3,4,5)	0.400	2.5	2	100
1,2,4-Trichlorobenzene ^(3,4,5)	0.400	2.5	2	100
1,3,5-Trichlorobenzene ⁽⁶⁾		2.0	2	100
1,1,1-Trichloroethane ^(3,4,5)	0.05	0.5	0.5	25
1,1,2-Trichloroethane ^(3,4,5)	0.200	0.75	1	50
Trichloroethene ^(3,4,5)	0.200	0.5	0.5	25
Trichlorofluoromethane ^(3,4,5)	0.01	2.5	4	200
1,2,3-Trichloropropane ^(3,4)		5.0	10	500
1,2,4-Trimethylbenzene ^(3,4)		2.5	2	100
1,3,5-Trimethylbenzene ^(3,4)		2.5	2	100
n-Undecane ⁽⁷⁾		N/A	5	250
Vinyl acetate ^(3,4)		5.0	10	500
Vinyl chloride ^(3,4,5)	0.01	1.0	1	50
m/p-Xylenes ^(3,4,5)	0.200	1.0	2	100
o-Xylene ^(3,4,5)	0.200	1.0	1	50
Isobutyl Alcohol		10.0	N/A	N/A
2-Butanol		25.0	N/A	N/A
4-penten-2-ol		100.0	N/A	N/A
2-methyl-2-butanol		25.0	N/A	N/A
4-methyl-2-pentanol		25.0	N/A	N/A
Chloropicrin		20.0	100	5000
Halothane		2.5	10	500
1,4-Dioxane ⁽⁵⁾ SIM		3.0	N/A	N/A
1,1,2,2-Tetrachloroethane SIM		0.1	N/A	N/A

(1) Detection Limits are for Low-level Aqueous preserved samples.

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- (2) Detection Limits are for High-level Methanol preserved samples.
- (3) Analyte reported by standard 8260 reporting list.
- (4) Analyte reported by New York TCL reporting list.
- (5) Analyte reported by New Jersey TCL reporting list.
- (6) Analyte reported for New Hampshire in addition to standard 8260 reporting list.
- (7) Analyte only reported for New York TCL report upon client request.
- (8) Note: Reporting Limits are based on standard 8260 reporting list, RL's may vary for NY and NJ reporting lists.

Table 2

QUALITY CONTROL ACCEPTANCE CRITERIA

Surrogate Spike Percent Recovery	Aqueous Limits		Soil Limits	
	Lower Control Limit	Upper Control Limit	Lower Control Limit	Upper Control Limit
1,2-Dichloroethane-d ₄	70%	130%	70%	130%
4-Bromofluorobenzene	70%	130%	70%	130%
Toluene-d ₈	70%	130%	70%	130%
Dibromofluoromethane	70%	130%	70%	130%

Table 3

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95 (not required)
75	30 to 60% of m/z 95 (not required)
95	50 to 200% of m/z 174
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	50 to 200% of m/z 95
175	5 to 9% of m/z 174
176	95% to 105% of m/z 174
177	5 to 10% of m/z 176

Table 4

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Soil	Stock (ug/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Fluorobenzene	2500	20	20	20	20	20	20	20	20	20	20
Dichlorodifluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorodifluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Vinyl chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Bromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Trichlorofluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Ethanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
1,1-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
Carbon disulfide	2000	0.5	1	2	4	20	40	60	100	200	300
Freon-113	2000	0.5	1	2	4	20	40	60	100	200	300
Iodomethane	2000	0.5	1	2	4	20	40	60	100	200	300
Acrolein	2000	0.5	1	2	4	20	40	60	100	200	300
Allyl chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Methylene chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Isopropyl alcohol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Acetone	2000	0.5	1	2	4	20	40	60	100	200	300
trans-1,2-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
Hexane	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl tert-butyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
tert-Butyl alcohol	2000	2.5	5	10	20	100	200	300	500	1000	1500
Diisopropyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
1,1-Dichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Halothane	2000	0.5	1	2	4	20	40	60	100	200	300
Acrylonitrile	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl tert-butyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Vinyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
cis-1,2-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
2,2-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
Bromochloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Cyclohexane	2000	0.5	1	2	4	20	40	60	100	200	300

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Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Chloroform	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
Carbon tetrachloride	2000	0.5	1	2	4	20	40	60	100	200	300
Tetrahydrofuran	2000	0.5	1	2	4	20	40	60	100	200	300
Dibromofluoromethane	2500	20	20	20	20	20	20	20	20	20	20
1,1,1-Trichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
2-Butanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
2-Butanone	2000	0.5	1	2	4	20	40	60	100	200	300
1,1-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Heptane	2000	0.5	1	2	4	20	40	60	100	200	300
Benzene	2000	0.5	1	2	4	20	40	60	100	200	300
tert-Amyl methyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichloroethane-d4	2500	20	20	20	20	20	20	20	20	20	20
1,2-Dichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Isobutyl alcohol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
2-Methyl-2-butanol	2000	2.5	5	10	20	100	200	300	500	1000	1500
Methyl cyclohexane	2000	0.5	1	2	4	20	40	60	100	200	300
Trichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Dibromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
4-penten-2-ol	2000	2.5	5	10	20	100	200	300	500	1000	1500
2-Chloroethyl vinyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Bromodichloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl acrylate	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dioxane	2000	N/A	40	80	200	1000	2000	3000	5000	10000	15000
cis-1,3-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorobenzene-d5	2500	20	20	20	20	20	20	20	20	20	20
Octane	2000	0.5	1	2	4	20	40	60	100	200	300
Toluene-d8	2500	20	20	20	20	20	20	20	20	20	20
Toluene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Methyl-2-pentanone	2000	0.5	1	2	4	20	40	60	100	200	300
Tetrachloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
2-Nitropropane	2000	0.5	1	2	4	20	40	60	100	200	300

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Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Chloropicrin	2000	N/A	50	N/A	100	200	300	400	500	600	700
trans-1,3-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl isothiocyanate	2000	0.5	1	2	4	20	40	60	100	200	300
4-Methyl-2-pentanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Ethyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,2-Trichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorodibromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dibromoethane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butyl Acetate	2000	0.5	1	2	4	20	40	60	100	200	300
2-Hexanone	2000	0.5	1	2	4	20	40	60	100	200	300
Nonane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Ethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,1,2-Tetrachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
p/m Xylene	2000	1	2	4	8	40	80	120	200	400	600
o Xylene	2000	1	2	4	8	40	80	120	200	400	600
Styrene	2000	1	2	4	8	40	80	120	200	400	600
1,4-Dichlorobenzene-d4	2500	20	20	20	20	20	20	20	20	20	20
Bromoform	2000	0.5	1	2	4	20	40	60	100	200	300
Butyl acrylate	2000	0.5	1	2	4	20	40	60	100	200	300
Isopropylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Bromofluorobenzene	2500	20	20	20	20	20	20	20	20	20	20
Bromobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Decane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Propylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dichlorobutane	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,2,2-Tetrachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
4-Ethyltoluene	2000	0.5	1	2	4	20	40	60	100	200	300
2-Chlorotoluene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3,5-Trimethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,3-Trichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
trans-1,4-Dichloro-2-butene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Chlorotoluene	2000	0.5	1	2	4	20	40	60	100	200	300

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
tert-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Pentachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4-Trimethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Limonene	2000	0.5	1	2	4	20	40	60	100	200	300
sec-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
p-Isopropyltoluene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
trans-Decahydronaphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
Undecane	2000	0.5	1	2	4	20	40	60	100	200	300
p-Diethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Hexachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
cis-Decahydronaphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4,5-Tetramethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dibromo-3-chloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
1,3,5-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Nitrobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Hexachlorobutadiene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Naphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,3-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dioxolane	2000	N/A	25	N/A	50	100	250	500	1000	1500	2000

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Stock Standard Concentrations and Suggested Calibration Concentration Levels

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
Fluorobenzene	2500	10	10	10	10	10	10	10	10	10	10	10
Dichlorodifluoromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Vinyl chloride	2000	0.19	0.5	2	10	30	50	80	100	120	160	200
Bromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Trichlorofluoromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Ethanol	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Carbon disulfide	2000		0.5	2	10	30	50	80	100	120	160	200
Freon-113	2000		0.5	2	10	30	50	80	100	120	160	200
Iodomethane	2000		0.5	2	10	30	50	80	100	120	160	200
Acrolein	2000		0.5	2	10	30	50	80	100	120	160	200
Methylene chloride	2000		0.5	2	10	30	50	80	100	120	160	200
Isopropyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
Acetone	2000		0.5	2	10	30	50	80	100	120	160	200
trans-1,2-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Methyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200
Methyl tert-butyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
tert-Butyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
Diisopropyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Halothane	2000		0.5	2	10	30	50	80	100	120	160	200
Acrylonitrile	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl tert-butyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Vinyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200
cis-1,2-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
2,2-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
Bromochloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Cyclohexane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloroform	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200

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Water	Stock (ug/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5	Level 6	Level 7	Level 8	Level 9	Level 10
							(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)
							Optional		Optional		Optional	
Carbon tetrachloride	2000	0.19	0.5	2	10	30	50	80	100	120	160	200
Tetrahydrofuran	2000		0.5	2	10	30	50	80	100	120	160	200
Dibromofluoromethane	2500	10	10	10	10	10	10	10	10	10	10	10
1,1,1-Trichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
2-Butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Butanone	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
Benzene	2000	0.19	0.5	2	10	30	50	80	100	120	160	200
tert-Amyl methyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichloroethane-d4	2500	10	10	10	10	10	10	10	10	10	10	10
1,2-Dichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Isobutyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Methyl-2-butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
Methyl cyclohexane	2000		0.5	2	10	30	50	80	100	120	160	200
Trichloroethene	2000	0.19	0.5	2	10	30	50	80	100	120	160	200
n-Butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
Dibromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
4-penten-2-ol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Chloroethyl vinyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Bromodichloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl acrylate	2000		0.25	1	5	15	25	40	50	60	80	100
Methyl methacrylate	2000		0.25	1	5	15	25	40	50	60	80	100
1,4-Dioxane	2000		100	400	500	600	1000	800	1000	1200	1600	2000
cis-1,3-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorobenzene-d5	2500	10	10	10	10	10	10	10	10	10	10	10
Toluene-d8	2500	10	10	10	10	10	10	10	10	10	10	10
Toluene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Methyl-2-pentanone	2000		0.5	2	10	30	50	80	100	120	160	200
Tetrachloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Chloropicrin	2000		30	50	80	120	200	160	400	200	320	400
trans-1,3-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Methyl-2-pentanol	2000		2.5	10	50	150	250	400	500	600	800	1000

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Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
Ethyl methacrylate	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,2-Trichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorodibromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dibromoethane	2000		0.5	2	10	30	50	80	100	120	160	200
2-Hexanone	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Ethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,1,2-Tetrachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
p/m Xylene	2000		1	4	20	60	100	160	200	240	320	400
o Xylene	2000		1	4	20	60	100	160	200	240	320	400
Styrene	2000		1	4	20	60	100	160	200	240	320	400
1,4-Dichlorobenzene-d4	2500	10	10	10	10	10	10	10	10	10	10	10
Bromoform	2000		0.5	2	10	30	50	80	100	120	160	200
Butyl acrylate	2000		0.25	1	5	15	25	40	50	60	80	100
Isopropylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Bromofluorobenzene	2500	10	10	10	10	10	10	10	10	10	10	10
Bromobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
n-Propylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dichlorobutane	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,2,2-Tetrachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
4-Ethyltoluene	2000		0.5	2	10	30	50	80	100	120	160	200
2-Chlorotoluene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3,5-Trimethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,3-Trichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
trans-1,4-Dichloro-2-butene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Chlorotoluene	2000		0.5	2	10	30	50	80	100	120	160	200
tert-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Pentachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
n-Butyl methacrylate	2000		0.25	1	5	15	25	40	50	60	80	100
1,2,4-Trimethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
sec-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200

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Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
p-Isopropyltoluene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
p-Diethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
n-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,4,5-Tetramethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dibromo-3-chloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
1,3,5-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Hexachlorobutadiene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,4-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Naphthalene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,3-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dioxolane	2000		10	40	100	250	N/A	500	N/A	750	N/A	1000
Pentachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dioxane (SIM)	100		0.5	2	10	20	30	50	100	200	N/A	N/A
1,1,2,2-Tetrachloroethane (SIM)	100		0.05	0.1	0.2	0.5	1	2	5	10	N/A	N/A

- For Low Level Soil analysis, the calibration levels are the same in µg/Kg units.
- For High Level Soil analysis, the calibration levels are at 50x the levels listed due to sample preparation requirements.

Table 5
8260D Volatile Internal Standards
with Corresponding Target Compounds
and Surrogates Assigned for Quantitation

Fluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
Dichlorodifluoromethane	Toluene-d8 (surr)	Isopropylbenzene
Chloromethane	Toluene	Bromoform
Vinyl Chloride	Ethyl Methacrylate	1,4-dichloro-2-butane
Bromomethane	Trans-1,3-dichloropropene	1,1,2,2,-tetrachloroethane
Chloroethane	1,1,2-trichloroethane	4-bromofluorobenzene (surr)
Trichlorofluoromethane	2-hexanone	1,2,3-trichloropropane
Ethyl Ether	1,3-dichloropropane	trans-1,4-dichloro-2-butene
Freon 113	Tetrachloroethene	n-propylbenzene
Acrolein	Chlorodibromomethane	Bromobenzene
Acetone	1,2-dibromoethane	4-ethyltoluene
Ethanol	Chlorobenzene	1,3,5-trimethylbenzene
1,1,-dichloroethene	1,1,1,2-tetrachloroethane	2-chlorotoluene
Tert-Butyl Alcohol	Ethylbenzene	4-chorotoluene
Methyl Acetate	p/m xylene	tert-butylbenzene
Carbon Disulfide	o xylene	1,2,4-trimethylbenzene
Methylene Chloride	Styrene	sec-butylbenzene
Acrylonitrile	Octane	p-isopropyltoluene
Methyl Tert Butyl Ether	2-Nitropropane	1,3-dichlorobenzene
Halothane	Methyl isothiocyanate	1,4-dichlorobenzene
Trans-1,2-dichloroethene	n-Butyl acetate	n-butylbenzene
Diisopropyl Ether	Nonane	p-diethylbenzene
Vinyl Acetate		1,2-dichlorobenzene
1,1-dichloroethane		1,2,4,5-tetramethylbenzene
Ethyl-Tert-Butyl-Ether		1,2-dibromo-3-chloropropane
2-butanone		1,3,5-trichlorobenzene
2,2-dichloropropane		1,2,4-trichlorobenzene
Cis-1,2-dichloroethene		Hexachlorobutadiene
Chloroform		Naphthalene
Bromochloromethane		1,2,3-trichlorobenzene
Tetrahydrofuran		Cyclohexanone
Dibromofluoromethane (surr)		Nitrobenzene

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1,1,1-trichloroethane		Pentachloroethane
Cyclohexane		Decane
1,1-dichloropropene		Limonene
carbon Tetrachloride		trans-Decahydronaphthalene
Tertiary-Amyl Methyl Ether		Undecane
1,2-dichloroethane-d4 (surr)		Hexachloroethane
1,2-dichloroethane		cis-Decahydronaphthalene
Benzene		
Trichloroethene		
Methyl cyclohexane		
1,2-Dichloropropane		
Bromodichloromethane		
1,4-Dioxane		
Dibromomethane		
2-Chloroethylvinyl Ether		
4-methyl-2-pentanone		
cis-1,3-Dichloropropene		
Iodomethane		
Methyl methacrylate		
n-Butanol		
Ethyl acetate		
Isopropyl alcohol (IPA)		
Hexane		
n-Propyl bromide		
Chlorodifluoromethane		
Allyl chloride		
Heptane		

Table 6
8260D Quantitation Ions

Analyte	Quantitation Ion	Analyte	Quantitation Ion
Dichlorodifluoromethane	85	Ethyl Methacrylate	69
Chloromethane	50	Trans-1,3-dichloropropene	75
Vinyl Chloride	62	1,1,2-trichloroethane	83
Bromomethane	94	2-hexanone	43
Chloroethane	64	1,3-dichloropropane	76
Trichlorofluoromethane	101	Tetrachloroethene	166
Ethyl Ether	74	Chlorodibromomethane	129
Freon 113	101	1,2-dibromoethane	107
Acrolein	56	Chlorobenzene	112
Acetone	43	1,1,1,2-tetrachloroethane	131
1,1,-dichloroethene	96	Ethylbenzene	91
Tert-Butyl Alcohol	59	p/m xylene	106
Methyl Acetate	43	o xylene	106
Carbon Disulfide	84	Styrene	104
Methylene Chloride	76	Isopropylbenzene	105
Acrylonitrile	53	Bromoform	173
Methyl Tert Butyl Ether	73	1,4-dichloro-2-butane	55
Halothane	117	1,1,2,2,-tetrachloroethane	83
Trans-1,2-dichloroethene	96	1,2,3-trichloropropane	75
Diisopropyl Ether	45	Trans-1,4-dichloro-2-butene	53
Vinyl Acetate	43	n-propylbenzene	91
1,1-dichloroethane	63	Bromobenzene	156
Ethyl-Tert-Butyl-Ether	59	4-ethyltoluene	105
2-butanone	43	1,3,5-trimethylbenzene	105
2,2-dichloropropane	77	2-chlorotoluene	91
Cis-1,2-dichloroethene	96	4-chlorotoluene	91
Chloroform	83	tert-butylbenzene	119
Bromochloromethane	128	1,2,4-trimethylbenzene	105
Tetrahydrofuran	42	sec-butylbenzene	105
1,1,1-trichloroethane	97	p-isopropyltoluene	119
Cyclohexane	56	1,3-dichlorobenzene	146
1,1-dichloropropene	75	1,4-dichlorobenzene	146
Carbon Tetrachloride	117	n-butylbenzene	91
Tertiary-Amyl Methyl Ether	73	p-diethylbenzene	119
1,2-dichloroethane	62	1,2-dichlorobenzene	146
Benzene	78	1,2,4,5-Tetramethylbenzene	119
Trichloroethene	95	1,2-Dibromo-3-chloropropane	75
Methyl Cyclohexane	83	1,3,5-Trichlorobenzene	180
1,2-Dichloropropane	63	1,2,4-Trichlorobenzene	180
Bromodichloromethane	83	Hexachlorobutadiene	225
1,4-dioxane	88	Naphthalene	128
Dibromomethane	93	1,2,3-trichlorobenzene	180
2-Chloroethylvinyl Ether	63	Ethanol	45
4-methyl-2-pentanone	58	Cyclohexanone	55
Cis-1,3-dichloropropene	75	Ethyl acetate	43

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Table 6
8260D Quantitation Ions (continued)

Analyte	Quantitation Ion	Analyte	Quantitation Ion
Toluene	92	Iodomethane	142
Methyl methacrylate	69	n-Butanol	56
Pentachloroethane	167	Isopropyl Alcohol (IPA)	45
Hexane	57	n-Propyl bromide	43
Chlorodifluoromethane	51	Iodomethane	142
Allyl chloride	76	Heptane	71
Octane	85	2-Nitropropane	41
Methyl isothiocyanate	73	n-Butyl Acetate	43
Nonane	57	Decane	57
Limonene	68	Undecane	57
trans-Decahydronaphthalene	138	cis-Decahydronaphthalene	138
Hexachloroethane	117	Nitrobenzene	77

Table 7

List of 8260 Difficult Analytes:

- 1,1,2,2-Tetrachloroethane
- 1,2-Dibromo-3-chloropropane (DBCP)
- 1,4-Dioxane
- 2-Butanone
- 2-chloroethylvinyl ether
- 2-Hexanone
- 2,2-dichloropropane
- 4-Methyl-2-pentanone
- Acetone
- Bromoform
- Bromomethane
- Carbon disulfide
- Chloroethane
- Chloromethane
- cis-1,3-Dichloropropene
- Dichlorodifluoromethane (Freon 12)
- Ethanol
- Iodomethane
- Isobutyl Alcohol
- Naphthalene
- Nitrobenzene
- n-butanol
- Styrene
- Tert-Butyl Alcohol
- Trichlorofluoromethane (Freon 11)
- Isopropyl Alcohol (IPA)

Table 8

ICAL Low Point re-quantitation ranges

Water	Level 11 (ug/L)	L11 (70% - 130%)	Level 1 (ug/L)	L1 (70% - 130%)	Level 2 (ug/L)	L2 (70% - 130%)	Level 3 (ug/L)	L3 (70% - 130%)
Dichlorodifluoromethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Chloromethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Vinyl chloride	0.19	0.133 - 0.247	0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Bromomethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Chloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Trichlorofluoromethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethanol			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1-Dichloroethene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Carbon disulfide			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Freon-113			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Iodomethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Acrolein			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Methylene chloride			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Isopropyl alcohol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65

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Water	Level 11 (ug/L)	L11 (70% - 130%)	Level 1 (ug/L)	L1 (70% - 130%)	Level 2 (ug/L)	L2 (70% - 130%)	Level 3 (ug/L)	L3 (70% - 130%)
Acetone			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
trans-1,2-Dichloroethene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Methyl acetate			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Methyl tert-butyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
tert-Butyl alcohol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
Diisopropyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1-Dichloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Halothane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Acrylonitrile			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethyl tert-butyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Vinyl acetate			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
cis-1,2-Dichloroethene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
2,2-Dichloropropane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Bromochloromethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Cyclohexane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Chloroform			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethyl acetate			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Carbon tetrachloride	0.19	0.133 - 0.247	0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Tetrahydrofuran			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1,1-Trichloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
2-Butanol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
2-Butanone			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1-Dichloropropene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Benzene	0.19	0.133 - 0.247	0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
tert-Amyl methyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2-Dichloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Isobutyl alcohol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
2-Methyl-2-butanol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
Methyl cyclohexane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Trichloroethene	0.19	0.133 - 0.247	0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
n-Butanol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
Dibromomethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2-Dichloropropane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
4-penten-2-ol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
2-Chloroethyl vinyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Bromodichloromethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethyl acrylate			0.25	0.175 - 0.325	1	0.7 - 1.3	5	3.5 - 6.5
Methyl methacrylate			0.25	0.175 - 0.325	1	0.7 - 1.3	5	3.5 - 6.5
1,4-Dioxane			100	70 - 130	400	280 - 520	500	350 - 650
cis-1,3-Dichloropropene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Toluene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
4-Methyl-2-pentanone			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Tetrachloroethene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0

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Water	Level 11 (ug/L)	L11 (70% - 130%)	Level 1 (ug/L)	L1 (70% - 130%)	Level 2 (ug/L)	L2 (70% - 130%)	Level 3 (ug/L)	L3 (70% - 130%)
Chloropicrin			30	21 - 39	50	35 - 65	80	56 - 104
trans-1,3-Dichloropropene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
4-Methyl-2-pentanol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
Ethyl methacrylate			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1,2-Trichloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Chlorodibromomethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,3-Dichloropropane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2-Dibromoethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
2-Hexanone			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Chlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1,1,2-Tetrachloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
p/m Xylene			1	0.7 - 1.3	4	2.8 - 5.2	20	14 - 26
o Xylene			1	0.7 - 1.3	4	2.8 - 5.2	20	14 - 26
Styrene			1	0.7 - 1.3	4	2.8 - 5.2	20	14 - 26
Bromoform			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Butyl acrylate			0.25	0.175 - 0.325	1	0.7 - 1.3	5	3.5 - 6.5
Isopropylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Bromobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
n-Propylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,4-Dichlorobutane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1,1,2,2-Tetrachloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
4-Ethyltoluene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
2-Chlorotoluene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,3,5-Trimethylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2,3-Trichloropropane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
trans-1,4-Dichloro-2-butene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
4-Chlorotoluene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
tert-Butylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Pentachloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
n-Butyl methacrylate			0.25	0.175 - 0.325	1	0.7 - 1.3	5	3.5 - 6.5
1,2,4-Trimethylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
sec-Butylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
p-Isopropyltoluene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,3-Dichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,4-Dichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
p-Diethylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
n-Butylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2-Dichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2,4,5-Tetramethylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2-Dibromo-3-chloropropane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,3,5-Trichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Hexachlorobutadiene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2,4-Trichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0

Naphthalene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2,3-Trichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,3-Dioxolane			10	7 - 13.0	40	28 - 52	100	70 - 130
Pentachloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,4-Dioxane (SIM)			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1,2,2-Tetrachloroethane (SIM)			0.05	0.035 - 0.065	0.1	0.07 - 0.13	0.2	0.14 - 0.26

ICAL Low Point re-quantitation ranges

Soil	Level 0 (ug/kg)	L0 (70% - 130%)	Level 1 (ug/kg)	L1 (70% - 130%)	Level 1.5 (ug/kg)	L1.5 (70% - 130%)	Level 2 (ug/kg)	L2 (70% - 130%)	Level 3 (ug/kg)	L3 (70% - 130%)
Dichlorodifluoromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chlorodifluoromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chloromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Vinyl chloride	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Bromomethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Trichlorofluoromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethanol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 - 104	200	140 - 260
1,1-Dichloroethene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Carbon disulfide	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Freon-113	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Iodomethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Acrolein	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Allyl chloride	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Methylene chloride	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Isopropyl alcohol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 - 104	200	140 - 260
Acetone	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
trans-1,2-Dichloroethene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Methyl acetate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Hexane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26

Soil	Level 0 (ug/kg)	L0 (70% - 130%)	Level 1 (ug/kg)	L1 (70% - 130%)	Level 1.5 (ug/kg)	L1.5 (70% - 130%)	Level 2 (ug/kg)	L2 (70% - 130%)	Level 3 (ug/kg)	L3 (70% - 130%)
Methyl tert-butyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
tert-Butyl alcohol	2.5	1.75 - 3.25	5	3.5 - 6.5	10	7.0 - 13.	20	14 - 26	100	70 - 130
Diisopropyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1-Dichloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Halothane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Acrylonitrile	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethyl tert-butyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Vinyl acetate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
cis-1,2-Dichloroethene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
2,2-Dichloropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Bromochloromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Cyclohexane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chloroform	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethyl acetate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Carbon tetrachloride	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Tetrahydrofuran	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1,1-Trichloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
2-Butanol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 - 104	200	140 - 260
2-Butanone	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1-Dichloropropene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Heptane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Benzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
tert-Amyl methyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2-Dichloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Isobutyl alcohol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 - 104	200	140 - 260
2-Methyl-2-butanol	2.5	1.75 - 3.25	5	3.5 - 6.5	10	7	20	14 - 26	100	70 - 130
Methyl cyclohexane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26

Soil	Level 0 (ug/kg)	L0 (70% - 130%)	Level 1 (ug/kg)	L1 (70% - 130%)	Level 1.5 (ug/kg)	L1.5 (70% - 130%)	Level 2 (ug/kg)	L2 (70% - 130%)	Level 3 (ug/kg)	L3 (70% - 130%)
Trichloroethene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
n-Butanol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 - 104	200	140 - 260
Dibromomethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2-Dichloropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
4-penten-2-ol	2.5	1.75 - 3.25	5	3.5 - 6.5	10	7	20	14 - 26	100	70 - 130
2-Chloroethyl vinyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Bromodichloromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethyl acrylate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Methyl methacrylate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,4-Dioxane	N/A	N/A	40	28 - 52	80	56 - 104	200	140 - 260	1000	700 - 1300
cis-1,3-Dichloropropene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Octane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Toluene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
4-Methyl-2-pentanone	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Tetrachloroethene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
2-Nitropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chloropicrin	N/A	N/A	50	35 - 65	N/A	N/A	100	70 - 130	200	140 - 260
trans-1,3-Dichloropropene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Methyl isothiocyanate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
4-Methyl-2-pentanol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 - 104	200	140 - 260
Ethyl methacrylate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1,2-Trichloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chlorodibromomethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,3-Dichloropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2-Dibromoethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
n-Butyl Acetate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26

Soil	Level 0 (ug/kg)	L0 (70% - 130%)	Level 1 (ug/kg)	L1 (70% - 130%)	Level 1.5 (ug/kg)	L1.5 (70% - 130%)	Level 2 (ug/kg)	L2 (70% - 130%)	Level 3 (ug/kg)	L3 (70% - 130%)
2-Hexanone	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Nonane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1,1,2-Tetrachloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
p/m Xylene	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	8	5.6 - 10.4	40	28 - 52
o Xylene	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	8	5.6 - 10.4	40	28 - 52
Styrene	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	8	5.6 - 10.4	40	28 - 52
Bromoform	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Butyl acrylate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Isopropylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Bromobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Decane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
n-Propylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,4-Dichlorobutane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1,2,2-Tetrachloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
4-Ethyltoluene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
2-Chlorotoluene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,3,5-Trimethylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2,3-Trichloropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
trans-1,4-Dichloro-2-butene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
4-Chlorotoluene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
tert-Butylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Pentachloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
n-Butyl methacrylate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2,4-Trimethylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26

Soil	Level 0 (ug/kg)	L0 (70% - 130%)	Level 1 (ug/kg)	L1 (70% - 130%)	Level 1.5 (ug/kg)	L1.5 (70% - 130%)	Level 2 (ug/kg)	L2 (70% - 130%)	Level 3 (ug/kg)	L3 (70% - 130%)
Limonene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
sec-Butylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
p-Isopropyltoluene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,3-Dichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,4-Dichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
trans-Decahydronaphthalene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Undecane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
p-Diethylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
n-Butylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Hexachloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2-Dichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
cis-Decahydronaphthalene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2,4,5-Tetramethylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2-Dibromo-3-chloropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,3,5-Trichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Nitrobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Hexachlorobutadiene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2,4-Trichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Naphthalene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2,3-Trichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,3-Dioxolane	N/A	N/A	25	17.5 - 32.5	N/A	N/A	50	35 - 65	100	70 - 130

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Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS)

Reference Method No.: EPA 8270 E

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update VI (Phase II), June, 2018.

1. Scope and Application

Matrices: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and wastewater samples.

This method is used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone.

Table 9 lists "difficult" compounds that may require special treatment when being determined by this method.

Approval of any method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (Section 13.2).

2. Summary of Method

The samples are introduced into the GC/MS by injecting 1 μ L of the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards run on the same GC/MS system. Quantitation is accomplished by comparing the response of quantitation ion relative to an internal standard using a calibration curve.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 6 lists our routine reporting limits.

4. Interferences

- 4.1 Only high purity helium is used in the GC system to eliminate this source of possible contamination. The helium (carrier gas) is certified by the gas supplier.
- 4.2 Preventive instrument maintenance is performed routinely. Section 10.5 details the maintenance steps.
- 4.3 Glassware must be scrupulously cleaned. This procedure is detailed in the [Organic Extraction Glassware Cleaning & Handling](#) SOP/1953.
- 4.4 Contaminated solvents or reagents are also possible sources of contamination. All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free.
- 4.5 Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered (concentrations greater than 2x the highest concentration) and the next sample has reportable hits this sample should to be re-analyzed for confirmation based on analyst discretion.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the [Chemical Hygiene Plan](#).

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

- 5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.
- 5.2 All solvent and extract transfers must be handled in the vented bench area in the GC/MS laboratory.
- 5.3 All stock standards, working standards, and vial sample extracts must be placed into the waste bucket in the lab for future disposal by the Health and Safety Officer. The container must be labeled properly with hazard warning labels indicating the container contents.
- 5.4 Flammable solvent bottles must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L amber glass jars with teflon-lined lids. For LVI, aqueous samples are collected in two 275mL amber glass jars with teflon-lined lids. Solid samples are collected in 250mL wide-mouth glass jars with teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of $4 \pm 2^{\circ}\text{C}$. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of $4 \pm 2^{\circ}\text{C}$.

6.3 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph/Mass Spectrometer System:

7.1.1 Gas Chromatograph, Hewlett Packard 6890 (or equivalent): An analytical system complete with a temperature-programmable gas chromatograph configured for split/splitless-injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.

7.1.2 Column: Rxi-5Sil MS30m x 0.32mm ID, 0.25 μm film thickness or column of similar configuration.

7.1.3 Mass Spectrometer, Hewlett Packard 5973 (or equivalent): Scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 1 when 1 μL of the GC/MS tuning standard is injected through the GC (50ng of DFTPP).

7.1.4 Data System: A computer system is interfaced to the Mass Spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows the analyst to search for any GC/MS data file for ions of specific mass and plot such ion abundances versus time or scan number. *HP ChemServer* software is used for data acquisition and *MSD Chemstation/Enviroquant version E.02.02* is used for data reduction.

7.2 Syringe: 10 μL .

7.3 Volumetric Flasks, Class A: Appropriate sizes with ground-glass stoppers.

7.4 Vials: Glass autosampler vials with polytetrafluoroethylene (PTFE)-lined crimp top caps.

8. Reagents and Standards

8.1 Stock Standard Solutions

Certified stock standard solutions, traceable to NIST, when available, are purchased from commercial vendors. They can be replaced with different standards as long as they contain all target analytes.

All stock standards, lot number, catalog number, expiration date, preparation date and initials are recorded in a logbook. Standards are stored in the refrigerator or freezer. Store away from any light source at 6 °C when not in use (-10 °C is recommended).

Stock standard expire 6 months from the date of preparation or on the earliest expiration date of any of the stock solution used to prepare it.

Please note that the following preparation instructions and stock standards are included for illustration purposes and may be modified as needed (ex. to accommodate standard availability or client requests), however final concentrations for the initial calibration levels shall always follow the example in 8.1.4.

<u>Vendor</u>	<u>Standard</u>	<u>Catalog#</u>	<u>Concentration</u>
Restek	8270 Mega Mix	31850	500-1000ug/mL
	Benzoic Acid Mix	31879	2000ug/mL
	Acid Surrogate Mix	31087	10000ug/mL
	B/N Surrogate Mix	31086	5000ug/mL
	Benzaldehyde Standard	33017	2000ug/mL
	Custom AP9 ICAL Standard	571813-FL	2000ug/mL
	Custom ADP Standard	572745-FL	2000ug/mL
	Benzidine Mix	31834	2000ug/mL
	SV Internal Standard Mix	31206	2000ug/mL
	1,4-Dioxane	30287	2000ug/mL
	Custom CLP 04.1 BNA Surrogate Mix	571320-FL	1000ug/mL
Absolute	Aromatic Amines Mix	99410	2000ug/ml
Ultra	Semi-Volatiles GC/MS Tuning Standard	GCM-150-1	1000ug/mL

8.1.1 ABN Stock Standard, 200ug/mL

Use 5mL of each of the following:

Benzoic Acid Mix
Benzidine Mix

and use 10mL of each of the following:

8270 Mega Mix
Custom CLP 04.1 BNA Surrogate Mix

Bring up to 50mL volume with DCM.

8.1.2 AP9 Additional Compounds Stock Standard, 200ug/mL

Use 5mL of each of the following:

Custom AP9 ICAL Standard
Benzaldehyde Standard

Bring up to 50mL volume with DCM.

8.1.3 ADP Stock Standard, 200ug/ml

Use 5ml of:

Custom ADP Standard

Bring up to 50mL volume with DCM.

8.1.4 Calibration Standard

A minimum of 5 calibration standards must be included for each analyte:

Calibration Curve Levels	
Level	Concentration ug/mL
1	1.0
2	2.0
3	3.0
4	5.0
5	10
6	20
7	50
8	100
9	150
10	200

LVI Calibration Curve Levels	
Level	Concentration ug/mL
1	0.2
2	0.4
3	1.0
4	2.0
5	3.0
6	5.0
7	10
8	15
9	35
10	50

*LVI- Low Volume Initiative

8.2 Internal Standard Solution

The internal standards are:

1,4-dichlorobenzene-d₄
naphthalene-d₈
acenaphthene-d₁₀
phenanthrene-d₁₀
chrysene-d₁₂
perylene-d₁₂

Each 500µL of standards, blank and sample extracts are spiked with 10µL of SV Internal Standard Mix, resulting in a concentration of 40ng/ µL.

For the LVI method, a 1:10 dilution is made of the Internal Standard Stock Solution. 500µL of standards, blank and sample extracts are spiked with 10µL of this preparation, resulting in a concentration of 4ng/ µL.

8.3 GC/MS Tuning Standard

The tuning standard is a methylene chloride solution containing 50ng/µL of decafluorotriphenylphosphine (DFTPP). The standard also contains 50ng/µL each of 4,4' DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance.

Prepare the GC/MS Tuning Standard with 25µL GCM-150 and 475µL Dichloromethane.

8.4 Surrogate Spiking Solution

8.4.1 Extraction Surrogate Preparation

In a 1000mL volumetric flask, add 5ml of 31086 and 31087. Bring up to volume with Acetone. The final concentration is 50µg/mL for the acid surrogates and 25µg/mL for the B/N surrogates.

8.4.2 LVI Extraction Surrogate Preparation

The LVI surrogate is a 10 fold dilution of the surrogate solution prepared in 8.4.1. For example, to make 200mL of LVI surrogate, add 20mL of 8.4.1 to a 200mL volumetric flask

and fill to volume with Acetone. The resulting surrogate concentration is 5µg/mL for the acid surrogates and 2.5µg/mL for the B/N surrogates.

8.5 Spike Solution (LCS, MS, MSD)

Spike Solution Preparation

ABN SPK1:

In a 500ml volumetric flask, add 20ml of 8270 Mega Mix #31850, 10ml of Benzoic Acid Mix #31879, 10ml Custom AP9 ICAL Standard #571813-FL and 10ml Benzaldehyde Standard #33017. Bring up to volume with Acetone. The final concentration is 40µg/ml.

Note: the LVI ABN SPK1 is prepared by making an 8 fold dilution of the 40µg/ml ABN SPK (in acetone), resulting in a 5µg/ml LVI ABN SPK1.

ABN SPK2:

In a 500ml volumetric flask, add 10ml Benzidine Mix #31834 and 10mL Custom ADP Standard #572945-FL. Bring up to volume with Acetone. The final concentration is 40µg/ml.

Note: the LVI ABN SPK2 is prepared by making an 8 fold dilution of the 40µg/ml ABN SPK (in acetone), resulting in a 5µg/ml LVI ABN SPK2.

8.6 Dichloromethane (DCM): Pesticide quality.

8.7 Acetone: Pesticide quality.

9. Quality Control

9.1 Blank(s)

Extraction blanks are performed with each extraction batch of 20 or less samples. The extraction blank must not contain any of the reportable analytes above the reporting limit. Corrective actions:

- No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample
- If the blank have reportable hits and re-extraction could not be performed due to lack of additional sample volume, the sample results are reported and qualified with "B" flag for any associated samples that concentration is less than 10x the blank concentration

For NJ regulatory work the method blank must have all the target analytes less than RL except for Phthalates which must be less than 5x of the RL. Sample results are qualified with "B" flag for analytes observed in the blank greater than RL and the Phthalates observed in the blank greater than 5x RL

The surrogate recoveries must also be within the acceptance criteria listed in Table 2. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary.

9.2 Laboratory Control Sample and Laboratory Control Sample Duplicate (LCS / LCSD)

A Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD) are extracted and analyzed with each analytical batch of 20 or fewer samples.

The LCS/LCSD acceptance criteria are based on in-house control limits. Less than 10% of total compounds may be outside of control limits provided that recoveries are >10%. Note: this does not apply to difficult analytes listed in Table 9 which may be accepted at recoveries <10. If >10% of analytes are recovered above control limits, this is deemed acceptable as long as the analytes in question are not detected in associated samples.

If these criteria are not met, the entire batch is re-extracted. If re-extraction is not possible, due to insufficient sample or holding time exceedance, the analyst must write up the failure on a narrative sheet for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike and Matrix Spike Duplicate (MS / MSD)

A matrix spike/matrix spike duplicate pair is extracted and analyzed for each batch of 20 or fewer samples per client request. The MS/MSD acceptance criteria are based on in-house control limits. If the recovery criteria are not met, but are met in the LCS/LCSD, this is noted on a narrative sheet for inclusion in the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

All extracted samples and associated QC are spiked with surrogates. The acceptable surrogate recovery limits are listed in Table 2.

Corrective action: Up to one surrogate can be out in each fraction (Acid and Base/Neutral) but not less than 10% recovery, before any corrective action is necessary. Otherwise, analysis must be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

Re-extraction is not required if surrogate recoveries are high and target analytes are not detected in the sample.

9.7.2 Internal Standards

If the area for any of the internal standards in the samples changes by a factor of two (-50% to +100%) from that in the CCV, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is:

- Degradation Check
- DFTPP
- Continuing or Daily Standards (1 – 3)*
 - (1) ABN 50 ppm
 - (2) AP9 50 ppm
 - (3) ADP 50 ppm
- Method/Instrument Blank
- Samples
- QC (as required)

***Additional Continuing standards may be run at the analyst's discretion or by client request.**

10. Procedure

10.1 Equipment Set-up

10.1.1 GC/MS Operating Conditions:

Typical GC/MS operating conditions are listed below, but may be altered as long as method performance criteria are met.

Mass range:	35 – 500 amu
Scan time:	3.15 second / scan
Initial temperature:	50°C, hold for 1.5 minutes
Temperature program:	28°C/minute to 250°C then 9°C/minute to 320°C
Final temperature:	320°C for 1.50 min
Injector temperature:	300°C
Transfer line temperature:	280°C
Source temperature:	230°C
Injector:	split ratio 5:1; 11.7mL/min
Injection volume:	1µL
Carrier gas:	helium at 523 cm/second (2.0 mL/min) constant flow

GC/MS Operating Conditions for LVI method:

Mass range:	35 – 550 amu
Scan time:	3.15 second / scan
Initial temperature:	45°C, hold for 4 minutes
Temperature program:	25°C/minute to 250°C then 20°C/minute to 320°C
Final temperature:	320°C for 4.3 min
Injector temperature:	270°C
Transfer line temperature:	280°C
Source temperature:	320°C
Injector:	split ratio 5:1; 8.57mL/min
Injection volume:	2µL
Carrier gas:	helium at 1.7148mL/min) constant flow

10.1.2 GC/MS Tune:

At the beginning of every 12 hour sequence, analyze DFTPP tuning solution (Section 8.3).

The resultant mass spectrum for DFTPP must meet the criteria given in Table 1 before sample analysis begins. The mass spectrum of DFTPP should be acquired in the following manner:

- (1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
- (2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of DFTPP.

The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD must not exceed 20%. Benzidine and pentachlorophenol must be present at their normal responses and no peak tailing must be visible.

The tailing factor for benzidine and pentachlorophenol must be calculated in every DFTPP run. (See Table 4)

If degradation is excessive and/or poor chromatography is noted, the system needs maintenance (see Section 10.5).

10.2 Initial Calibration

10.2.1 Prepare calibration standards for all target analytes at a minimum of five concentration levels as specified in Section 8.1.4.

10.2.2 Add 10 μ L of Internal Standard to each calibration standard directly into the autosampler vial containing 500 μ L of standard. Analyze each calibration standard under the conditions specified in Section 10.1.1.

10.2.3 Record the calibration standard, unique lab identifier code (lot), concentration, and analyst's initials in the analytical sequence list.

10.2.4 In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

It is recommended that a minimum response factor for the most common target analytes, as noted in Table 8, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity.

10.2.5 Initial Calibration %RSD Criteria:

For all analytes, the RSD must be \leq 20% for the mean response factor to be used for sample quantitation.

If the RSD for any target analyte exceeds 20%, the fit must be ≥ 0.99 quadratic or ≥ 0.995 linear fit for all target analytes. A minimum of 5 levels are required for a linear calibration model and a minimum of 6 levels are required for a quadratic calibration model. When calculating calibration using the linear regression model, a quantitation check must be performed on the lowest calibration point, or the point that corresponds to that compound's established reporting limit. The recalculated concentration of the low calibration point (especially where the linear regression fits are used) should be within $\pm 50\%$ of the standard's true concentration, and the recalculated concentration of any calibration standards above the LLOQ should be $\pm 30\%$. If this criterion is not met then the corrective action must take place and a new initial calibration must be performed prior to sample analysis

For RCP, the RSD must be $\leq 15\%$ for the mean response factor to be used for sample quantitation

10.2.6 Evaluation of Retention Times:

The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units.

10.2.7 Initial Calibration Verification (Second Source Verification)

10.2.7.1 The initial calibration (Section 10.2) for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing second source calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.2.7.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each analyte is $\pm 30\%$, then the calibration is assumed to be valid. If this criterion is not met, then corrective action must be taken prior to the analysis.

For RCP, if the % Difference for each analyte is $\pm 20\%$, then the calibration is assumed to be valid. If this criterion is not met, then corrective action must be taken prior to the analysis.

10.2.7.3 In cases where compounds fail (greater than 30% difference), they may still be reported as non-detects.

10.2.7.4 Once the calibration curve is accepted and prior to processing any samples, the analyst must demonstrate through the analysis of a Method Blank or Instrument Blank that equipment and reagents are free from contaminants and interferences. If a peak is found in the blank that would prevent the identification or bias the measurement of an analyte, the analyst should determine the source of the contaminant peak and eliminate it, if possible. Blank are generally considered to be acceptable if target analyte concentration are less than one half the LLOQ or are less than project specific requirements. Blanks may contain analyte concentrations are greater than acceptance limits if the associated samples in the batch are unaffected.

10.3 Equipment Operation and Sample Processing

10.3.1 GC/MS Analysis of Samples

- 10.3.1.1 Allow the sample extracts to warm to room temperature.
- 10.3.1.2 Transfer all of the sample extract to a 1.5mL vial. Remove 500 μ L of sample extract to another vial, and add 10 μ L of the internal standard solution (Section 8.2).
- 10.3.1.3 The autosampler is programmed to inject 1 μ L aliquot of the sample extract into the GC/MS system, using the same instrument conditions that were used for calibration. The injection volume of the sample must be the same as the volume used for the calibration standard.
- 10.3.1.4 If the response of any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed.

10.3.2 Qualitative Identification

Perform first level data review. Obtain the primary m/z (Table 5) masses for each parameter of interest. The following criteria must be met to make qualitative identification:

- Compare the background subtracted mass spectra for the sample to the reference spectra. The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.
- The retention time must fall within ± 0.1 minutes of the retention time of the compound in the analytical standard. However, analyst experience must be used in making the qualitative identification.
- The relative peak height of the one characteristic mass must fall within 30% of the relative intensity of the mass in a reference mass spectrum. The reference spectrum is obtained from a standard analyzed on the GC/MS system.

Structural isomers that have very similar mass spectra are identified only if the resolution between authentic isomers in a standard mix is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.4 Continuing Calibration

10.4.1 The initial calibration (Section 10.2) for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.4.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each CCV analyte is $\leq 20\%$, then the calibration is assumed to be valid. If the criterion is not met for more than 20% of the compounds then corrective action must be taken.

Due to the large number of analytes present, allowances may be made for a RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples.

10.4.3 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.4 If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed.

10.4.5 Internal Standard Retention Time

The retention times of the internal standards in the calibration verification standard is evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard of the most recent initial calibration, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.6 Internal Standard Response

Refer to section 9.7.2

10.5 Preventive Maintenance

When poor sensitivity is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the sensitivity does not improve it may be necessary to replace the split line or the injector weldment assembly. If the problem persists, it may be necessary to replace the GC column.

Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

11. Data Evaluation, Calculations and Reporting

When a parameter is identified, the quantitation of that parameter must be based on the integrated abundance of the quantitation characteristic m/z given in Table 5

Calculate the concentration in the sample using the average response factor (RF) from the initial calibration curve according to the formula in Alpha's Quality Manual.

After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

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

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 9 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample

volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in [Alpha SOP/1732](#). These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to [Alpha SOP/1739](#) for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's [Chemical Hygiene Plan](#) and [Waste Management and Disposal SOP](#) for further pollution prevention and waste management information.

15. Referenced Documents

[Chemical Hygiene Plan](#)

[Alpha SOP/1732](#) DL/LOD/LOQ Generation

[Alpha SOP/1739](#) IDC/DOC Generation

[Alpha SOP/1729](#) Waste Management and Disposal SOP

16. Attachments

Table 1: DFTPP Key Ions and Ion Abundance Criteria

Table 2: Acceptable Surrogate Spike Recovery Limits

Table 3A: Acceptable Aqueous QC Limits

Table 3B: Acceptable Soil QC Limits

Table 4: Tailing Factor Calculation

Table 5: Characteristic Ions for Semivolatile Compounds

Table 6: Reported Detection Limits

Table 7: Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Table 8: Recommended Minimum Response Factor Criteria

Table 9: Difficult analytes

TABLE 1
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
68	< 2% of mass 69
69	Present
70	< 2% of mass 69
197	< 2% of mass 198
198	Base peak or present
199	5-9% of mass 198
365	> 1% of Base Peak
441	Present but less than 24% mass 442
442	Base peak, or present
443	15-24% of mass 442

TABLE 2
ACCEPTABLE SURROGATE SPIKE RECOVERY LIMITS

Analytical Fraction	Surrogate Compound	Water	Soil/Sediment
BN-8270D	Nitrobenzene-d ₅	23-120%	23-120%
BN-8270D	2-Fluorobiphenyl	15-120%	30-120%
BN-8270D	p-Terphenyl-d ₁₄	41-149%	18-120%
Acid-8270D	Phenol-d ₆	10-120%	10-120%
Acid-8270D	2-Fluorophenol	21-120%	25-120%
Acid-8270D	2,4,6-Tribromophenol	10-120%	10-136%

It is allowable for one surrogate from each fraction be outside acceptance criteria, provided a minimum recovery of 10% has been achieved.

TABLE 3A
ACCEPTABLE AQUEOUS QC LIMITS

Analyte	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
1,2,4,5-Tetrachlorobenzene			70-130	20	40-140	20
1,2,4-Trichlorobenzene	39-98	30	70-130	20	40-140	20
1,2-Dichlorobenzene	40-140	30	70-130	20		
1,3-Dichlorobenzene	40-140	30	70-130	20		
1,3-Dinitrobenzene	15-130	30				
1,4-Dichlorobenzene	36-97	30	70-130	20		
1-Methylnaphthalene	41-103	30				
2,3,4,6-Tetrachlorophenol			70-130	20		
2,4,5-Trichlorophenol	30-130	30	70-130	20	30-130	20
2,4,6-Trichlorophenol	30-130	30	70-130	20	30-130	20
2,4-Dichlorophenol	30-130	30	70-130	20	30-130	20
2,4-Dimethylphenol	30-130	30	70-130	20	30-130	20
2,4- Dimethylaniline	40-140	30	70-130	20		
3,4- Dimethylaniline	40-140	30	70-130	20		
2,3- Dimethylaniline	40-140	30	70-130	20		
2,4,5-Dimethylaniline	40-140	30	70-130	20		
4-Chlorotoluidine	40-140	30	70-130	20		
2-Ethylaniline	40-140	30	70-130	20		
O-toluidine	40-140	30	70-130	20		
2-Naphthylamine	40-140	30	70-130	20		
2,4-Dinitrophenol	20-130	30	20-130	20	30-130	20
2,4-Dinitrotoluene	24-96	30	70-130	20	40-140	20
2,6-Dinitrotoluene	40-140	30	70-130	20	40-140	20
2-Chloronaphthalene	40-140	30	70-130	20	40-140	20
2-Chlorophenol	27-123	30	70-130	20	30-130	20
2-Methylnaphthalene	40-140	30	70-130	20	40-140	20
2-Methylphenol	30-130	30	70-130	20	30-130	20
2-Nitroaniline	52-143	30	70-130	20	40-140	20
2-Nitrophenol	30-130	30	70-130	20	30-130	20
3,3'-Dichlorobenzidine	40-140	30	70-130	20	40-140	20
3,3'-Dimethylbenzidine			20-160	20		
3-Methylphenol/4-Methylphenol	30-130	30	20-160	20	30-130	20
3-Nitroaniline	25-145	30	70-130	20	40-140	20
4,6-Dinitro-o-cresol	20-164	30	70-130	20	30-130	20
4-Bromophenyl phenyl ether	40-140	30	70-130	20	40-140	20
4-Chloroaniline	40-140	30	20-160	20	40-140	20
4-Chlorophenyl phenyl ether	40-140	30	70-130	20	40-140	20
4-Nitroaniline	51-143	30	70-130	20	40-140	20
4-Nitrophenol	10-80	30	20-160	20	30-130	20
Acenaphthene	37-111	30	70-130	20	40-140	20
Acenaphthylene	45-123	30	70-130	20	40-140	20

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Analyte	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
Acetophenone	39-129	30	70-130	20		
Aniline	40-140	30	20-160	20	40-140	20
Anthracene	40-140	30	70-130	20	40-140	20
Atrazine			70-130	20		
Azobenzene	40-140	30	70-130	20		
Benzaldehyde			20-160	20		
Benzidine	10-75	30	20-160	20		
Benzo(a)anthracene	40-140	30	70-130	20	40-140	20
Benzo(a)pyrene	40-140	30	70-130	20	40-140	20
Benzo(b)fluoranthene	40-140	30	70-130	20	40-140	20
Benzo(ghi)perylene	40-140	30	70-130	20	40-140	20
Benzo(k)fluoranthene	40-140	30	70-130	20	40-140	20
Benzoic Acid	10-164	30	20-160	20		
Benzyl Alcohol	26-116	30	20-160	20		
Biphenyl	40-140	30	70-130	20		
Bis(2-chloroethoxy)methane	40-140	30	70-130	20	40-140	20
Bis(2-chloroethyl)ether	40-140	30	70-130	20	40-140	20
Bis(2-chloroisopropyl)ether	40-140	30	70-130	20	40-140	20
Bis(2-Ethylhexyl)phthalate	40-140	30	70-130	20	40-140	20
Butyl benzyl phthalate	40-140	30	70-130	20	40-140	20
Caprolactam			20-160	20		
Carbazole	55-144	30	70-130	20	40-140	20
Chrysene	40-140	30	70-130	20	40-140	20
Dibenzo(a,h)anthracene	40-140	30	70-130	20	40-140	20
Dibenzofuran	40-140	30	70-130	20	40-140	20
Diethyl phthalate	40-140	30	70-130	20	40-140	20
Dimethyl phthalate	40-140	30	70-130	20	40-140	20
Di-n-butylphthalate	40-140	30	70-130	20	40-140	20
Di-n-octylphthalate	40-140	30	70-130	20	40-140	20
Fluoranthene	40-140	30	70-130	20	40-140	20
Fluorene	40-140	30	70-130	20	40-140	20
Hexachlorobenzene	40-140	30	70-130	20	40-140	20
Hexachlorobutadiene	40-140	30	70-130	20	40-140	20
Hexachlorocyclopentadiene	40-140	30	20-160	20	40-140	20
Hexachloroethane	40-140	30	20-160	20	40-140	20
Indeno(1,2,3-cd)Pyrene	40-140	30	70-130	20	40-140	20
Isophorone	40-140	30	70-130	20	40-140	20
Naphthalene	40-140	30	70-130	20	40-140	20
Nitrobenzene	40-140	30	70-130	20	40-140	20
NitrosoDiPhenylAmine(NDPA)/ Diphenylamine (DPA)	40-140	30	70-130	20	40-140	20
n-Nitrosodimethylamine	22-74	30	20-160	20		
n-Nitrosodi-n-propylamine	29-132	30	70-130	20	40-140	20
P-Chloro-M-Cresol	23-97	30	70-130	20	30-130	20
Pentachlorophenol	9-103	30	20-160	20	30-130	20
Pentachloronitrobenzene					40-140	20

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Analyte	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
Phenanthrene	40-140	30	70-130	20	40-140	20
Phenol	12-110	30	20-160	20	30-130	20
Pyrene	26-127	30	70-130	20	40-140	20
Pyridine	10-66	30			40-140	20
2-Fluorophenol	21-120		15-110		15-110	
Phenol-d6	10-120		15-110		15-110	
Nitrobenzene-d5	23-120		30-130		30-130	
2-Fluorobiphenyl	15-120		30-130		30-130	
2,4,6-Tribromophenol	10-120		15-110		15-110	
4-Terphenyl-d14	41-149		30-130		30-130	

TABLE 3B
ACCEPTABLE SOIL QC LIMITS

Analyte	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
1,2,4,5-Tetrachlorobenzene	40-117	50	70-130	30	40-140	30
1,2,4-Trichlorobenzene	38-107	50	70-130	30	40-140	30
1,2-Dichlorobenzene	40-140	50	70-130	30		
1,3-Dichlorobenzene	40-140	50	70-130	30		
1,3-Dinitrobenzene	40-140	50				
1,4-Dichlorobenzene	28-104	50	70-130	30		
1-Methylnaphthalene	26-130	50				
2,3,4,6-Tetrachlorophenol	40-140	50	70-130	30		
2,4,5-Trichlorophenol	30-130	50	70-130	30	30-130	30
2,4,6-Trichlorophenol	30-130	50	70-130	30	30-130	30
2,4-Dichlorophenol	30-130	50	70-130	30	30-130	30
2,4-Dimethylphenol	30-130	50	70-130	30	30-130	30
2,4-Dinitrophenol	4-130	50	20-160	30	30-130	30
2,4-Dinitrotoluene	28-89	50	70-130	30	40-140	30
2,6-Dinitrotoluene	40-140	50	70-130	30	40-140	30
2-Chloroaniline	30-130	50				
2-Chloronaphthalene	40-140	50	70-130	30	40-140	30
2-Chlorophenol	25-102	50	70-130	30	30-130	30
2-Methylnaphthalene	40-140	50	70-130	30	40-140	30
2-Methylphenol	30-130.	50	70-130	30	30-130	30
2-Nitroaniline	47-134	50	70-130	30	40-140	30
2-Nitrophenol	30-130	50	70-130	30	30-130	30
3,3'-Dichlorobenzidine	40-140	50	70-130	30	40-140	30
3,3'-Dimethylbenzidine	15-115	50				
3-Methylphenol/4-Methylphenol	30-130	50	20-160	30	30-130	30
3-Nitroaniline	26-129	50	70-130	30	40-140	30
4,6-Dinitro-o-cresol	10-130	50	70-130	30	30-130	30
4-Bromophenyl phenyl ether	40-140	50	70-130	30	40-140	30
4-Chloroaniline	40-140	50	20-160	30	40-140	30
4-Chlorophenyl phenyl ether	40-140	50	70-130	30	40-140	30
4-Nitroaniline	41-125	50	70-130	30	40-140	30
4-Nitrophenol	11-114	50	20-160	30	30-130	30
Acenaphthene	31-137	50	70-130	30	40-140	30

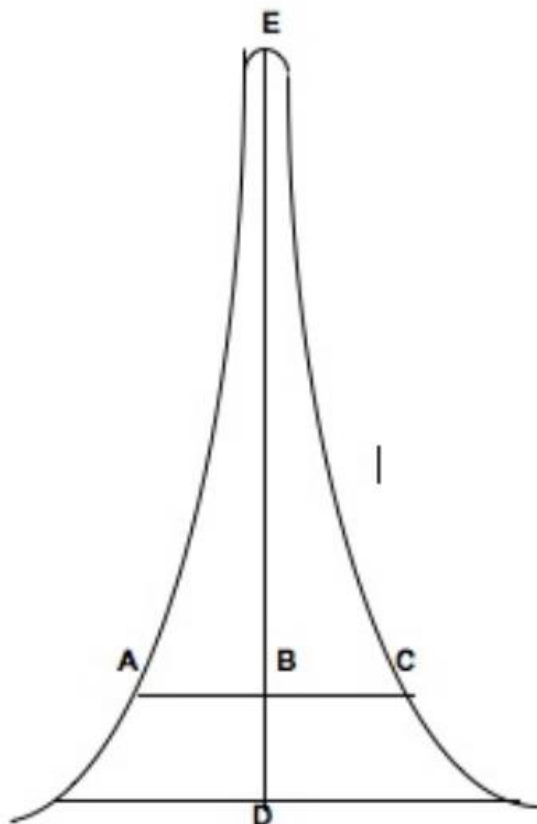
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Analyte	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
Acenaphthylene	40-140	50	70-130	30	40-140	30
Acetophenone	14-144	50	70-130	30	40-140	30
Aniline	40-140	50	20-160	30	40-140	30
Anthracene	40-140	50	70-130	30	40-140	30
Atrazine	40-140	50	70-130	30		
Azobenzene	40-140	50	70-130	30		
Benzaldehyde	40-140	50	20-160	30		
Benzidine	10-66	50	20-160	30		
Benzo(a)anthracene	40-140	50	70-130	30	40-140	30
Benzo(a)pyrene	40-140	50	70-130	30	40-140	30
Benzo(b)fluoranthene	40-140	50	70-130	30	40-140	30
Benzo(e)pyrene	40-140	50				
Benzo(ghi)perylene	40-140	50	70-130	30	40-140	30
Benzo(k)fluoranthene	40-140	50	70-130	30	40-140	30
Benzoic Acid	10-110	50	20-160	30		
Benzyl Alcohol	40-140	50	20-160	30		
Biphenyl	37-127	50	70-130	30		
Bis(2-chloroethoxy)methane	40-117	50	70-130	30	40-140	30
Bis(2-chloroethyl)ether	40-140	50	70-130	30	40-140	30
Bis(2-chloroisopropyl)ether	40-140	50	70-130	30	40-140	30
Bis(2-Ethylhexyl)phthalate	40-140	50	70-130	30	40-140	30
Butyl benzyl phthalate	40-140	50	70-130	30	40-140	30
Caprolactam	15-130	50	20-160	30		
Carbazole	54-128	50	70-130	30	40-140	30
Chrysene	40-140	50	70-130	30	40-140	30
Dibenzo(a,h)anthracene	40-140	50	70-130	30	40-140	30
Dibenzofuran	40-140	50	70-130	30	40-140	30
Diethyl phthalate	40-140	50	70-130	30	40-140	30
Dimethyl phthalate	40-140	50	70-130	30	40-140	30
Di-n-butylphthalate	40-140	50	70-130	30	40-140	30
Di-n-octylphthalate	40-140	50	70-130	30	40-140	30
Diphenamid	40-140	50				
Fluoranthene	40-140	50	70-130	30	40-140	30
Fluorene	40-140	50	70-130	30	40-140	30
Hexachlorobenzene	40-140	50	70-130	30	40-140	30
Hexachlorobutadiene	40-140	50	70-130	30	40-140	30
Hexachlorocyclopentadiene	40-140	50	20-160	30	40-140	30
Hexachloroethane	40-140	50	20-160	30	40-140	30
Indeno(1,2,3-cd)Pyrene	40-140	50	70-130	30	40-140	30
Isophorone	40-140	50	70-130	30	40-140	30

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Analyte	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
Naphthalene	40-140	50	70-130	30	40-140	30
Nitrobenzene	40-140	50	70-130	30	40-140	30
NitrosoDiPhenylAmine(NDPA)/ Diphenylamine (DPA)	36-157	50	70-130	30	40-140	30
n-Nitrosodimethylamine	22-100	50	20-160	30		
n-Nitrosodi-n-propylamine	32-121	50	70-130	30	40-140	30
Parathion, ethyl	40-140	50	20-160	30		
P-Chloro-M-Cresol	26-103	50	70-130	30	30-130	30
Pentachloronitrobenzene	42-153	50			40-140	30
Pentachlorophenol	17-109	50	20-160	30	30-130	30
Phenanthrene	40-140	50	70-130	30	40-140	30
Phenol	26-90	50	20-160	30	30-130	30
Pyrene	35-142	50	70-130	30	40-140	30
Pyridine	10-93	50	20-160	30	40-140	30
Thionazin	40-140	50				
2-Fluorophenol	25-120		30-130		30-130	
Phenol-d6	10-120		30-130		30-130	
Nitrobenzene-d5	23-120		30-130		30-130	
2-Fluorobiphenyl	30-120		30-130		30-130	
2,4,6-Tribromophenol	10-136		30-130		30-130	
4-Terphenyl-d14	18-120		30-130		30-130	

TABLE 4 – Tailing Factor Calculation



$$\text{Tailing Factor} = \frac{BC}{AB}$$

Example calculation:

Peak Height = DE = 100mm
10% Peak Height = BD = 10mm
Peak Width at 10% Peak Height = AC = 23mm

AB = 11mm
BC = 12mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

Tailing factor for benzidine < 2.0

Tailing factor for pentachlorophenol <2.0

TABLE 5
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
Acenaphthene	154	153, 152
Acenaphthylene	152	151, 153
Acetophenone	105	71, 51, 120
Aniline	93	66, 65
Anthracene	178	176, 179
Atrazine	200	202, 215
Azobenzene	77	182, 105
Benzaldehyde	105	77
Benzidine	184	92, 185
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic acid	105	122, 77
Benzyl alcohol	79	77,108
Biphenyl	154	153,152
Bis (2-chloroethoxy) methane	93	95, 123
Bis (2-chloroethyl) ether	93	63, 95
Bis (2-chloroisopropyl) ether	45	77, 121
Bis (2-ethylhexyl) phthalate	149	167, 279
4-Bromophenyl phenyl ether	248	250, 141
Butyl Benzyl phthalate	149	91, 206
Caprolactam	55	85, 113
Carbazole	167	168, 166
4-Chloro-3-methylphenol	107	144, 142
2-Chloroaniline	127	129, 65
3-Chloroaniline	65	127, 129
4-Chloroaniline	65	127,129
2-Chloronaphthalene	162	127, 164
4-Chlorophenyl phenyl ether	204	206, 141
2-Chlorophenol	128	64,130
Chrysene	228	226, 229
Dibenzo(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
1,2-Dichlorobenzene	146	148, 111

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1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
3,3'-Dichlorobenzidine	252	254, 126
2,4-Dichlorophenol	162	164, 98
Diethyl phthalate	149	177, 150

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
3,3-Dimethylbenzidine	212	211, 213
Dimethyl phthalate	163	194, 164
2,4-Dimethylphenol	107	121,122
Di-n-butyl phthalate	149	150, 104
Di-n-octyl phthalate	149	167, 43
4,6-Dinitro-2-methylphenol	198	51, 105
O-Toluidine	106	107, 77
2-Ethylaniline	106	121, 77
2,4-Dimethylaniline	121	120, 106
2,3-Dimethylaniline	106	121, 120
3,4- Dimethylaniline	121	120,106
2,4,5-Trimethylaniline	120	135, 134
4-Chlorotoluidine	106	141, 140
2-Naphthylamine	143	115, 116
2,4-Dinitrophenol	184	107,91
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
Diphenamide	167	72, 165
1,4-Dioxane	88	58,43
Ethyl parathion	109	97, 291
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Hexachlorobenzene	284	142, 249
Hexachlorobutadiene	225	223, 227
Hexachlorocyclopentadiene	237	235, 272
Hexachloroethane	117	201, 199
Indeno(1,2,3-cd)pyrene	276	138, 227
Isophorone	82	95, 138
1-Methylnaphthalene	115	141, 142
2-Methylnaphthalene	142	141
2-Methylphenol	108	107,90
3/4-Methylphenol	108	107,90

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Naphthalene	128	129, 127
2-Nitroaniline	65	92, 138
3-Nitroaniline	138	92,65
4-Nitroaniline	138	65, 108, 92, 80, 39
Nitrobenzene	77	123, 65

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
2-Nitrophenol	139	109, 65
4-Nitrophenol	65	109, 139
n-Nitrosodimethylamine	74	42,44
n-Nitrosodi-n-butylamine	84	57, 41, 116, 158
n-Nitrosodi-n-propylamine	70	42, 101, 130
n-Nitrosodiphenylamine/Diphenylamine	169	168, 167
Pentachlorobenzene	250	252, 108, 248, 215, 254
Pentachloronitrobenzene	237	142, 214, 249, 295, 265
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Phenol	94	65, 66
Pyrene	202	200, 203
Pyridine	79	52
1,2,4,5-Tetrachlorobenzene	216	214, 179, 108, 143, 218
2,3,4,6-Tetrachlorophenol	232	131, 230, 166, 234, 168
m-Toluidine	106	107, 79
1,2,4-Trichlorobenzene	180	182, 145
2,4,5-Trichlorophenol	196	200,198
2,4,6-Trichlorophenol	196	198, 200
Acenaphthene-d ₁₀ (IS)	164	162, 160
Chrysene-d ₁₂ (IS)	240	120, 236
1,4-Dichlorobenzene-d ₄ (IS)	152	150, 115
Naphthalene-d ₈ (IS)	136	68
Perylene-d ₁₂ (IS)	264	260, 265
Phenanthrene-d ₁₀ (IS)	188	94, 80
2-Fluorobiphenyl (Surrogate)	172	171
2-Fluorophenol (Surrogate)	112	64
Nitrobenzene-d ₅ (Surrogate)	82	128, 54
Phenol-d ₆ (Surrogate)	99	42, 71
Terphenyl-d ₁₄ (Surrogate)	244	122, 212
2,4,6-Tribromophenol (Surrogate)	330	62,141

TABLE 6
REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS *

Analyte	RDL (µg/L)	RDL (µg/Kg)
Acenaphthene	2	133.34
Acenaphthylene	2	133.34
Acetophenone	5	333.34
Aniline	2	133.34
Anthracene	2	133.34
Atrazine	10	666.67
Azobenzene	2	500
Benzaldehyde	5	333.34
Benzidine	20	1333.34
Benzo(a)anthracene	2	133.34
Benzo(b)fluoranthene	2	133.34
Benzo(k)fluoranthene	2	133.34
Benzo(ghi)perylene	2	133.34
Benzo(a)pyrene	2	133.34
Benzoic acid	50.0	3333.34
Benzyl alcohol	2	133.34
Biphenyl	2	366.67
Bis(2-chloroethyl)ether	2	133.34
Bis(2-chloroisopropyl)ether	2	133.34
Bis(2-chloroethoxy)methane	5.0	333.34
Bis(2-ethylhexyl)phthalate	3	200
4-Bromophenyl phenyl ether	2	133.34
Butyl benzyl phthalate	5.0	333.34
Caprolactam	10	666.67
Carbazole	2	166.67
2-Chloroaniline	2	na
3-Chloroaniline	10	na
4-Chloroaniline	5	333.34
p-Chloro-m-cresol (4-chloro-3-cresol)	2	133.34
2-Chloronaphthalene	2	133.34
2-Chlorophenol	2	133.34
4-Chlorophenyl phenyl ether	2	133.34
Chrysene	2	133.34
m/p-Methylphenol (3/4-methylphenol)	5.0	333.34
o-Methylphenol (2-methylphenol)	5.0	333.34

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Dibenzo(a,h)anthracene	2	133.34
Dibenzofuran	2	133.34
Di-n-butylphthalate	5.0	333.34
1,2-Dichlorobenzene	2	133.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
1,3-Dichlorobenzene	2	133.34
1,3-Dinitrobenzene	2	N/A
1,4-Dichlorobenzene	2	133.34
3,3-Dichlorobenzidine	5	333.34
2,4-Dichlorophenol	5	333.34
O-Toluidine	2	N/A
2-Ethylaniline	2	N/A
2,4-Dimethylaniline	2	N/A
2,3-Dimethylaniline	2	N/A
3,4-Dimethylaniline	2	N/A
2,4,5-Trimethylaniline	2	N/A
4-Chlorotoluidine	2	N/A
2-Napthylamine	2	N/A
2,6-Dichlorophenol	10.0	666.67
Diethyl phthalate	5.0	333.34
3,3-Dimethylbenzidine	4	500
2,4-Dimethylphenol	5	333.34
Dimethyl phthalate	5.0	333.34
4,6-Dinitro-o-cresol	10	666.67
2,4-Dinitrophenol	20	1333.4
2,4-Dinitrotoluene	5.0	333.34
2,6-Dinitrotoluene	5.0	333.34
Di-n-octylphthalate	5.0	333.34
Diphenamide	5	N/A
1,4-Dioxane	5	166.67
Ethyl Parathion	N/A	166.67
Fluoranthene	2	133.34
Fluorene	2	133.34
Hexachlorobenzene	2	133.34
Hexachlorobutadiene	2	133.34
Hexachlorocyclopentadiene	20	1333.34
Hexachloroethane	2	133.34
Indeno(1,2,3-cd)pyrene	2	133.34
Isophorone	5.0	333.34

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1-Methylnaphthalene	2	166.67
2-Methylnaphthalene	2	133.34
Naphthalene	2	133.34
2-Nitroaniline	5.0	333.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS *

Analyte	RDL (µg/L)	RDL (µg/Kg)
3-Nitroaniline	5.0	333.34
4-Nitroaniline	5.0	333.34
Nitrobenzene	2	133.34
2-Nitrophenol	10.0	666.67
4-Nitrophenol	10.0	666.67
Nitrosodi-n-butylamine	10.0	666.67
n-Nitrosodimethylamine	2	133.34
n-Nitrosodiphenylamine/Diphenylamine	2	133.34
Nitrosodipiperidine	20.0	2000
n-Nitrosodi-n-propylamine	5.0	333.34
Pentachlorobenzene	20.0	1333.34
Pentachloronitrobenzene	10.0	150
Pentachlorophenol	10.0	666.67
Phenanthrene	2	133.34
Phenol	5.0	333.34
Pyrene	2	133.34
Piridine	5	666.67
1,2,4,5-Tetrachlorobenzene	10	666.67
1,2,4-Trichlorobenzene	5.0	333.34
2,4,5-Trichlorophenol	5.0	333.34
2,4,6-Trichlorophenol	5.0	333.34
2,3,4,6-Tetrachlorophenol	5.0	166.66
m-Toluidine	5	300

* Note: Reporting Limits are based on standard 8270 reporting list. RLs may vary for other reporting lists.

Table 7
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
O-Toluidine	2-Ethylaniline	2-Naphthylamine	3,3-Dimethylbenzidine	3,3'-Dichlorobenzidine	Benzo(g,h,i)perylene
1,2,4-Trichlorobenzene	2,4-Dimethylaniline	2,3,4,6-Tetrachlorophenol	Anthracene	Benzo(a)Anthracene	Dibenzo(a,h)anthracene
1,2-Dichlorobenzene	3,4-Dimethylaniline	2,3,5,6-Tetrachlorophenol	Benzidine	Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene
1,3-Dichlorobenzene	2,3-Dimethylaniline	2,4,6-Tribromophenol, surr	Benzyl butyl phthalate	Benzo(b)fluoranthene	
1,4-Dichlorobenezne	2,4,5-Trimethylaniline	2,4-Dinitrophenol	Carbazole	Benzo(k)fluoranthene	
2,4-Dichlorophenol	4-Chlorotoludine	2,4-Dinitrotoluene	Di-n-Butylphthalate	Bis(2-ethylhexyl) phthalate	
2,4-Dimethylphenol	1,2,4,5-Tetrachlorobenzene	3-Nitroaniline	Diphenamid	Chrysene	
2-Chloroaniline	1,2-Dichlorobenzene	4,6-Dinitro-2-methylphenol	Fluoranthene	Di-n-octylphthalate	
2-Chlorophenol	1,3-Dichlorobenzene	4-Bromophenyl-phenyl ether	n-Octadecane		
2-Fluorophenol, surr	1,4-Dichlorobenzene	4-Chlorophenyl-phenyl ether	Parathion		
2-Methylphenol	1-chloror-2-nitrobenzene	4-Nitroaniline	Phenanthrene		
2-Nitrophenol	1-Methylnaphthalene	4-Nitrophenol	Pyrene		
3-Methylphenol / 4-Methylphenol	2,4,5-Trichlorophenol	Acenaphthene	Terphenyl-d14, surr		
Acetophenone	2,4,6-Trichlorophenol	Atrazine			
Aniline	2,6-Dichlorophenol	Azobenzene			
Benzaldehyde	2,6-Dinitrotoluene	Dibenzofuran			
Benzyl Alcohol	2-Chloronaphthalene	Dichloran			
Bis(2-chloroethoxy)methane	2-Fluorobiphenyl, surr	Diethyl phthalate			
Bis(2-chloroethyl)ether	2-Methylnaphthalene	Fluorene			

Printouts of this document may be out of date and should be considered uncontrolled. To accomplish work, the published version of the document should be viewed online.

**Table 7 (cont.)
 Semivolatile Internal Standards with Corresponding
 Target Compounds and Surrogates Assigned for Quantitation**

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
bis(2-Chloroisopropyl)ether	2-Nitroaniline	Hexachlorobenzene			
Hexachloroethane	3-Choloroaniline	NDPA/DPA			
Isophorone	4-Chloro-3-Methylphenol	Pentachloronitrobenzene			
m-Toluidine	4-Chloroaniline	Pentachlorophenol			
n-Decane	Acenaphthylene				
Nitrobenzene	a-Terpineol				
Nitrobenzene-d5, surr	Benzoic Acid				
N-Nitrosodimethylamine	Biphenyl				
N-Nitrosodi-n-propylamine	Caprolactam				
Phenol	Dimethyl Phthalate				
Phenol-d6, surr	Hexachlorobutadiene				
Pyridine 1,4-Dioxane	Hexachlorocyclopentadiene				
Phenol-d6, surr	Naphthalene				

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Table 8

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration
Verification Using the Suggested Ions in Table 5

Analyte	MRF
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010

Table 8 (cont.)

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration
Verification Using the Suggested Ions in Table 5

Analyte	MRF
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Table 9
Difficult analytes

Aniline

Benzaldehyde
Benzidine
Benzoic acid
Benzyl alcohol

Caprolactam
4-Chloroaniline
4-chloro-3-methylphenol (p-chloro-m-cresol)

3,3-Dimethylbenzidine
Dimethylphthalate
2,4 Dinitrophenol
4,6-dinitro-2-methylphenol (4,6-dinitro-o-cresol)

Hexachlorocyclopentadiene
Hexachloroethane

2-Methylphenol
3-Methylphenol/4-Methylphenol

2-nitroaniline
3-nitroaniline
4-nitroaniline
4-Nitrophenol
Nitrosodiphenylamine and diphenylamine (NDPA/DPA)
n-Nitrosodimethylamine

Parathion
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

Inductively Coupled Plasma - Atomic Emission Spectrometry

Reference Method No.: **Method 6010D** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update V, July, 2014.

SM 2340B, Hardness by Calculation, Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF, 21st Edition, 1997.

1. Scope and Application

Matrices: Digestates from all matrices.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion unless chemical interferences are suspected. Samples which are not digested are matrix matched with the standards. Refer to Metals Preparation SOPs for the appropriate digestion procedures.

Table 1 lists the elements for which this method is applicable. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 lists the recommended analytical wavelengths for the elements in clean aqueous matrices. Table 3 lists the Reported Detection Limits. The reported detection limit data may be used to estimate instrument and method performance for other sample matrices. Elements other than those listed in Table 1 may be analyzed by this method if performance at the concentration levels of interest (see Section 9) is demonstrated.

Users of the method should state the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to using the method for analysis.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is made by one of the following laboratory personnel before performing the modification: Area Supervisor, Metals Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

This method describes multielemental determinations by ICP-AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. In one mode of analysis the position used must be as free as possible from spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 4.0 must also be recognized and appropriate corrections made; tests for their presence are described in Section 9.7. Alternatively, users may choose multivariate calibration methods. In this case, point selections for background correction are superfluous since whole spectral regions are processed.

This SOP includes the manual calculations for Total Hardness and Calcium Hardness, according to SM 2340B.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Refer to Table 3 for method Reporting Limits.

4. Interferences

4.1 Spectral

Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

4.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for

routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans must be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

- 4.1.2** To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.
- 4.1.3** Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.
- 4.1.4** When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that each instrument may exhibit somewhat different levels of interference. The interference effects must be evaluated for each individual instrument since the intensities will vary.

Major known interferences are Fe, Al, Ca, Mg, V, Ni, Cu, and Cr. To minimize any of these interferences, every analyte is analyzed on each instrument at or near its linear range and corrected for these interferences. This is done on an annual basis, and data is kept on file.

- 4.1.5 Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear must be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples may contain uncommon elements that could contribute spectral interferences.
- 4.1.6 The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.
- 4.1.7 The primary wavelength for each analyte is based upon the instrument manufacturer's recommendations. An alternate wavelength is chosen if there is an indication of elevated background or overlap of another spectral wavelength. The wavelength for each analyte must be as free from interferences as possible.
- 4.1.8 If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution must fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change must be determined and corrected and the correction factor updated. The interference check solutions must be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.
- 4.1.9 When inter-element corrections are applied, their accuracy must be verified, daily, by analyzing the spectral interference check solution. The correction factor or multivariate correction matrices tested on a daily basis. All inter-element spectral correction factors or multivariate correction matrices are verified and updated when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. The standard solution must be inspected to ensure that there is no contamination that may be perceived as a spectral interference.
- 4.1.10 When inter-element corrections are not used, verification of absence of interferences is required.
- 4.1.10.1 One method is to use a computer software routine for comparing the determinative data to limits, files for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration, (i.e., greater than) the analyte

instrument detection limit, or false negative analyte concentration, (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).

- 4.1.10.2** Another method is to analyze an Interference Check Solution(s) which contains similar concentrations of the major components of the samples (>10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is >20% of the analyte concentration, the analyte must be determined using (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.

4.2 Physical

Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, using a peristaltic pump, use of an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers. The test described in Section 10.3.4.1 will help determine if a physical interference is present.

4.3 Chemical

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Additionally, if filtered samples are found to have an organic or sulfur like odor they are processed by heating after the addition of the acids to matrix match. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.4 Memory

Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample must be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit must be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.

4.5 Other Interferences

- 4.5.1 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations must be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic bottles.

6.2 Sample Preservation

If samples are for soluble metals analysis, filtration must take place prior to preservation with 1:1 HNO₃ to a pH < 2. Soluble samples must be held at pH < 2 for at least 24 hours prior to digestion if not preserved at the time of filtration. Samples for total metals analysis are preserved with 1:1 HNO₃ to a pH < 2. Samples must be pH <2 for at least 24 hours prior to digestion if not preserved at the time of collection.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples to be analyzed for soluble metals, that have not been filtered, must be filtered and preserved within 24 hours of sample collection.

Preserved samples have a hold time of 6 months, and are stored at ambient temperature.

7. Equipment and Supplies

7.1 Inductively coupled argon plasma emission spectrometer:

- Thermo Scientific ICAP Duo 6500 (Trace7)

7.1.1 Computer-controlled emission spectrometer with background correction.

- 7.1.2 Radio-frequency generator compliant with FCC regulations.
- 7.1.3 Optional mass flow controller for argon nebulizer gas supply.
- 7.1.4 Optional peristaltic pump.
- 7.1.5 Optional Autosampler.
- 7.1.6 Argon gas supply - high purity.

7.2 Volumetric flasks of suitable precision and accuracy.

7.3 Volumetric pipets of suitable precision and accuracy.

8. Standards and Reagents

Reagent semiconductor and/or trace grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration of the contamination is less than the MDL then the reagent is acceptable.

8.1 Hydrochloric acid (conc), HCl. Stored at room temperature in acid resistant cabinet. Expiration date if defined by vendor.

8.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL DI water and dilute to 1 liter in an appropriately sized beaker. Stored at room temperature in polypropylene bottle, expiration date if defined by vendor..

8.3 Nitric acid (conc), HNO₃. Stored at room temperature in acid resistant cabinet. Expiration date if defined by vendor.

8.4 Nitric acid (1:1), HNO₃. Add 500 mL concentrated HNO₃ to 400 mL DI water and dilute to 1 liter in an appropriately sized beaker. Stored at room temperature in polypropylene bottle, expiration date if defined by vendor..

8.5 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Refer to Chapter One for a definition of reagent water.

8.6 Standard stock solutions may be purchased or prepared from ultra- high purity grade chemicals or metals (99.99% pure or greater). All stock standards are ordered through ISO and American Association for Lab Accreditation vendors. All standards are in aqueous solutions and are generally at concentrations of 1000ppm and 10,000ppm.

8.7 Mixed calibration standard solutions

Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. Add the appropriate types and volumes of acids so that the standards are matrix matched with the sample digestates. Care must be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to HDPE or polypropylene bottles for storage. Fresh

mixed standards must be prepared, as needed, with the realization that concentration can change on aging as evidenced by failures in the ICV.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. For this acid combination, the silver concentration must be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.

Additionally, sulfur standards are stand-alone single element standards and therefore are not to be combined in a mixed calibration standard solution.

8.8 Blanks

Three types of blanks are required for the analysis for samples. The calibration blank is used in establishing the analytical curve, and the method blank is used to identify possible contamination resulting from varying amounts of the acids used in the sample processing. The rinse blank is used to flush the system between all samples and standards.

8.8.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations (see Sections 10.2 and 10.4). Refer to Section 10.4.1.2 for acceptance criteria and/or corrective actions.

8.8.2 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis. Refer to Section 9.1 for acceptance criteria and/or corrective actions.

8.8.3 The rinse blank consists of HNO₃ (1% or 2%) (v/v) in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

8.9 The Initial Calibration Verification Standard (ICV) and the Continuing Calibration Verification Standard (CCV)

These ICV is prepared by the analyst by combining compatible elements from a standard source different than that of the calibration standard at a concentration at or near the mid-point of the calibration curve. The CCV is prepared from the same source as the calibration standards and must be at a concentration near the mid-point of the calibration curve.

8.10 Low Level of Quantification, (LLOQ)

The LLOQ is initially verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method at or below the lowest calibration point. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be +/- 35% of the true value and RSD should be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.

Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

8.11 Spectral Interference Check Solution

These solutions are prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Analysts are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests.

Single element interference checks - At a minimum, single element SIC checks must be performed for the following elements: Aluminum 500mg/L; Boron 50mg/L, Barium, 50mg/L, Calcium 500mg/L; Copper 50mg/L; Iron 200mg/L; Magnesium 500mg/L; Manganese 50mg/L; Molybdenum 20mg/L; Sodium 1000mg/L; Nickel 20mg/L; Selenium 20mg/L; Silicon 200mg/L; Tin 20mg/L; Vanadium 20mg/L; Zinc 20mg/L The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be less than two times the analytes' LLOQ.

The concentration of the SIC checks are suggested, but become the highest concentration allowed in a sample analysis, and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Note that reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements. The single element SIC checks are performed when the instrument is setup and periodically (at least once every 6 months) thereafter.

Mixed element interference check - The mixed element SIC solution is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, or alternatively the LLOQ may be raised to twice the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions These may be present up to the concentration documented plus the LLOQ.

Mixed element SIC solution: Aluminum, 500mg/L; Calcium, 500mg/L; Iron, 200mg/L; Magnesium, 500mg/L

8.12 Ongoing Low Level of Quantification, (LLOQ)

Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

8.13 Internal Standard

The internal standard consists of a multi-element solution; each internal standard covers a range of the spectrum (low, middle, or high wavelengths) and the elements within that range.

0.003 mg/L Y

Note: The standard is used to monitor instrument fluctuations including but not limited to nebulization efficiency, plasma variations, environmental temperature changes, peristaltic pump pulsations, etc. Therefore, the solution used to start an analysis calibration cannot be added to or changed out during analysis without requiring subsequent full recalibration.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank is a volume of reagent water carried through the same preparation process as a sample.

The method blank results must be less than $\frac{1}{2}$ of the LLOQ for all analytes of concern. If the results of the method blank exceed the RDL for any analyte, perform re-analysis of a new aliquot of the method blank.

If the results continue to exceed the RDL, proceed as follows:

If all of the samples for the analyte are non-detected, and the method blank is at or above the RDL, no action is required.

If one or more associated samples for that analyte have positive results at or above the RDL, those samples must be considered to be out of control, and are re-digested and reanalyzed.

9.2 Laboratory Control Sample (LCS)

Analyze one LCSW/SRM per sample batch. A LCS/SRM sample is a spiked volume of reagent water that is brought through the entire preparation and analytical process. The LCSW must have a % Recovery of $\pm 20\%$ within the actual value or within vendor control limits (95% confidence limits) for the solid SRM.

If the LCSW or SRM % Recovery is outside the acceptable limits as stated in Table 2, or outside any vendor control limits, the LCS is rerun once. If upon reanalysis the LCS is still out of control, the failed analytes are re-prepped and re-analyzed. Otherwise, a nonconformance report form is raised to document the exact problem and this form is then authorized by the QA/QC Director and/or the Laboratory Manager(s).

9.3 Initial Calibration Verification (ICV)

For all analytes and determinations, the laboratory must analyze an ICV (Section 8.9), and a calibration blank (ICB, Section 8.8.1), immediately following daily calibration. The results of the ICV are to agree within 10% of the expected value; if not, re-analyze once, if still failing terminate the analysis, correct the problem, and recalibrate the instrument.

9.4 Continuing Calibration Verification (CCV)

A calibration blank (CCB, Section 8.8.1) and a calibration verification standard (CCV, Section 8.9) must be analyzed after every tenth sample and at the end of the sample run. Analysis of the calibration verification (CCV) must verify that the instrument is within 10% of the calibration with the relative standard deviation $< 5\%$ from replicate (minimum of two) integrations.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The batch sheet is noted. If the standard fails again, instrument maintenance must be performed and the CCV/CCB standard is reanalyzed. If the standard passes, then all samples run after the last passing CCV/CCB pair must be re-analyzed.

If the standard fails after instrument maintenance, the instrument is recalibrated. A new ICV/ICB is performed, and all previous data after the last passing CCV/CCB is reanalyzed.

9.5 Matrix Spike

Analyze matrix spike samples at a frequency of one per matrix batch. The matrix spike is the same solution as used for the LCS (Table 4). A matrix spike sample is a sample brought through the entire sample preparation and analytical process.

9.5.1 The percent recovery is to be calculated as follows:

$$\% \text{ Recovery} = \frac{\text{MS} - \text{S}}{\text{C}} \times 100$$

where:

MS = Matrix Spike value

S = Sample value.

C = Concentration of the Spiking solution.

9.5.2 If the Matrix Spike falls outside of the limits as stated in Table 2, or outside any historical documentation for analytes of interest a post analytical spike is performed for the failed analytes. The same sample from which the MS/MSD aliquots were prepared should be spiked with a post digestion spike at a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. The acceptable % Recovery of the post analytical spike is 75-125%. A nonconformance is noted in the LIMS and approved in secondary peer review and/or by the Metals Manager.

9.5.3 If the Post Spike fails the dilution test should be performed. If the analyte concentration is sufficiently high (minimally, a factor of 25 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution should agree within $\pm 20\%$ of the original determination. If not, then a chemical or physical interference effect should be suspected.

9.6 Laboratory Duplicate

A duplicate sample is analyzed once per matrix batch. This sample is brought through the entire sample preparation and analytical process.

9.6.1 The relative percent difference between duplicate determinations is to be calculated as follows:

$$\text{RPD} = \frac{|D_1 - D_2|}{(|D_1 + D_2|) / 2} \times 100$$

where:

RPD = relative percent difference.

D₁ = first sample value.

D₂ = second sample value (replicate).

9.6.2 If the Duplicate falls outside of the limits as stated in Table 2, or outside any historical documentation and the concentrations of the failing analytes are less than 5x the RL or a matrix interference is found a nonconformance is noted in the LIMS and approved in secondary peer review and/or by the Metals Manager.

9.7 Method-specific Quality Control Samples

9.7.1 Spectra Interference Check Standard

A mixed check solution is analyzed once daily (section 8.11). One solution (SIC) has only elevated concentrations of Fe, Al, Ca, Mg to ensure no interferences occur. The concentrations of the analytes of interest must have an absolute value of the LLOQ. This solution is analyzed at the beginning of the first analytical run of the day.

The high level interferences are not evaluated for recovery. If the SIC fails take corrective action which may include re-evaluation of the inter-element correction values (IECs) after running single element SIC. The instrument calibration routine must then be performed and confirmed by the ICV/ICB pair and the SIC re-analyzed before proceeding with analysis. Otherwise, the nonconformance issue is raised to the Department Supervisor and/or the QA Department.

9.7.2 Internal Standard

The internal standards are added prior to the nebulizer and corrects for intensity differences in the instrument response between the standard's and sample's matrix. They are monitored for any variation in response during the sample analyses and used to ratio the sample response to the internal standard response of the calibration blank. The ratio is applied to compensate for instrument conditions in the plasma or nebulization caused by the matrix. The internal standard is monitored for 50-150% recovery or laboratory generated control ranges difference from the calibration blank IS response to ensure the proper functioning of the internal standard introduction system and matrix interferences. If an injection falls outside of this acceptance range the sample or QC check is rerun once to check for an introduction error.

If a sample continues to fail it's to be run on successive increasing dilutions until the internal standards associated with the elements of interest are within range. If a QC check fails on the single rerun the analysis is stopped, the root cause investigated, corrected and the instrument re-calibrated/verified. The analysis begins again with all samples that were run after the last acceptable CCV/CCB pair.

9.7.3 LDR Check Solution: A multiple element or single element solution run at a point above the highest calibration standard under the same calibration used to quantify the associated sample data. The LDR check must be within +/-10% of the true value of each element of interest to be considered valid.

9.8 Method Sequence

- Calibration of instrument
- Initial Calibration Verification Standard
- Initial Calibration Blank
- Mixed SIC Solution
- Continuing Calibration Verification Standard
- Continuing Calibration Blank
- 10 samples
- Continuing Calibration Verification Standard

- Continuing Calibration Blank
- 10 Samples
- Continuing Calibration Verification Standard
- Continuing Calibration Blank

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been prefiltered and acidified will not need acid digestion. Samples which are not digested must be matrix matched with the standards.

NOTE: Sample digestates intended for Silver analysis must be analyzed as soon as possible.

10.1.2 Instrument Set-Up

Set up the instrument with proper operating parameters established as detailed below. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

Startup Procedures

For iCAP Duo 6500

- Turn on power to the chiller
- Click on ThermoSpec Icon; enter analyst initials in login screen
- Click on Plasma icon to start instrument
- Allow to warm up for 30 minutes
- Enter analytical workgroup number (obtained from LIMS) globally under the Instrument menu by selecting Tools, then Options, then Analyst.
- Click on the Sequence tab and enter the sequence by selecting New Autosampler Table, Add Sequence, Add # of spaces.
- Enter the sample locations and IDs
- Press Run Auto-Session button (▶) in menu bar.

- 10.1.2.1** Specific wavelengths are listed in Table 1. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality to the program and data user.

Operating conditions for axial plasma will vary from 1100 – 1500 watts forward power, 15-19 Liters/min argon coolant flow, 0.5 – 0.7 L/min argon nebulizer flow, 140 – 200 rpm pump rate and a default 1 minute preflush time and 10 second measurement time is recommended for all simultaneous instruments.

- 10.1.2.2** The plasma operating conditions need to be optimized prior to use of the instrument. This routine is not required on a daily basis, but only when first

setting up a new instrument or following a change in operating conditions. The following procedure is recommended or follow manufacturer's recommendations. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure.

- 10.1.2.2.1 The Thermo ICP's typically use a Meinhard Nebulizer. The nebulizer flow for each instrument is 1.0 +/- 0.2 mL/min.
 - 10.1.2.2.2 The 6500 Duo instruments automatically perform a wavelength check at start up without user interaction.
 - 10.1.2.2.3 The instrument operating condition finally selected as being optimum must provide the lowest reliable instrument detection limits and method detection limits.
 - 10.1.2.2.4 If either the instrument operating conditions, such as incident power or nebulizer gas flow rate are changed, or a new torch injector tube with a different orifice internal diameter is installed, the plasma and argon pressures must be reoptimized.
 - 10.1.2.2.5 After completing the initial optimization of operating conditions, but before analyzing samples, the laboratory must establish and initially verify an interelement spectral interference correction routine to be used during sample analysis. A general description concerning spectral interference and the analytical requirements for background correction in particular are discussed in the section on interferences. Criteria for determining an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls within $\pm \frac{1}{2}$ LLOQ. The upper control limit is the analyte instrument detection limit. Once established, the entire routine is periodically verified every six months. In between that time, IEC's are done on a need be basis per analyte. Only a portion of the correction routine must be verified more frequently or on a daily basis. Initial and periodic verification of the routine must be kept on file. Special cases where continual verification is required are described elsewhere.
- 10.1.2.3 Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.
- 10.1.2.3.1 Method detection limits must be established for all wavelengths utilized for each type of matrix commonly analyzed. The matrix used for the MDL calculation must contain analytes of known concentrations within 3-5 times the anticipated detection limit.
 - 10.1.2.3.2 Determination of limits using reagent water MDLs represent a best case situation and do not represent possible matrix effects of real world samples.

10.1.2.3.3 If additional confirmation is desired, reanalyze the seven replicate aliquots on two more non-consecutive days and again calculate the method detection limit values for each day. An average of the three values for each analyte may provide for a more appropriate estimate.

10.1.2.3.4 The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses with 10% of the true value of each element from a concentration standard at the upper limit of the range run on the same calibration as required by the sample responses above the calibration range. The range which may be used for the analysis of samples must be no more than 90% of the resulting data. Determined analyte concentrations that are above the upper range limit must be diluted and reanalyzed. The analyst must also be aware that if an inter-element correction from an analyte above the linear range exists, a second analyte where the inter-element correction has been applied may be inaccurately reported.

NOTE: Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self-absorption effects. These curves may be used if the instrument allows; however the effective range must be checked and the second order curve fit must have a correlation coefficient of 0.995 or better. Third order fits are not acceptable. These curves are much more sensitive to changes in operating conditions than the linear lines and must be checked whenever there have been moderate equipment changes.

10.1.2.4 The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

10.2 Initial Calibration

Calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 8.7. Flush the system with the calibration blank (Section 8.8.1) between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a calibration blank, RL standard and a high level standard. Calibration curve verification is accomplished through the analysis of the ICV, ICB and SIC standards.

10.3 Equipment Operation and Sample Processing

10.3.1 For all analytes and determinations, the laboratory must analyze an ICV (Section 8.9), and a calibration blank (ICB, Section 8.8.1), immediately following daily calibration.

A calibration blank (CCB, Section 8.8.1) and a calibration verification standard (CCV, Section 8.9) must be analyzed after every tenth sample and at the end of the sample run. Analysis of the calibration verification (CCV) must verify that the instrument is within 10% of the calibration with the relative standard deviation < 5% from replicate (minimum of three) integrations.

If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV/ICB, or CCV/CCB must be reanalyzed. The analysis

data for the calibration blank, check standard, and ICV or CCV must be kept on file with the sample analysis data.

- 10.3.2 Rinse the system with the rinse blank solution (Section 8.8.3) before the analysis of each sample. The suggested default rinse time is one minute. Each ICP instrument may establish a reduction in this rinse time through a suitable demonstration.
- 10.3.3 Dilute and reanalyze samples that exceed the linear range or use a calibrated alternate, less sensitive line for which quality control data is already established.
- 10.3.4 If less than acceptable accuracy and precision data are generated a series of tests are performed prior to reporting concentration data for analyte elements. At a minimum, these tests should be performed with each batch of samples prepared/analyzed with corresponding unacceptable data quality results. These tests, as outlined in Sections 10.3.5 and 10.3.6, will ensure that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.
- 10.3.5 **Post Digestion Spike Addition:** If the sample concentrations are insufficient to perform a dilution test a post digestion spike added to a portion of a prepared sample, or its dilution for the elements failing the matrix spike recoveries must be run, recovery limits equal to 75% to 125% of the known spike value. If the spike is not recovered within the specified limits If the post-digestion recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values
- 10.3.6 **Dilution Test:** If the analyte concentration is sufficiently high (minimally, a factor of 25 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution must agree within $\pm 20\%$ of the original determination. Elements that fail the dilution test are reported as estimated values.
- 10.3.7 **CAUTION:** If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

10.4 Continuing Calibration

- 10.4.1 Check calibration with an ICV following the initial calibration (Section 8.9). Verify calibration with the Continuing Calibration Verification (CCV) Standard (Section 8.9) at the end of the initial calibration sequence (ICV, ICB), after every ten samples, and at the end of an analytical run. Use a calibration blank (Section 8.8.1) immediately following daily calibration, after every 10 samples and at the end of the analytical run.
 - 10.4.1.1 The results of the ICV are to agree within 10% of the expected value, and CCVs are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument. Each may be rerun once to confirm or cure the initial failure.
 - 10.4.1.2 The results of the calibration blank should be below $\frac{1}{2}$ of LLOQ or RL (whichever is lower). If not, repeat the analysis and if the failure is repeated terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be performed before continuation of the run.

10.4.2 Verify the inter-element and background correction factors at the beginning of each analytical run. Do this by analyzing the SIC (Section 8.10). Results must be less than +/- LLOQ for all non-spiked elements.

10.4.3 When low-level sensitivity is required, a check standard at the requested limit of quantitation is analyzed to confirm the reported detection limit (RDL). This is performed on a project-by-project basis.

10.5 Preventive Maintenance

Whenever instrument maintenance is performed, it is noted in the instrument's Maintenance Logbook.

10.5.1 Daily

Inspect the nebulizer pump tubing from the Autosampler to the Nebulizer. Replace if necessary.

10.5.2 Monthly or as needed

Remove the torch, "shot glass", nebulizer and spray chamber. Clean each with 10% Nitric Acid and rinse with tap water. Coat the inside of the spray chamber and shot glass with concentrated Sulfuric Acid and soak for one hour, then rinse well with DI water. Soak the torch and nebulizer in aqua regia overnight, then rinse with DI water.

10.5.3 Every 6 months

Preventive Maintenance is performed by the Vendor or in-house personnel as follows:

- check the cooling system
- flush/refill the chiller with distilled water and antibacterial conditioner
- clean the instrument to regain intensity
- clean/replace air filters.

11. Data Evaluation, Calculations and Reporting

11.1 If dilutions were performed, the appropriate factors must be applied to sample values. All results must be reported with up to three significant figures.

11.2 Soil samples

Soil samples are calculated as follows:

$$A = \frac{\text{Sample weight (grams)}}{\text{Final Volume (mL)}}$$

$$B \text{ (concentration in mg/Kg)} = \frac{\text{Concentration of analyte (mg/L)}}{A}$$

11.2.1 Dry weight correction

The LIMS calculates the dry weight correction, however it is calculated as follows:

$$\text{Final concentration in mg/Kg dry weight} = \frac{\text{B}}{\% \text{ Solids}}$$

11.3 Liquid samples

Liquid samples are calculated as follows:

$$\text{Dilution Factor} = \frac{\text{Final Volume (mL)}}{\text{Sample Volume (mL)}}$$

$$\text{Final concentration in mg/L} = \text{Concentration of analyte (mg/L)} \times \text{Dilution Factor}$$

11.4 Calculations for Hardness

The method for determining hardness is to compute it from the results of separate determinations of Calcium and Magnesium on aqueous samples.

11.4.1 Total Hardness

$$\text{Total Hardness, mg equivalent CaCO}_3/\text{L} = [2.497 (\text{Ca, mg/L})] + [4.118 (\text{Mg, mg/L})]$$

11.4.2 Calcium Hardness

$$\text{Calcium Hardness, mg equivalent CaCO}_3/\text{L} = [2.497 (\text{Ca, mg/L})]$$

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

Also refer to Section 9 for Quality Control and acceptance criteria.

If the SIC is outside of the recovery window, then the standard is reanalyzed. If the standard failure continues, the IECs for the element/elements in question are reviewed and recalculated if necessary.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The raw data is noted. If the standard fails again, the problem must be found and corrected and the instrument is recalibrated. The ICV/ICB standard is reanalyzed and all previous data that had failed back to the previous passing CCV/CCB is reanalyzed.

The reanalysis procedure outline above is also conducted for a failing LCS or Method Blank; they may be rerun alone on the new or any subsequent passing bracket. The LCS or Method Blank do not qualify a bracket of samples but the batch run itself.

If the Matrix Spike does not meet acceptance criteria, a dilution test is performed. If the levels of the native sample is inadequate (see section 10.3.6) The RPD must be within 20% of the true value of the native sample. If the dilution test fails or the concentrations in the native sample are inadequate, the post spike is analyzed and evaluated (section 10.3.5). If these criteria are met, then the Matrix Spike data is reported, with the post spike narrated on the final report. If the post spike fails the data is reported as estimated.

If sample Duplicates are outside of the acceptance criteria, the analyst examines the sample for homogeneity. If the sample is not homogenous, this is narrated on the final report. Clean, homogenous samples are reanalyzed and if still outside of the acceptance limits, redigested and reanalyzed.

Sample nonconformance regarding a Matrix Spike recovery or a duplicate %RSD is narrated on the final report along with the corrective action(s) taken.

The mixed element SIC solution is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, instrument is recalibrated, verified with the ICV/ICB and the SIC is then re-analyzed. Alternatively, the LLOQ may be raised to twice the concentration observed in the SIC solution if approved by the Department Manger or QA Department and the level is below the regulatory action limit or project specific requirements. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions These may be present up to the concentration documented plus the LLOQ. If failure continues notify the Department Supervisor or Manager.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05 unless supersede within this SOP. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan
SOP #1732 MDL/LOD/LOQ Generation
SOP# 1739 IDC/DOC Generation
SOP# 1728 Waste Management and Disposal

16. Attachments

TABLE 1: Element Wavelengths

TABLE 2: Precision and Accuracy Acceptance Criteria

TABLE 3: Reporting Limits

TABLE 4: LCS and Matrix Spike Concentrations

**TABLE 1
ELEMENT WAVELENGTHS**

Element	6500 Duo wavelength (nm)
Pb	220.3
Se	196.0
Sb	206.8
As	189.0
Ba	455.4
Be	313.0
Cd	214.4
Co	228.6
Cu	324.7
Cr	267.7
Fe	259.9
Mn	257.6
Mo	202.0
Ni	231.6
Ag	328.0
Tl	190.8
V	292.4
Zn	206.2
Al	396.1
Ca	315.8
Mg	279.0
B	208.9
Si	212.9
Sn	189.9
Sr	421.5
Ti	334.9
Bi	223.0
Na	589.5
K	766.4
S	180.7

**TABLE 2
 PRECISION AND ACCURACY ACCEPTANCE CRITERIA**

Element	% Recovery LCS		Aqueous % Recovery MS		Soil % Recovery SRM *		Duplicate	
	Lower Control Limit	Upper Control Limit	Lower Control Limit	Upper Control Limit	Lower Control Limit	Upper Control Limit	Aqueous %RPD	Soil %RPD
Aluminum	80	120	75	125	29	171	20	20
Antimony	80	120	75	125	4	196	20	20
Arsenic	80	120	75	125	81	119	20	20
Barium	80	120	75	125	83	118	20	20
Beryllium	80	120	75	125	83	117	20	20
Boron	80	120	75	125	70	129	20	20
Cadmium	80	120	75	125	82	117	20	20
Calcium	80	120	75	125	83	117	20	20
Chromium	80	120	75	125	80	119	20	20
Cobalt	80	120	75	125	83	117	20	20
Copper	80	120	75	125	83	117	20	20
Iron	80	120	75	125	51	150	20	20
Lead	80	120	75	125	80	120	20	20
Magnesium	80	120	75	125	74	126	20	20
Manganese	80	120	75	125	83	117	20	20
Molybdenum	80	120	75	125	81	119	20	20
Nickel	80	120	75	125	82	117	20	20
Potassium	80	120	75	125	74	126	20	20
Sulfur	80	120	75	125	NA	NA	20	20
Selenium	80	120	75	125	80	120	20	20
Silica (SiO ₂)	80	120	75	125	NA	NA	20	20
Silver	80	120	75	125	66	134	20	20
Sodium	80	120	75	125	74	127	20	20
Strontium	80	120	75	125	80	120	20	20
Thallium	80	120	75	125	79	120	20	20
Tin	80	120	75	125	69	131	20	20
Titanium	80	120	75	125	82	118	20	20
Vanadium	80	120	75	125	79	121	20	20
Zinc	80	120	75	125	82	119	20	20

** Ranges of the SRM are presented as an example of a typical SRM; actual limits may vary by lot provided by the vendor.

**TABLE 3
 REPORTING LIMITS**

Element	Aqueous (mg/L)	Soil (mg/Kg)
ALUMINIUM	0.10	4.0
ANTIMONY	0.05	2.0
ARSENIC	0.005	0.40
BARIUM	0.01	0.40
BERYLLIUM	0.005	0.20
BORON	0.03	1.2
CADMIUM	0.005	0.40
CALCIUM	0.10	4.0
CHROMIUM	0.01	0.40
COBALT	0.02	0.80
COPPER	0.01	0.40
IRON	0.05	2.0
LEAD	0.01	2.0
MAGNESIUM	0.10	4.0
MANGANESE	0.01	0.40
MOLYBDENUM	0.05	2.0
NICKEL	0.025	1.0
POTASSIUM	2.5	100
SULFUR	0.25	10
SELENIUM	0.01	0.80
SILICA	0.50	20
SILVER	0.007	0.40
SODIUM	2.0	80
STRONTIUM	0.01	2.0
THALLIUM	0.02	0.80
TIN	0.05	4.0
TITANIUM	0.01	0.40
VANADIUM	0.01	0.40
ZINC	0.05	2.0

TABLE 4
LCS and Matrix Spike

Analyte	Liquid Concentration (mg/L)	Soil Concentration * (MS spike only) (mg/Kg)
Antimony	0.5	160
Arsenic	0.12	160
Barium	2.00	160
Beryllium	0.05	80
Cadmium	0.051	80
Chromium	0.20	160
Copper	0.25	160
Lead	0.51	160
Nickel	0.50	160
Selenium	0.12	160
Silver	0.05	40
Thallium	0.12	160
Zinc	0.50	160
Iron	1.00	800
Manganese	0.50	160
Calcium	10.0	800
Magnesium	10.0	800
Potassium	10.0	800
Sodium	10.0	800
Silica	1.0	800
Aluminum	2.00	800
Cobalt	0.50	160
Vanadium	0.50	160
Boron	1.0	NA
Molybdenum	1.0	NA
Titanium	1.0	NA

*MS spike of a solid based on 1.25g and a final volume of 50 mL.

Note: Solids LCS is an SRM with certified value provided by the vendor on a lot basis.

Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)

Reference Method No.: EPA 7471B

Reference: SW-846, Test Methods for Evaluating Solid Waste:
Physical/Chemical Methods, EPA SW-846, Update III,
February 2007.

1. Scope and Application

Matrices: Method 7471 is approved for measuring total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be subjected to an appropriate dissolution step prior to analysis. If this dissolution procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix.

Definitions: Refer to Alpha Analytical Quality Manual.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Mercury Analyzer and in the interpretation of Mercury data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability..

2. Summary of Method

Prior to analysis, the solid or semi-solid samples must be prepared according to the procedures discussed in this method.

Method 7471, a cold-vapor atomic absorption method, is based on the absorption of radiation at the 253.7-nm wavelength by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.1 Method Modifications from Reference

Alpha analyzes only one 0.3g aliquot of sample. The original method does not address the automated instrument procedure. A reduced volume of sample is digested in disposable digestion tubes on a hot block digester.

3. Reporting Limits

The reporting limit for this method is 0.08mg/Kg.

4. Interferences

Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/Kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.

Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg had no effect on recovery of mercury from spiked samples.

Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL).

Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

Because mercury vapor is toxic, precaution must be taken to avoid its inhalation.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples may be collected in plastic or glass containers.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are stored under refrigeration at 4°C and analyzed as soon as possible after collection. The samples have a 28-day holding time from the time of collection.

7. Equipment and Supplies

7.1 Instrumentation:

Perkin Elmer FIMS 100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. The PE FIMS is designed specifically

for the measurement of mercury using the cold-vapor technique with BOC (background offset correction) performed by a survey scan prior to each sample introduction. PE S10 autosampler is coupled to the instrument.

Cetac M-6100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. This instrument employs a reference cell off-set correction and full automation through the CETAC software. A Cetac ASX-260 autosampler is coupled to the instrument.

Nippon Instrument model# RA-4300A analyzer with integrated 80 position autosampler:

The instrument adds a stannous chloride (II) solution to the sample post digestion, the divalent mercury ion (Hg^{2+}) is reduced to zero-valent metallic mercury and turns into mercury gas by bubbling. $\text{Hg}^{2+} + \text{SnCl}_2 \rightarrow \text{Hg}^0 \uparrow$

After removing the acid mist and water vapor generated by bubbling with an electronic cooling unit, the instrument measures the absorbance of mercury at 253.7 nm absorption wavelength. It measures the known mercury amount, creates a calibration curve, and then calculates the mercury amount from the absorbance.

- 7.2 **Hot Block Digestor:** Environmental Express, 54 position, capable of maintaining a temperature of $95^\circ\text{C} \pm 3^\circ\text{C}$.
- 7.3 **Graduated cylinder.** Rinse once with 50% Nitric Acid, then rinse with reagent water prior to use.
- 7.4 **Volumetric Flasks, Class A, various volumes.** Rinse once with 50% Nitric Acid, then rinse with reagent water prior to use.
- 7.5 **Polypropylene Digestion Vessels:** 50 mL volume, with plastic screw caps
- 7.6 **Pump Tubing:** Environmental Express, three stop and two stop tubing in various IDs.
- 7.7 **PTFE membranes:** Pall TF1000 disks
- 7.8 **Dilution vials:** 20mL capacity, used when making analytical dilutions.
- 7.9 **Laboratory Wipes**
- 7.10 **Compressed Air**
- 7.11 **Whatman 41 or equivalent filter paper**
- 7.12 **Eppendorf pipets:** Accurate means to make trace standards

8. Reagents and Standards

- 8.1 **Reagent Water:** Reagent water is DI water shown to be interference free. All references to water in this method refer to reagent water unless otherwise specified.

- 8.2 Aqua regia:** Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO₃.
- 8.3 Concentrated Nitric Acid, (HNO₃):** Trace grade. Store at room temperature in the appropriately marked acid cabinet.
- 8.4 Concentrated Hydrochloric Acid, (HCl):** Trace grade. Store at room temperature in the appropriately marked acid cabinet.
- 8.5 Reductant, Stannous Chloride in 3% HCl:** This is the *reductant* for the Instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl and 11g SnCl₂ · 2H₂O. Mix to dissolve the solid and bring to volume with reagent water. Store at room temperature, prepare as needed.
- 8.6 Carrier, Hydrochloric Acid, 3%:** This is the *carrier* for the instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl (Section 8.4). Bring to volume with reagent water. Store at room temperature, prepare as needed.
- 8.7 Potassium permanganate, mercury-free, 5% solution (w/v):** Dissolve 5 g of potassium permanganate in 100 mL of reagent water. Store at room temperature.
- 8.8 Sodium chloride-hydroxylamine hydrochloride solution:** Dissolve 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride in reagent water and dilute to 100 mL. Store at room temperature.
- 8.9 Mercury stock solution, 1000ppm:** This solution is purchased commercially prepared with a certificate of analysis. Three solutions are purchased, each from a different source. Store at room temperature. Expires according to manufacturer's specifications.
- 8.9.1 10ppm Mercury Stock Standard:** To a 100mL volumetric flask, add 5mL of concentrated HNO₃ and 1.0mL of 1000ppm Mercury Stock Solution (Section 8.9). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.
- 8.9.1.1 0.1ppm Mercury Working Stock / Matrix Spike Solution:** To a 500mL volumetric flask, add 5mL of concentrated HNO₃ and 5 mL of 10ppm Mercury Stock Standard (Section 8.9.1). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.
- 8.9.2 10ppm Mercury ICV Stock Standard:** To a 100mL volumetric flask, add 5mL of concentrated HNO₃ and 1.0mL of Mercury Stock Solution (Section 8.9, from an alternate source than that used in Section 8.9.1). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.
- 8.9.2.1 0.3ppm Mercury ICV Working Stock:** To a 100mL volumetric flask, add 5mL of concentrated HNO₃ and 3.0mL of 10ppm Hg ICV Stock Standard (Section 8.9.2). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.
- 8.9.3 10ppm Mercury LCS Stock Standard:** To a 100mL volumetric flask, add 5mL of concentrated HNO₃ and 1.0mL of 1000ppm Mercury Stock Solution (Section 8.9, from an alternate source than that used in Sections 8.9.1 and 8.9.2). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.
- 8.9.3.1 0.1ppm Mercury LCS Working Stock:** To a 100mL volumetric flask, add 5mL of concentrated HNO₃ and 1.0mL of 10ppm Mercury LCS Stock Standard (Section

8.9.3). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.

8.10 SRM: Purchased from ERA in 300-500g lots.

9. Quality Control and Data Assessment

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

The Method Blank consists of the 0ppm standard as prepared in Section 10.1.2.1.1. Analyze one Method Blank per analytical batch of twenty samples or less. The Method Blank must be less than the Reporting Limit (RL). See Section 12.1 for corrective action.

9.1.1 PBS

A preparation blank is analyzed once per batch of twenty samples or less. It is prepared in the same manner as the 0ppm standard (Section 10.1.2.1.1). The PBS must be recovered within ± 0.2 $\mu\text{g/L}$.

9.1.2 ICB

The ICB is analyzed after the ICV, and is prepared in the same manner as the 0ppm standard (Section 10.1.2.1.1). The ICB must be recovered within ± 0.2 $\mu\text{g/L}$.

9.1.3 CCB

The CCB is analyzed after the CCV. It is prepared in the same manner as the 0ppm standard (Section 10.1.2.1.1). The CCB must be recovered within ± 0.2 $\mu\text{g/L}$.

9.2 Laboratory Control Sample (LCS)

The LCS is analyzed once per each analytical batch of twenty samples or less. It is prepared in the same manner as in Section 10.1.2.3. The LCS must be recovered within 80 – 120% of the true value. See Section 12.3 for corrective action.

9.3 Initial Calibration Verification (ICV)

The ICV is analyzed after the calibration curve. It is prepared in the manner specified in Section 10.1.2.2. The ICV must be recovered within $\pm 10\%$ of the true value. See Section 12.2 for corrective action.

9.4 Continuing Calibration Verification (CCV)

The CCV is analyzed after every ten analytical samples. It is prepared in the same manner as the 5.0ppb calibration standard (Section 10.1.2.1.5). The CCV must be recovered within 20% of the true value. See Section 12.2 for corrective action.

9.5 Matrix Spike

Analyze one matrix spike per twenty or less analytical samples. The recovery of the matrix spike must be between 80 – 120%. Calculate percent recovery using Section 11.2.

If the recovery of the matrix spike is out of range, a post-analytical spike is analyzed. Prepare the post analytical spike by adding 1.25mL of 0.020ppm Calibration Standard

(Section 10.1.2.1.6) and 1.25mL of the sample digestate to a 15mL centrifuge tube for a final concentration of 0.0105mg/L. Analyze the post spike as outlined in Section 10.3.

Calculate the post spike concentration as follows:

Post Analytical Spike Sample Concentration (mg/L) =

$$[\text{Sample Concentration (mg/L)} \times (0.5)] + 0.010\text{mg/L}$$

The percent recovery of the post-analytical spike must be between 75 – 125%.

See Section 12.4 for corrective action.

9.6 Laboratory Duplicate

Analyze one sample in duplicate per twenty or less analytical samples. The RPD between the sample and its duplicate must be $\leq 20\%$ (as calculated in Section 11.3). See Section 12.5 for corrective action.

9.7 Method-specific Quality Control Samples

None

9.8 Method Sequence

- Sample preparation
- Sample digestion
- Standards preparation:
 - Calibration standards
 - ICV standard
 - LCS standard
- Standards digestion
- Analysis of calibration standards
- Generation of calibration curve
- Analysis of samples and standards:
 - ICV
 - ICB
 - analytical samples
 - CCV
 - CCB
 - analytical samples
 - CCV
 - CCB
 - etc.

10. Procedure

10.1 Equipment Set-Up

- 10.1.1 Sample preparation:** Weigh a 0.3-g portion of untreated and homogenized sample and place in the bottom of a polypropylene digestion vessel. Record the weight in the laboratory notebook. NOTE: When preparing the batch, include one sample duplicate aliquot to be prepared in the same manner.

Add 2.5 mL of reagent water and 2.5 mL of aqua regia (Section 8.2). Heat 2 min on a hot block at 95°C +/-3°C. Cool; then add 15 mL reagent water, and 7.5 mL potassium permanganate solution (Section 8.7) to the digestion vessel. Wait 15 minutes to be sure the potassium permanganate is not exhausted (purple color disappears), if it does add additional potassium permanganate to all samples and QC until stable. Mix thoroughly and place in the hot block for 30 min at 95°C +/-3°C. Cool and add 3 mL of sodium chloride-hydroxylamine hydrochloride (Section 8.8) to reduce the excess permanganate.

CAUTION: Perform this addition under a hood, as Cl₂ could be evolved.

Bring up to a final volume of 50 mL with reagent water. Continue as described under Section 10.3.1.

10.1.2 Standard preparation: Standard preparation is performed each time samples are digested.

10.1.2.1 Calibration Standards

10.1.2.1.1 0 ppb: Add 10mL of reagent water to a polypropylene digestion vessel. This aliquot may be used for the CCB. Another separate aliquot is prepared for use as the ICB and the diluent for any samples with concentration greater than 90% the highest calibration standard used to determine the linear range.

10.1.2.1.2 0.5ppb: Add 10 mL of reagent water to a polypropylene digestion vessel. Using a volumetric pipet, add 0.25 mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vessel.

10.1.2.1.3 1.0ppb: Add 10 mL of reagent water to a polypropylene digestion vessel.. Using a volumetric pipet, add 0.5 mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vessel.

10.1.2.1.4 2.0ppb: Add 10 mL of reagent water to a polypropylene digestion vessel.. Using a volumetric pipet, add 1.0mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vessel.

10.1.2.1.5 5.0ppb/CCV: Add 10 mL of reagent water to a polypropylene digestion vessel.. Using a volumetric pipet, add 2.5 mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vessel.

10.1.2.1.6 10ppb: Add 10 mL of reagent water to a polypropylene digestion vessel.Using a volumetric pipet, add 5.0 mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vessel.

10.1.2.1.7 20ppb: Add 10 mL of reagent water to a polypropylene digestion vessel.Using a volumetric pipet, add 10.0 mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vessel.

10.1.2.2 ICV Standard, 3.0ppb: This standard is used for calibration verification.

Add 10.0 mL of reagent water to a digestion vessel. Using a volumetric pipet, add 0.5 mL of 0.3ppm Mercury Working Stock (Section 8.9.2.1) to the digestion vessel. Digest the ICV Standard as in Section 10.1.3.

10.1.2.3 LCS Standard, 1.0ppb: This standard is prepared and analyzed with each analytical batch.

Add 5.0mL of reagent water to a digestion vessel. Add 0.15g of SRM (Section 8.10). Digest the LCS Standard as in Section 10.1.3.

10.1.2.4 Matrix Spike, 0.001mg/L: Weigh two aliquots of the sample designated to be the batch matrix spike.

Add 10.0 mL of the reagent water to the digestion vessel containing the weighed sample aliquot. Add a 5 mL aliquot of 0.1ppm Mercury LCS Working Stock (Section 8.9.3.1). Digest the MS as in Section 10.1.3.

10.1.3 Standard Digestion:

To each standard (Sections 10.1.2.1 through 10.1.2.3), add 2.5 mL of reagent water and 2.5 mL of aqua regia (Section 8.2) and heat 2 min on the hot block at 95°C +/- 3°C. Allow the standard to cool; add 15 mL reagent water and 7.5 mL of KMnO₄ solution (Section 8.7) to each bottle and return to the hot block for 30 min. Cool and add 3 mL of sodium chloride-hydroxylamine hydrochloride solution (Section 8.8) to reduce the excess permanganate. Bring up to final volume of 50 mL with reagent water, continue as described in Section 10.3.3.

Note: Alternate volumes of standards may be made base on need as long as they are made with the same proportions as describe above.

10.2 Initial Calibration

Construct a calibration curve by plotting the absorbances of prepared standards (Section 10.1.2) versus micrograms of mercury. (See Section 11.1.) Determine the peak height of the unknown from the absorbance maxima on the spectrometer, and read the mercury value from the standard curve.

The curve correlation coefficient (cc) must be greater than or equal to 0.995 in order for the curve to be linear. If the correlation coefficient is less than 0.995, find and correct the problem. When the problem has been corrected, re-analyze either the previous standards or new standards. When the curve has generated an acceptable cc, then the analysis can continue with the ICV/ICB.

Analyze an Initial Calibration Verification Standard (ICV) (Section 9.3), an Initial Calibration Blank sample (ICB) (Section 9.1.2) at the start of the analytical run. The results for the ICV must be within 10% of the true value. If results are outside this range, refer to Section 12.2 for corrective action.

10.3 Equipment Operation and Sample Processing

10.3.1 Instrument Setup

10.3.1.1 Turn the instrument on. The autosampler will initialize itself.

10.3.1.2 Choose the instrument software from the desktop menu. The autosampler will initialize again.

FIMS 100 NOTE: The instrument must be turned on before the application is started. Otherwise, an error message will result.

10.3.1.3 Enter the appropriate fields for sample identification, and data storage.

10.3.1.4 Fill the carrier and reductant bottles.

10.3.1.4.1 The Carrier is 3% HCl (Section 8.6).

10.3.1.4.2 The Reductant is 1.1% SnCl₂ in 3% HCl (Section 8.5).

- 10.3.1.5 Allow the instrument to warm up while clearing samples. Samples that are cloudy or with particulate present after clearing must be filtered through Whatman 41 filter paper (Section 7.11) before analysis.
- 10.3.1.6 Place carrier uptake line and reductant uptake line.
- 10.3.1.7 Load carrier and reductant lines into pump magazines
- 10.3.1.8 Load the two waste lines into the pump magazines below the roller.
- 10.3.1.9 Lock the magazines into place.
FIMS100 only:
- 10.3.1.10 Remove the cap from the liquid/vapor separator and wipe dry with a Lab Wipe (Section 7.9). Compressed air (Section 7.10) through the vapor transfer line to dry it out.
- 10.3.1.11 Place a PTFE membrane (Section 7.7), rough side up, in the liquid/vapor separator; replace the cap and reattach the vapor transfer line to the sample absorption cell.
- 10.3.1.12 Adjust the gas flow by turning the black knob below the air flow meter to obtain a reading of just over 50.

10.3.2 Calibration and Sample Analysis

- 10.3.2.1 The instrument will now run the calibration standards; verify a CC of 0.995 or better before proceeding with the ICV and ICB. Ten analytical samples, a CCV and CCB, ten analytical samples, CCV, CCB, etc. The CCBs and CCVs must be recovered within the proper ranges (Sections 9.4 and 9.1.3) for analysis to continue.
- 10.3.2.2 If the sample result is beyond 90% of the concentration of the highest point on the calibration curve or LDR study used to establish the linear range, dilute the sample extract with a portion of one of the prepared blanks (ICB, CCB or PBS) to produce an analytical result that is within the range.

10.3.3 Instrument Shut Down

- 10.3.3.1 When analysis is complete place reagent uptake lines in a beaker of DI water.
 - 10.3.3.1.1 Continue to run the pumps for several minutes to flush reagents out of the lines.
 - 10.3.3.1.2 Continue to run the pumps for several more minutes to flush the lines thoroughly with DI water.
- 10.3.3.2 Pull the reagent uptake lines out of the DI water beaker to allow the pump to draw air completely through the lines.
- 10.3.3.3 Unlock the top and bottom pump magazines and remove tubing from the magazines.
- 10.3.3.4 Exit from the software application.

Note: This procedure must be followed every time the instrument is shut down to prevent water or reagents from being draw back into the instrument which can cause severe damage.

10.3.3.4.1 Dump the samples and instrument waste in the Metals/WetChem waste drum located in the transfer room only after all data has been processed through final review and package generation.

10.4 Continuing Calibration

After every 10 samples, analyze a Continuing Calibration Verification Standard (CCV), and a Continuing Calibration Blank sample (CCB). Determine the concentrations from the calibration curve. The results for the CCV must be within 20% of the true value.

10.5 Preventive Maintenance

Preventative maintenance is conducted per the manufacturer's instructions. All preventative maintenance is recorded in the Instrument Maintenance Logbook.

11. Data Evaluation, Calculations and Reporting

11.1 Calculate Mercury Concentration From the Daily Calibration Curve

The curve is generated utilizing a straight-line equation defined as:

$$A = k_1 + k_2C$$

Where:

A = Average peak height of the sample/standard integrations
C = Sample/Standard Concentration, $\mu\text{g/L}$
 k_1 = y-intercept
 k_2 = slope

The instrument will plot peak height against concentration ($\mu\text{g/L}$). The result is generated in $\mu\text{g/L}$. This value is divided by 1000 to convert the units to mg/L . The mg/L units are converted to mg/Kg by multiplying by L/Kg . A dilution factor (DF) is applied if necessary. The Result is then divided by the % Total Solids prior to release to the client.

$$\text{Result, mg/Kg} = (\text{concentration, } \mu\text{g/L}) \times (1\text{mg}/1000\mu\text{g}) \times (\text{DF}) \times (\text{L/Kg})$$

Where:

$$\text{L/Kg} = \frac{\text{Final volume of digestate, in L}}{\text{Weight of original sample, in Kg}}$$

11.2 Calculate Percent Recovery for the Matrix Spike corrected for concentrations measured in the unfortified sample. Percent recovery is calculated using the following equation:

$$\% \text{ Recovery} = \frac{(C_m - C)}{s} \times 100$$

Where:

C_m = measured Mercury in the fortified sample
C = measured native mercury sample concentration
S = concentration equivalent of spike added to sample

11.3 Calculate the Relative Percent Difference (RPD) for each Duplicate of the initial quantitated concentration (IC) and duplicate quantitated concentration (Dc) using the following formula:

$$RPD = \frac{|(IC - Dc)|}{\{(IC + Dc) / 2\}} \times 100$$

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

12.1 Method Blank Failure

When a prep blank mercury concentration is $\geq 10\%$ of the mercury concentration determined for any associated sample, or is greater than 2.2x the MDL value (whichever is greater), the entire batch associated with the prep blank must be redigested.

12.2 ICV / CCV Failure

If the ICV %Recovery is outside of acceptance criteria, analysis is terminated until the problem is found and corrected. If the CCV %Recovery is outside of acceptance criteria, all samples analyzed since the last acceptable CCV must be reanalyzed following correction of the problem.

12.3 LCS Failure

If the LCS is not recovered within acceptance criteria, the associated batch and another LCS must be redigested (Section 10.1).

12.4 Matrix Spike/Post Digestion Spike Failure

If the recovery of the matrix spike is outside of the acceptance criteria of 80 – 120%, a post digestion spike is performed (Section 9.5). If the post digestion spike is beyond 75 – 125%, the sample and its spike are redigested (Section 10.1).

12.5 Duplicate Failure

If the RPD between the sample and its duplicate is greater than 20%, visually evaluate the sample matrix. If the sample matrix appears clean, the sample and its duplicate are removed from the batch and redigested (Section 10.1). If the matrix appears problematic, the sample digestate may be diluted and reanalyzed, or a narrative included with the data to explain the matrix problem.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

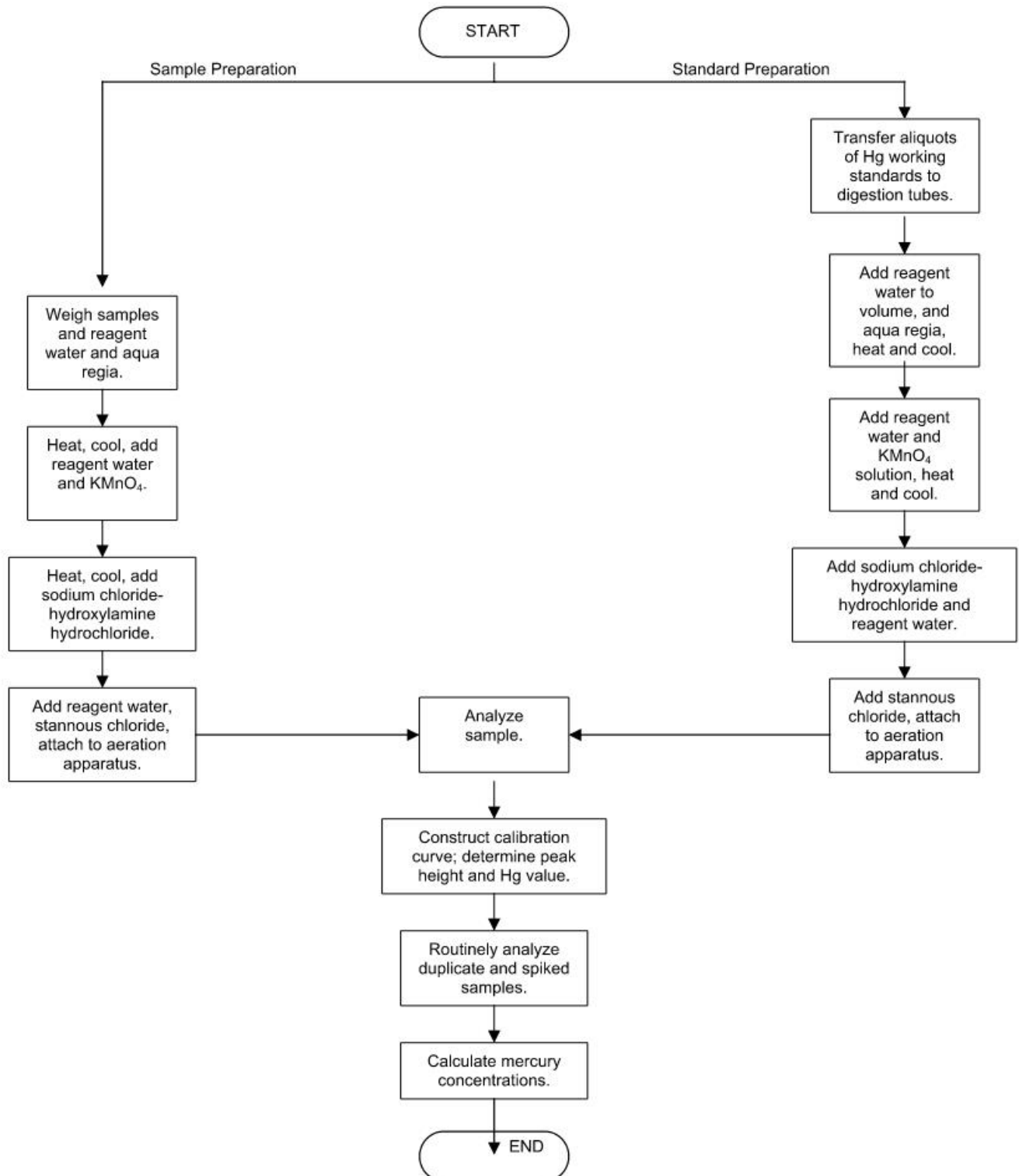
15. Referenced Documents

Chemical Hygiene Plan
SOP/1732 DL/LOD/LOQ Generation
SOP/1739 IDC/DOC Generation
SOP/1797 Waste Management and Disposal SOP

16. Attachments

FIGURE 1: Flow Chart for Method 7471B

Figure 1
Method 7471B Flow Chart



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TCLP/SPLP Extraction - Volatile Organics EPA 1311/1312

Reference: EPA 1311, EPA 1312 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Update I, July 1992.

1. Scope and Application

Matrices: The TCLP/SPLP extraction process is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

Definitions: Refer to Alpha Analytical Quality Manual.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of experienced analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

For liquid wastes, (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP/SPLP extract.

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately, and the results are mathematically combined to yield a volume-weighted average concentration.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The analytical method detection limits determined by the laboratory are on file for review. See the 8260 SOP 32639.

4. Interferences

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. This includes wearing personal protective equipment such as a lab coat, safety glasses, gloves and respirator (as necessary).

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples are collected in Teflon-lined septum capped vials and stored at $4 \pm 2^\circ \text{C}$). Samples are opened only immediately prior to extraction.

SPLP samples may be collected in 25g En Core sampling device.

6.2 Sample Preservation

Preservatives shall not be added to samples before extraction. For SPLP samples arriving in En Cores they must be extruded and tumbled within 48 hours of collection. Extrude the entire sample, but no more 25g since the vessel can only hold 500 mLs of fluid.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) must be extracted.

TCLP/SPLP extracts are prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.

Samples must undergo TCLP/SPLP extraction within the time periods listed in Table 1.

7. Equipment and Supplies

7.1 Agitation Apparatus: End-over-end 30 ± 2 rpm.

7.2 Zero Headspace Extraction Vessel

7.3 Glass Fiber Filters: 0.6 to 0.8 μm , 90mm

7.4 50mL Gastight Syringe

7.5 Graduated Cylinder: 500 mL, 1L

7.6 Laboratory Balance: 0.01g tolerance

7.7 Pressure Gauge: 0-60 psi

7.8 Pressure Filtration Device

7.9 Drying Oven: 100 ± 2 °F

7.10 Fluid Metering Pump

7.11 pH Meter: The meter should be accurate to + 0.05 units at 25°C

8. Reagents and Standards

8.1 Analytical Standards: The standards shall be prepared according to the appropriate volatile organic analysis method 8260 SOP 32639.

8.2 Hydrochloric Acid (HCl), 1N: Dilute 83 mL conc. HCl to 1L with DI water.

8.3 Nitric Acid (HNO₃), 1N: Dilute 64 mL conc. HNO₃ to 1L with DI water.

8.4 Sodium Hydroxide (NaOH), 1N: Dissolve 40 gs NaOH in 1L of water.

8.5 Glacial acetic acid: CH₃COOH.

8.6 Ottawa sand: SiO₂

8.7 Extraction Fluid #1: Add 5.7 mL glacial acetic acid to 500 mL of DI water, add 64.3 mL of 1N NaOH, and dilute to a volume of 1L. Check pH of solution which must be 4.93 ± 0.05 when correctly prepared. The laboratory reagent number is referenced in the TCLP sample logbook.

NOTE: This extraction fluid is monitored frequently for impurities. The pH is checked prior to use to ensure that this fluid is prepared accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

8.8 Extraction Fluid #2: Organic-free reagent water.

8.9 Methanol: Purge and Trap Grade or equivalent. Store in flammables cabinet.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

A minimum of **one blank** must be analyzed for every batch or 20 extracts, whichever comes first. The blank is prepared by weighing 20g of Ottawa sand (SiO₂) and utilizing the same extraction fluid as used for the samples. The blank is then analyzed following the same procedures as analytical samples.

9.2 Laboratory Control Sample (LCS)

A laboratory control sample is not extracted. Refer to the 8260 procedure for the LCS information.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike

Upon a client's request, a spike solution composed of all regulated compounds can be added to the TCLP/SPLP extract before analysis. See Method 8260 for more information.

9.6 Laboratory Duplicate

Not Applicable.

9.7 Method-specific Quality Control Samples

Not Applicable.

9.8 Temperature Measurement Guidelines

The ambient temperature of the tumbler rooms must be monitored from the beginning to the end of the TCLP, SPLP and ZHE extractions by use of a MIN/MAX thermometer. The temperature of the room must stay within the required range of 21-25 deg C during the entire extraction period.

If a determination is made that a MAX/MIN thermometer reading is outside of the acceptance range, it must be confirmed by comparison to the data logger records. This is the only way to make certain the temperature reading is accurate.

Any MAX/MIN temperature reading that exceeds acceptance criteria must be reviewed by the Department Manager, Team Leader, or QA.

If a temperature exceedance is confirmed, the following narrative must be included on the Alpha job report.

Narrative # 2068

TCLP / SPLP TEMPERATURE EXCEEDANCE

[list] [qcsamplist]: The ambient temperature of the area in which the TCLP/SPLP extraction was performed was outside the required 21-25 degrees C temperature range.

9.9 Method Sequence

The extraction sequence is:

- Disassemble ZHE; clean all parts with warm H₂O and soap; dry.
- Check sample; ensure there is enough sample for analysis.
- Determine %solids of the waste.
- Extract the TCLP/SPLP.
 - Record the temperature of the extraction room.
 - Following extraction, perform a leak test on the ZHE canisters.
- Draw sample with syringe from ZHE after extraction.
- Prepare sample for 8260 analysis

10. Procedure

10.1 Equipment Set-up

10.1.1 Preliminary Evaluation

10.1.1.1 Determine % Solids :

10.1.1.1.1 If waste contains no free liquid, proceed to Section 10.1.1.2 where the waste is the solid portion.

10.1.1.1.2 If the waste contains less than 0.5% solids, proceed to Section 10.3.2.

10.1.1.1.3 Multiphasic waste - separate liquid and solid portion

The following is performed by the Login Department.

10.1.1.1.3.1 Using a pressure filtration device pre-weigh the filter and the container to receive the initial filtrate. Record the weight in the laboratory notebook.

10.1.1.1.3.2 Assemble the pressure filter according to the manufacturer's directions.

10.1.1.1.3.3 Weigh a subsample of the waste (100 g minimum) and record the weight.

10.1.1.1.3.4 Transfer the waste sample to the filter holder spreading evenly over the filter.

10.1.1.1.3.5 Gradually apply a gentle pressure of 1-10psi until the pressurizing gas moves through the filter, proceed to Section 10.1.1.1.3.8.

- 10.1.1.1.3.6 If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10psi increments.
- 10.1.1.1.3.7 Stop the filtration when pressurizing gas passes through the filter, or 50psi is reached and no additional liquid passes through the filter in any 2 minute interval.
- 10.1.1.1.3.8 The material in the filter holder is defined as the SOLID PHASE of waste and the filtrate is defined as the LIQUID PHASE.

Note: the SOLID PHASE may appear liquid in some samples. Do Not replace original filter. Use only one filter.

- 10.1.1.1.3.9 Weigh the filtrate filled container and record the weight in the laboratory notebook. Calculate the weight of the LIQUID PHASE as follows:

$$W_{LP} = W_F - W_C$$

Where:

W_{LP} = Weight of the LIQUID PHASE

W_F = Weight of the filtrate filled
container

W_C = Weight of container

- 10.1.1.1.3.10 Calculate the weight of the SOLID PHASE using the following formula:

$$W_{SP} = W_W - W_{LP}$$

Where:

W_{SP} = Weight of the SOLID PHASE

W_W = Weight of the waste sample

W_{LP} = Weight of the LIQUID PHASE

- 10.1.1.1.3.11 Calculate the percent solids using the following formula:

$$\%Solids = \frac{W_{sp}}{W_w \times 100}$$

Where:

W_{SP} = Weight of the SOLID PHASE

W_W = Weight of the waste sample

- 10.1.1.1.3.12 If the % Solids determined in Section 10.1.1.1.3.11, is less than 0.5%, then proceed to Section 10.3.2, where the filtrate is the TCLP/SPLP extract.
- 10.1.1.1.3.13 Remove the solid phase and filter from the filtration apparatus, dry at $100 \pm 2^\circ$ F, cool and weigh. Record the weight in the laboratory notebook.
- 10.1.1.1.3.14 Calculate the percent dry solids using the following formula:

$$\% \text{ dry solids} = \frac{W_{DW} - W_F}{W_W \times 100}$$

Where:

W_{DW} = Weight of dried waste + filter

W_F = Weight of the filter

W_W = Weight of waste sample

10.1.1.1.3.15 If the % dry solids determine above, 10.1.1.3.14, is less than 0.5%, then proceed to Section 10.3.2, where the filtrate is the TCLP extract.

10.1.1.2 Determine whether waste requires particle size reduction

The following is performed by the Metals Prep Department.

Using fresh portion of the waste, obtain another solid phase of the waste sample by pressure filtration as determined in Sections 10.1.1.1. Use enough wet sample to obtain a dry portion for tumbling: at least 100 g. Evaluate the solid phase for particle size. Particle size reduction is required unless the solid has a surface area greater to 3.1 cm² per g, or is smaller than 1 cm in its narrowest dimension.

10.1.1.3 Determine Compatibility of Liquids

The following is performed by the Metals Prep Department.

If a LIQUID PHASE was filtered, check its compatibility with the extraction fluid. If the liquids are compatible (miscible), then only one TCLP/SPLP extract needs to be analyzed. If the liquids are not compatible, then the two liquids will have to be analyzed separately. Record in the laboratory notebook.

10.2 Initial Calibration

Not Applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 After the preliminary examination has been completed, the sample used in the following step comes from supplied VOA sample vials. The extraction for Volatile Organics is accomplished in a zero headspace extractor, ZHE.

10.3.2 Waste containing less than 5% dry solids

10.3.2.1 For waste containing less than 5% dry solids, weigh out up to a 500 g subsample and record its weight. Filter enough of the sample so that two 40 mL VOA vials may be filled with the filtered liquid. The liquid portion of the waste, after filtration, is defined as the TCLP/SPLP extract for volatile organic analysis. Proceed to Section 10.3.4.20.

10.3.3 100% Solid Waste

10.3.3.1 If particle size reduction is required (Section 10.1.1.2.), prepare the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 10.1.1.2. Waste and appropriate reduction equipment should be refrigerated to 4 ± 2° C prior to particle size reduction.

10.3.3.2 Weigh 20 gs of prepared waste and record the weight. If a 25 g Encore is supplied extrude and record the weight.

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- 10.3.3.3 Quantitatively transfer the sample to the ZHE.
- 10.3.3.4 Assemble the ZHE according to manufacturer's instructions. Add the appropriate type and amount of fluid using the fluid metering pump. The fluid added is 20 times the amount of sample. For example, for a 20 g sample, add 400 mL of extraction fluid for TCLPs or 400 mL of organic free DI water for SPLPs. For a 25 g sample, 500 mLs of the appropriate fluid is added.
- 10.3.3.5 Position the ZHE in the vertical position with the liquid inlet/outlet valve on top and open.
- 10.3.3.6 Gradually apply pressure to the ZHE and bleed out any headspace.
- 10.3.3.7 Proceed to Section 10.3.4.16.

10.3.4 Waste containing greater than 5% dry solids

- 10.3.4.1 Determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge the ZHE} = \frac{20 \times 100}{\% \text{ solids}}$$

Weigh out a subsample of the waste of the appropriate size and record the weight

- 10.3.4.2 If particle size reduction of the solid portion of the waste was required in Section 10.1.1.2, prepare the waste for extraction by crushing, cutting or grinding the solid portion of the waste to a surface area or particle size as described in Section 10.1.1.2. Waste and appropriate reduction equipment should be refrigerated to $4 \pm 2^\circ \text{C}$ prior to particle size reduction.
- 10.3.4.3 Quantitatively transfer the entire sample quickly to the ZHE. Assemble the ZHE in accordance with the manufacturer's instructions.
- 10.3.4.4 Place the device in the vertical position with the inlet/outlet valve on top and open.
- 10.3.4.5 Gradually apply pressure to the ZHE and bleed out any headspace quickly.
- 10.3.4.6 At the first appearance of liquid from the liquid inlet/outlet valve quickly close the valve.
- 10.3.4.7 Attach a 50mL gastight syringe to the liquid inlet/outlet valve and open the valve.
- 10.3.4.8 Begin applying gentle pressure to force the liquid phase of the sample into the filtrate collection container. When no additional liquid has passed through the filter in any 2-minute interval, proceed to Section 10.3.4.9.
- 10.3.4.9 Stop filtration by closing the liquid inlet/outlet valve.
- 10.3.4.10 Disconnect and weigh the 50mL gastight syringe. Record the weight in the laboratory notebook.
- 10.3.4.11 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.
- 10.3.4.12 If the liquid phase is compatible with the extract (Section 10.1.1.3), store the filtrate at $4 \pm 2^\circ \text{C}$ until analysis. Then combine and analyze the filtrate as one extract.
- 10.3.4.13 If the liquid phase is not compatible with the solid extract, then analyze the filtrates separately and mathematically combine the results (Section 10.3.4.22).

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- 10.3.4.14** Determine the weight of extraction fluid #1 for TCLP or organic free DI water for SPLP to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = 20(W_S + W_T - W_{TF})$$

Where:

$$\begin{aligned} W_S &= \text{Weight Sample} \\ W_T &= \text{Weight of pre-weighed 50mL syringe} \\ W_{TF} &= \text{Weight of filtrate in 50mL syringe} \end{aligned}$$

For 100% solid samples, the above formula reduces to:

$$\text{Weight of extraction fluid} = 20 \times \text{weight of sample}$$

- 10.3.4.15** Add extraction fluid by backing piston off the filter and sucking fluid into the ZHE. Do not open the ZHE.
- 10.3.4.16** Re-pressurize the ZHE to 15 psi (1.03 bar) and place in the rotary agitation apparatus. Rotate at 30 ± 2 rpm for 18 ± 2 hours.
- 10.3.4.17** Post-extraction measure the final pressure of the ZHE canister. If pressure has decreased below 15 PSI the sample must be reset.
- 10.3.4.18** Following the agitation period separate the material in the extractor vessel into its liquid and solid phases.
- 10.3.4.18.1** 100% solid waste samples: Filter enough liquid phase to fill two 40 mL vials. The filtrate is defined as the TCLP/SPLP extract. Proceed to Section 10.3.4.20.
- 10.3.4.18.2** Compatible liquid phases: Filter the entire extract into a 50mL gastight syringe. Transfer the contents of the syringe into 40mL VOA vials. Combine the extracts (Section 10.3.4.22) and analyze together.
- 10.3.4.18.3** Non-compatible liquid phases. Filter a sufficient quantity of the solid extract to fill two 40 mL VOA vials and analyze. The two liquids are collectively defined as the TCLP/SPLP extract and results are mathematically combined after individual analysis.
- 10.3.4.19** Following collection of the TCLP/SPLP extract immediately prepare the extract for analysis and store with minimal headspace at $4 \pm 2^\circ\text{C}$ until analyzed.
- 10.3.4.20** Follow the volatile organic analyses method 8260 SOP 32639 to analyze the TCLP/SPLP extract.
- 10.3.4.21** If the individual liquid phases (i.e. non-compatible liquids) are analyzed separately, determine the volume of the individual phases, conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:

$$\begin{aligned} V_1 &= \text{Volume of the first phase, (L)} \\ C_1 &= \text{Analyte concentration first phase, (mg/L)} \\ V_2 &= \text{Volume of the second phase, (L)} \end{aligned}$$

C_2 = Analyte concentration second phase, (mg/L)

10.4 Continuing Calibration

Not Applicable.

10.5 Preventive Maintenance

Tumbler rotation speed is verified annually and documented in the TCLP Annual Calibration Log, Form No.: 13855.

Balances are calibrated semi-annually by an instrument service company. Certificates are kept on file.

ZHEs are cleaned after extraction in soapy water. Filter screens and o-rings may be changed if they show degradation or the extraction vessel can't maintain pressure. Extremely contaminated vessels maybe further solvent rinsed with methanol

11. Data Evaluation, Calculations and Reporting

Calculations are included in Section 10.

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

Holding time exceedance, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

Not Applicable.

13.2.2 Continuing (DOC)

Not Applicable.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

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15. Referenced Documents

2121 Chemical Hygiene Plan
1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP
1739 Demonstration of Capability (DOC) Generation SOP
1728 Hazardous Waste Management and Disposal SOP
32639 8260 SOP
13855 TCLP Annual Calibration Form

16. Attachments

Table1: Maximum Holding Times
Table 2: TCLP Analyte List

Table 1

MAXIMUM HOLDING TIMES

Sample Maximum Holding Times (Days)

	From: Field collection	From: TCLP/SPLP extraction	From: Preparative extraction	
	To: TCLP/SPLP extraction	To: Preparative extraction	To: Determinative analysis	Total Elapsed Time
Volatiles	14/48 (hrs.)	N/A	14	28
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Metals, except mercury	180	N/A	180	360

Table 2

TCLP Analyte List

Analyte	CAS No.
1,1 - Dichloroethene	75-35-4
1,2 - Dichloroethane	0107-06-02
1,4 - Dichlorobenzene	106-46-7
2-Butanone	78-93-3
Benzene	71-43-2
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
Tetrachloroethene	127-18-4
Trichloroethene	79-01-6
Vinyl chloride	75-01-4

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TCLP Extraction

Metals and Semi-Volatile Organics

Reference Method: **EPA 1311**

Reference: **SW-846**, Test Methods for Evaluating Solid Waste,
Physical/Chemical Methods, Update I, July 1992

State of Connecticut, DEP, TCLP by SW-846 Method 1311, Version 2.0,
December 2006

1. Scope and Application

The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Extractions Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

For liquid wastes, (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

For additional detailed instruction, see TCLP and SPLP Work Instruction (ID# 17618).

2.1 Method Modifications from Reference

- 10.1.1.3.3 – Shaker table is used instead of stir bars. Samples not covered when shaking.
- 8.2 – 4N NaOH is used instead of 1N NaOH.

3. Reporting Limits

The Reported Detection Limit is determined by the amount of sample used for preparation. Therefore a review of Client requirements for Reporting Limits is necessary prior to sample preparation. Refer to analytical method SOPs.

4. Interferences

- 4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment must be scrupulously cleaned, following the Organic Extraction Glassware Cleaning and Handling SOP/1953 and Work Instruction 10995, Solvent rinsing guide.
- 4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.
- 4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.
- 4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. This includes wearing personal protective equipment such as a labcoat, safety glasses, gloves and respirator (as necessary).

- 5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.
- 5.2 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.
- 5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.
- 5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

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- 5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.
- 5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.
- 5.7 All Field Samples must be opened and handled in a hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass containers. Soils are collected in 8oz amber glass jars, and aqueous samples are collected in 500mL amber glass jars.

6.2 Sample Preservation

Preservatives are not added to samples before extraction.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples may be refrigerated at $4 \pm 2^{\circ}\text{C}$, unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

Samples must undergo TCLP extraction within the time periods listed in Table 1.

7. Equipment and Supplies

7.1 **Agitation Apparatus:** End-over-end 30 ± 2 rpm. Calibrated Quarterly.

7.2 **Vacuum Filtration Unit**

7.3 **0.6 to 0.8 μm Glass Fiber Filter Paper:** 70 mm, 90 mm, and 120 mm, and acid prewashed 47 mm for all metals analysis.

7.4 **2 L Glass/Plastic-coated Bottles**

7.5 **7 L Plastic Extraction Vessel:** For Lamp Extraction

7.6 **pH Meter:** +/- 0.01 units resolution

7.7 **pH test paper:** 0-14 pH range

7.8 **Laboratory Balance:** +/- 0.1 g tolerance

7.9 **250 mL Beaker or equivalent**

7.10 **Watch Glass:** Used with Beaker (Section 7.8)

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7.11 Stir Bars

7.12 Hot Plate: 1x1 ft (120 V) Barnstead-Thermolyne – Type 2200

7.13 2 L Graduated Cylinder Class A

7.14 250 mL Graduated Cylinder Class A

7.15 Teflon coated Sieve: 9.5 mm (0.375-inch)

7.16 Oven: Capable of maintaining $100 \pm 20^{\circ}\text{C}$

7.17 Automatic Shaker Table

7.18 Vessel to store fluid #1

7.19 Vessel to store fluid #2

7.20 Calibrated thermometer: Temperature calibration point 50°C . Range: $0-100^{\circ}\text{C}$.

7.21 Bottle Top Dispenser: 0-5 mL, 0-100 mL

7.22 1 L Amber Bottle: Thermo Scientific, Item # 341-0950, Jar Tall Amber WM.

7.23 120 mL Plastic Bottle: Greenwood, Product # 07-120WMF24503, Natural HDPE WM Packer assembled w/38-400 F-217 Lined Cap.

7.24 250 mL Plastic Bottle: Greenwood, Product # 07-250OB45F22603, Natural HDPE WM Oblong Bottle Assembled w/45-400 F-217 Lined Cap.

8. Reagents and Standards

8.1 Hydrochloric Acid: 1N: Dilute 83 mL conc. HCl to 1 liter with reagent water. Store at room temperature; expires one year from date of prep.

8.2 Sodium Hydroxide: 4N: Dissolve 160 grams NaOH in 1 liter of reagent water. If larger volumes are required, prepare using similar ratios. Store at room temperature; expires one year from date of prep.

8.3 Glacial acetic acid: CH_3COOH . Store at room temperature; expires according to manufacturer's specifications.

8.4 Reagent Water, (ASTM Type II): All references to water within this SOP refer to reagent water unless otherwise specified. Reagent water is interference-free.

8.5 Extraction Fluid #1: Add 5.7 mL glacial acetic acid to 500 mL of reagent water, add 64.3 mL of 4N NaOH, and dilute to a volume of 4 liters. Check the pH of the solution after preparation and record the value in the Extraction Fluid Logbook. The pH is within 4.93 ± 0.05 when correctly prepared. In addition, the pH of the fluid is verified prior to each use and the pH recorded in the extraction logbook. Store at room temperature; no expiration date, however the pH must be maintained.

NOTE: Larger volumes or extraction fluid may be prepared, provided the ratios remain the same. For example, a typical preparation of 200 L of Extraction Fluid #1:

1140 mL Glacial Acetic Acid + 3215 mL 4N NaOH diluted to 200 L with reagent water

8.6 Extraction Fluid #2: Dilute 5.7 mL glacial acetic with reagent water to a volume of 1 liter. Check pH of solution which should be 2.88 ± 0.05 when correctly prepared. Record the pH in the Extraction Fluid Logbook.

The pH is checked prior to use to ensure that this fluid is prepared accurately. If the pH is not within the above specifications, the fluid is discarded and fresh extraction fluid prepared. Store at room temperature; no expiration date, however the pH must be maintained.

NOTE: Larger volumes of extraction fluid may be prepared, provided the ratios remain the same. For example, a typical preparation of 20L or 50L Extraction Fluid #2:

19,886 mL of reagent water + 114 mL Glacial Acetic Acid = 20 L of fluid.

49,715 mL of reagent water + 285 mL Glacial Acetic Acid = 50 L of fluid

8.7 Trace Nitric Acid (1:1 HNO₃): 500 mL HNO₃ diluted to 1 liter with DI water. Store at room temperature in hood. Expires 3 months from prep.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A minimum of **one blank** (using the same extraction fluid lot number as used for the samples) must be prepared and analyzed for every 20 extracts or every 48 hours, whichever comes first. The Blank vessel is a randomly selected container from the stock and is not one designated for blank use only.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Determine analysis to be performed (Metals, ABN, Pest, PCB, Herbicides)
- Check sample; ensure there is enough sample for the extraction
- Determine the %solids of the waste sample

- Determine particle size reduction requirement
- Determine the extraction fluid to be used
- Extract the sample for TCLP
- Filter the extract.
- Aliquot extract to proper preparation departments
- Prepare extracts according to analytical protocols

10. Procedure

10.1 Equipment Set-up

10.1.1 Preliminary Evaluation

Perform preliminary TCLP evaluations on a minimum of 100g aliquot of waste. If there is limited sample volume, the chemist must contact the project manager. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include:

- (1) determination of the percent solids (Section 10.1.1.1);
- (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Section 10.1.1.2);
- (3) determination of whether the solid portion of the waste requires particle size reduction (Section 10.1.1.2); and
- (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 10.1.1.3)

10.1.1.1 Determine % Solids

- 10.1.1.1.1** If waste contains no free liquid, proceed to Section 10.1.1.2 where the waste is the solid portion. If the waste contains less than 0.5% solids, proceed to Section 10.1.1.1.2.

10.1.1.1.2 Multiphasic waste - separate liquid and solid portion

- 10.1.1.1.2.1** Pre-weigh a filter. Pre-weigh a beaker or graduated cylinder that will hold the initial filtrate. Pre-weigh empty filtrate container. Record weights in the extraction logbook.
- 10.1.1.1.2.2** Assemble the vacuum filtration unit.
- 10.1.1.1.2.3** Weigh a subsample of the waste (100 g minimum), or pour 100 mL of the sample into a cylinder, and record the weight or volume in the extraction logbook.
- 10.1.1.1.2.4** Transfer the waste sample to the filter holder, spreading evenly over the filter.
- 10.1.1.1.2.5** Allow enough time for the liquid to pass through the filter (at least 15 minutes).

- 10.1.1.1.2.6 The material in the filter holder is defined as the SOLID PHASE of waste and the filtrate is defined as the LIQUID PHASE.

Note: The SOLID PHASE may appear liquid in some samples. Do Not replace original filter. Use only one filter.

- 10.1.1.1.2.7 Weigh the filtrate filled container and calculate the weight of the LIQUID PHASE as follows:

$$W_{LP} = W_F - W_C$$

Where:

W_{LP} = Weight of the LIQUID PHASE

W_F = Weight of the filtrate filled container

W_C = Weight of container

- 10.1.1.1.2.8 Calculate the weight of the SOLID PHASE using the following formula:

$$W_{SP} = W_W - W_{LP}$$

Where:

W_{SP} = Weight of the SOLID PHASE

W_W = Weight of the waste sample (Section 10.1.1.1.2.3)

W_{LP} = Weight of the LIQUID PHASE (Section 10.1.1.1.2.7)

- 10.1.1.1.2.9 Calculate the percent solids using the following formula:

$$\% \text{ Solids} = \frac{W_{SP}}{W_W} \times 100$$

Where:

W_{SP} = Weight of the SOLID PHASE (Section 10.1.1.1.2.8)

W_W = Weight of the waste sample (Section 10.1.1.1.2.3)

- 10.1.1.1.2.10 If the % Solids determined above (section 10.1.1.1.2.9) is less than 0.5%, then the filtrate is the TCLP extract. Filter sample until enough filtrate is obtained for analysis and proceed to Section 10.3.1.3.6.

- 10.1.1.1.2.11 If the % Solids determined above in section is greater than or equal to 0.5%, all liquids entrained in the filter and waste must be removed to determine the % dry solids. Remove the solid phase and filter from the filtration apparatus. Heat at $100 \pm 20^\circ$ C in an oven (section 7.16) until two successive weights yield the same value within $\pm 1\%$. Record weights in the extraction logbook.

- 10.1.1.1.2.12 Calculate the percent dry solids using the following formula:

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$$\% \text{ Dry Solids} = \frac{W_{DW} - W_F}{W_W} \times 100$$

Where:

W_{DW} = Weight of dried waste + filter (Section 10.1.1.1.2.11)

W_F = Weight of the filter (Section 10.1.1.1.2.1)

W_W = Weight of waste sample (Section 10.1.1.1.2.3)

10.1.1.1.2.13 If the % dry solids determined above (section 10.1.1.1.2.12), is less than 0.5%, then the filtrate is the TCLP extract. Filter sample until enough filtrate is obtained for analysis and proceed to Section 10.3.1.3.6.

10.1.1.1.2.14 If the % dry solids determined above is greater than or equal to 0.5%, the sample is multi-phasic. Determine the weight of the dry waste by subtracting the weight of the filter from the weight of the dry waste and filter as determined follows and record in extraction logbook:

Weight of dry solids = $W_{DW} - W_F$

Where:

W_{DW} = Weight of dried waste + filter (Section 10.1.1.1.2.11)

W_F = Weight of the filter (Section 10.1.1.1.2.1)

Proceed to Section 10.3.1.3. (**NOTE:** Do not dispose of dried waste + filter.)

10.1.1.2 Determine whether waste requires particle size reduction

Evaluate the solid phase of the waste sample (Section 10.1.1.1.2.6), to determine if particle size reduction is necessary. Particle size reduction is required unless the solid has a surface area greater than 3.1 cm² per gram or is smaller than 1 cm in its narrowest dimension (i.e.: will not pass through a 9.5 mm (0.375-inch) standard sieve).

If the surface area is smaller or the particle size is larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting or grinding the waste to a surface area or particle size as described above.

NOTE: Surface area criteria is meant for filamentous (e.g. paper, cloth, etc.) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, those samples should be reduced to meet the 1 cm dimension criteria.

10.1.1.3 Extraction Fluid Determination

10.1.1.3.1 Weigh out a 5 g subsample of the solid phase waste, if the sample is all solid. If the sample is multiphasic, weigh out a 5 g subsample of the solid waste from Section 10.1.1.1.2.6.

- 10.1.1.3.2 Transfer the 5 g aliquot to a 250 mL beaker (or equivalent) and add 96.5 mL of reagent water. (NOTE: Less weight and volume may be used, provided the ratio remains the same.)
- 10.1.1.3.3 Place beaker (or equivalent) in the automatic shaker (Section 7.17) for 5 minutes.
- 10.1.1.3.4 Measure using pH strip and record the pH in the extraction logbook.
- 10.1.1.3.5 If pH is less than 5.0, use extraction fluid #1, proceed to Section 10.3.
- 10.1.1.3.6 If pH is greater than 5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watch glass, heat to 50°C+/- 2°C on a hotplate and hold for 10 minutes.

Note: heating block load tests have shown heating times of up to 25 minutes prior to the sample reaching the specified temperature. The 10 minute hold begins only after the temperature is reached and monitored using a calibrated thermometer placed in a sample or one representative of the batch.

Cool solution to room temperature and record pH. If the pH is less than 5.0, use extraction fluid #1. Otherwise, use extraction fluid #2.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Extraction of SOLID PHASE

10.3.1.1 A 100 gram minimum aliquot of SOLID PHASE is extracted to produce the TCLP extract, as outlined below. In some cases, a larger sample size may be required in order to produce enough liquid for the required analysis. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analysis, more than one extraction may be performed and the extracts combined for analysis. If there is limited sample volume, the chemist must contact the project manager.

10.3.1.2 100% Solid Waste

10.3.1.2.1 If the waste is 100% solid, homogenize the sample and weigh out an aliquot (100 gram minimum), follow Sections 10.4.1.2 (particle size reduction) and then proceed to 10.3.1.3.2 (extraction fluid amount).

10.3.1.3 Liquid or Multiphasic Waste

10.3.1.3.1 Quantitatively transfer the solid material, along with the filter (from Section 10.1.1.1.2.14), into a glass extractor bottle (plastic may be used if only metals are being analyzed).

Repeat sections 10.1.1.1.2 through .6 if additional final volume is needed for the intended analysis.

10.3.1.3.2 Determine the amount of extraction fluid to add to the extractor vessel as follows:

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$$V_{EF} = \frac{20 \times \% \text{ Solid} \times W_w}{100}$$

Where:

V_{EF} = Volume of extraction fluid required.

(Density = 0.998g/L, therefore assume volume is equivalent to weight.)

$\% \text{ Solid}$ = Percent solid as determined in section 10.1.1.1.2.9
(100% if waste contains no free liquid as determined in section 10.1.1.1.1)

W_w = Weight of waste (Section 10.1.1.1.2.3)

- 10.3.1.3.3** Add the volume of the appropriate extraction fluid (determined in Section 10.1.1.3) to the extraction vessel. Glass amber 2.5 L for organics and metals, 2.5 L plastic if for metals only analysis. New (never used) 2.5 L HPDE plastic vessels for PFAS (Reference: PFAS by SPE and LC/MS/MS Isotope Dilution (ID#23528)).
- 10.3.1.3.4** Close the extractor bottle tightly. Secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours while maintaining room temperature at $23 \pm 2^\circ$ C. Record the agitation device (Tumbler) ID in the extraction logbook.
- 10.3.1.3.4.1** The room temperature ($^\circ\text{C}$) and the extraction time are recorded in the extraction logbook at both the beginning and the end of extraction.
- 10.3.1.3.4.2** To verify that the proper room temperature is maintained during extraction, a Maximum/Minimum thermometer is reset at the beginning of the extraction period. This thermometer is reviewed at the end of the extraction period and the Maximum and Minimum temperatures are noted in the extraction logbook.
- 10.3.1.3.4.3** If proper room temperature is not maintained during the extraction period, it is considered a variation from the method and must be written into a laboratory narrative and submitted with the reported data. The Department Supervisor is immediately notified to determine the proper corrective action to be taken.
- 10.3.1.3.4.4** If determination is made that a MAX/MIN thermometer reading is outside of the acceptance range, it must be confirmed by comparison to the data logger records. This is the only way to make certain the temperature reading is accurate. Any MAX/MIN temperature reading that exceeds acceptance criteria must be reviewed by the department manager, team leader, or QA.
- 10.3.1.3.5** Following extraction, vacuum filter the extract through glass fiber filters (acid pre-cleaned for metals analysis). For this final filtration, the glass fiber filter may be changed to facilitate quicker filtration but should be kept to a minimum when practical. Use of pre-filters is prohibited for TCLP.
- 10.3.1.3.6** Prepare the TCLP extract as follows:

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- 10.3.1.3.6.1 If the waste contained no initial liquid phase, the filtered extract obtained is defined as the TCLP extract. Proceed to 10.3.1.3.7.
- 10.3.1.3.6.2 If the waste contained a liquid phase, check its compatibility with the extraction fluid determined in Section 10.1.1.3. If the liquids are compatible (miscible), then the initial liquid phase (Section 10.1.1.1.2.6) and the filtered extract are combined and analyzed together.
- 10.3.1.3.6.3 If the initial liquid phase of the waste obtained in Section 10.1.1.1.2.6, is not compatible with the filtered extract, do not combine the liquids. Analyze these liquids, collectively defined as the TCLP extract, separately and mathematically combine the results, as described below. Determine the volume of the individual phases, conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$(\text{mg/L}), \text{ Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:

- V_1 = Volume of the first phase (L)
- C_1 = Analyte concentration first phase (mg/L)
- V_2 = Volume of the second phase (L)
- C_2 = Analyte concentration second phase(mg/L)

- 10.3.1.3.7 Record the pH of the TCLP extract in the extraction logbook prior to preservation of samples.
- 10.3.1.3.8 Metals analysis: transfer filtrate (or combined filtrates) to a 120 mL plastic bottle and add 2.5 mL of 1:1 HNO₃ preservative to all samples.
 - 10.3.1.3.8.1 Check the pH of the preserved samples to ensure that the sample is properly preserved at a pH of less than 2.
- 10.3.1.3.9 Non-volatile Organics: transfer filtrate (or combined filtrates) to two 1 L glass amber bottles, adhere pre-made sample ID labels and transfer to extraction department refrigerators.

10.3.2 Extraction of Lamps

- 10.3.2.1 Lamps (for both TCLP extraction and 120x extraction) are broken down into small particles (See Section 10.1.1.2) and extracted using Extraction Fluid #1. The extraction procedure from Section 10.3.1.3.2 through 10.3.1.3.8 is followed with the exception that the entire lamp weight including endcaps is used. 7 L plastic extraction vessels are utilized for larger lamps that exceed 100 g total weight. The vessel size must be recorded in the logbook for lamp samples.
 - 10.3.2.1.1 Check the sample comments for whether Mercuric Oxide (HgO) is required during the extraction. If so, make note in the logbook that the HgO packet was added to the extraction vessel prior to tumbling. Contact the project manager if HgO is requested but no packet is supplied.

- 10.3.2.2 The excess unfiltered extract is immediately decanted into the TCLP waste drum, making sure to leave behind in the extraction vessel the solid material that was extracted.
- 10.3.2.3 The remaining solid material is transferred to a plastic bag, labeled as TCLP Lamp Waste and brought to the Hazardous Waste department for proper disposal.

10.4 Continuing Calibration

Not Applicable.

10.5 Preventive Maintenance

None.

11. Data Evaluation, Calculations and Reporting

None.

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

- 12.1 All Holding time exceedences, improper preservation and Extraction Anomalies are to be reported to a Supervisor or Manager. Non Conformance Reports may need to be issues through the Qualtrax System.
- 12.2 Refer to determinative method SOPs for additional Corrective Action information.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732 These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

- 13.2.1 **Initial (IDC)**
Not Applicable.
- 13.2.2 **Continuing (DOC)**
Not Applicable.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

SOP 2121 Chemical Hygiene Plan
SOP 1732 MDL/LOD/LOQ Generation
SOP 1739 IDC/DOC Generation
SOP 1728 Waste Management and Disposal SOP
WI 17618 TCLP and SPLP Work Instruction

16. Attachments

Table 1: Maximum Holding Times

Table 1

MAXIMUM HOLDING TIMES

Sample Maximum Holding Times (Days)

	From: Field collection	From: TCLP extraction	From: Preparative extraction	To: TCLP extraction	To: Preparative extraction	To: Determinative analysis	Total Elapsed Time
Semi-volatiles	14	7	40				61
Mercury	28	N/A	28				56
Metals, except mercury	180	N/A	180				360

PCBs

By Capillary Column Gas Chromatography

Reference Methods: Method 8082A SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, 2007.

Quality Control Requirements and Performance Standards for Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography (GC) in Support of Response Action under the Massachusetts Contingency Plan (MCP), Revision No.1, July 1, 2010.

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

1. Scope and Application

Method 8082A is used to determine the concentrations of Polychlorinated Biphenyls (PCBs) as Aroclors in extracts from solid, oil and liquid matrices. This SOP details the analysis for PCBs using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).

Matrices: Extracts from solid, oil and liquid matrices.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Regulatory Parameter List: The standard compounds listed below are determined by this method.

Parameter	CAS#
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
* Aroclor 1262	37324-23-5
*Aroclor 1268	11100-14-4

*Note – not certified by NJ for the oil matrix

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must

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demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (see section 13.2).

2. Summary of Method

A measured volume or weight of sample (volumes and weights can vary but approximately 1L or 140-272 mls (LVI – Low Volume Initiative) for liquids, 1g for oil and up to 15g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

Liquid samples are extracted at neutral pH with methylene chloride using Method 3510C (separatory funnel), or other appropriate technique. See extraction SOP for details.

Solid samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet), or other appropriate technique. Solid samples may also be extracted with hexane:acetone (1:1) using Method 3546 (microwave). See extraction SOP for details.

Wipe samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet) or other appropriate technique. See extraction SOP for details.

Oil samples are diluted with hexane following the procedure outlined in the extraction SOP.

Sulfuric acid cleanup (Method 3665A), Copper cleanup (Method 3660B) and Silica Gel cleanup (Method 3630) are utilized for PCB extracts. See extraction SOP for details.

After cleanup, the extract is analyzed by injecting 1µL into a gas chromatograph equipped with narrow- or wide-bore fused silica capillary columns and electron capture (GC/ECD) detectors.

2.1 Method Modifications from Reference

Not applicable.

3. Reporting Limits

The reporting limits for this method as outlined is as follows:

- **Aqueous samples:** 0.25 ug/L / Aroclor (based on a 1L extraction or 140 ml LVI extraction)
- **Soil Samples:**
 - 33.3 ug/kg / Aroclor based on 15g extraction
 - 50.0 ug/kg / Aroclor based on 10g extraction
 - 100.0 ug/kg / Aroclor based on 5g extraction
 - 250.0 ug/kg / Aroclor based on 2g extraction
- **Oil Samples:** 5 mg/kg based on 1g extraction
- **Solid of Difficult Matrices** (i.e Caulking, Concrete, etc. are logged using the Alpha Low Level 8082 products): based on a 15g extraction
 - Aroclors 1016, 1221, 1232, 1242, 1254: 20 ug/kg
 - Aroclors 1248, 1260: 13.3 ug/kg
 - Aroclors 1262, 1268: 6.67 ug/kg

4. Interferences

4.1 Instrumental

- 4.1.1 Only high purity gases are used in the GC system to eliminate this source of possible contamination. Both the Hydrogen (carrier gas) and Nitrogen (detector make-up gas) are certified by the gas supplier.
- 4.1.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 10.6 describes the maintenance steps.
- 4.1.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Cleaning and Handling SOP/1953. Store dry glassware in a clean environment.

4.2 Parameters

- 4.2.1 All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free. All of these materials are routinely determined to be free of interferences by analysis of extraction blanks with every extraction batch performed.
- 4.2.2 Certain compounds (i.e. phthalates) can be extracted from the sample matrix and be detected by the ECD that could possibly result in false positive results or complicate the data interpretation. The use of the cleanup procedures detailed in the extraction SOP minimizes these possible interferences. Analyst experience is also crucial in making compound determinations.
- 4.2.3 Interferences co-extracted from the samples will vary considerably from waste to waste. While a general cleanup technique is referenced or provided as part of the method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

- 5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.
- 5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

- 5.3 All stock standards, working standards, and vialled sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.
- 5.4 Bottles containing flammable solvents must be stored in the flammable's cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L or two 140 ml (LVI) amber glass jars with Teflon-lined lids. Solid samples are collected in one 250 mL wide-mouth glass jar with a Teflon-lined lid. Oil samples are collected in a glass jar with Teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of $4 \pm 2^\circ \text{C}$. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of $4 \pm 2^\circ \text{C}$. Preservation is not required for oil samples.

6.3 Sample Handling

TCLP/SPLP tumbled extracts, NJ DKQP and RCP CT aqueous samples must be extracted within 7 days of sample collection, solid and oil samples within 14 days of collection (NJ DKQP allows 365 days for solids if frozen). All other samples, both aqueous and solid, have a 365-day hold time. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 **Gas Chromatograph, Agilent 6890, 7890:** An analytical system completed with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.2 **GC Columns:** Alpha utilizes dual-column analyses. The dual-column approach involves a single injection that is split between two columns that are mounted in a single gas chromatograph. Typical column pair used is listed below. Other columns may be used as long as method performance criteria can be met.

Column pair:

RTX-CLP: Cat. #11141 from Restek or equivalent; 30m, 0.32mm, 0.32 μm

RTX-CLPII Cat. #11324 from Restek or equivalent; 30m, 0.32mm, 0.25 μm

7.3 **Guard Column:** Cat. #10027 from Restek or equivalent; 5m, 0.32mm

- 7.4 **Class "A" Volumetric Flasks:** 10mL and 25mL (and other volumes), for standards preparation
- 7.5 **Microsyringes:** 10 μ L – 1000 μ L
- 7.6 **Gooseneck splitless injecton liner,** Cat #20799-214.5 from Restek or equivalent
- 7.7 **Universal "Y" Press-tight tee split:** Cat. #20406 from Restek or equivalent /
Siltek MXT Connector: Cat. #21388 from Restek or equivalent

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at $4 \pm 2^\circ$ C in Teflon(R)-sealed containers in the dark. When a Lot of standards is prepared, aliquots of that Lot are stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

- 8.1 **n-Hexane:** Pesticide quality or equivalent.
- 8.2 **Acetone:** Pesticide quality or equivalent.
- 8.3 **Organic-free Reagent Water:** All references to water in this method refer to organic-free reagent water from Alpha's RO water treatment system.
- 8.4 **Stock Standard Solutions:** All stock standard solutions are purchased from commercial vendors as ampullated certified solutions. When an ampullated stock solution is opened, it is transferred to a labeled amber screw-cap vial. The expiration date of the stock solution is either the vendor specified expiration date, or 1 year from the date the ampule was opened, whichever is sooner.
- 8.5 **Calibration Standards:** Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the calibration standard. Calibrations are performed at the 6 concentration levels listed in Table 1. The list of ampullated calibration standards are obtain from **Agilent**:
 - Aroclor 1016, Cat. #PP-282, at 100ug/mL
 - Aroclor 1260, Cat. #PP-361, at 100ug/mL
 - Aroclor 1262, Cat. #PP-371, at 100ug/mL
 - Aroclor 1268, Cat. #PP-382, at 100ug/mL
 - Aroclor 1221, Cat. #PP-292, at 100ug/mL
 - Aroclor 1232, Cat. #PP-302, at 100ug/mL
 - Aroclor 1242, Cat. #PP-312, at 100ug/mL

- Aroclor 1248, Cat. #PP-342, at 100ug/mL
- Aroclor 1254, Cat. #PP-351, at 100ug/mL

8.5.1 Preparation of Initial Calibration Standards:

- **1660 L6:** (mix of Aroclors 1016 and 1260) is prepared by diluting
 - 2.5uL 1016 Standard (Agilent, Cat. #PP-282)
 - 2.5uL 1260 Standard (Agilent, Cat. #PP-361)
 - 1.6mL of PCB ICAL Surrogate Stock to 25ml of Hexane to achieve concentration of
 - 1016 and 1260 at 10ug/mL
 - TCMX at 0.64ug/mL and DCB at 1.28ug/mL.
- **For LVI,** this solution is diluted 10X more, achieving a concentration of
 - 1016 and 1260 at 1ug/mL
 - TCMX at 0.064ug/mL and DCB at 0.128ug/mL
- **2154 L6:** (mix of Aroclors 1221 and 1254) is prepared by diluting
 - 2.5uL 1221 Standard (Agilent, Cat. #PP-292)
 - 2.5uL 1254 Standard (Agilent, Cat. #PP-351)
 - to 25ml of Hexane to achieve concentration of
 - 1221 and 1254 at 10ug/mL
- **For LVI,** this solution is diluted 10X more, achieving a concentration of
 - 1221 and 1254 at 1ug/mL
- **4268 L6** (mix of Aroclors 1242 and 1268), **3262 L6** (mix of Aroclors 1232 and 1262) and **1248 L6;** all are prepared from their respective Stock Standards according to the formula used for L6 of 2154.
- Levels 1-5 are prepared by serial dilutions of L6s and formula with volumes needed can be found in Table 2.

8.6 Second Source Standards: (ICV) Initial Calibration Verification standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Initial Calibration Verification standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the standard. The list of ampullated standards are obtain from **Accustandard:**

- Aroclor 1016, Cat. #C-216S-H-10X, at 1000ug/ml
- Aroclor 1260, Cat. #C-260S-H-10X, at 1000ug/ml
- Aroclor 1262, Cat. #C-262S-H-10X, at 1000ug/ml
- Aroclor 1268, Cat. #C-268S-H-10X, at 1000ug/ml
- Aroclor 1221, Cat. #C-221S-H-10X, at 1000ug/ml
- Aroclor 1232, Cat. #C-232S-H-10X, at 1000ug/ml
- Aroclor 1242, Cat. #C-242S-H-10X, at 1000ug/ml
- Aroclor 1248, Cat. #C-248S-H-10X, at 1000ug/ml
- Aroclor 1254, Cat. #C-254S-H-10X, at 1000ug/ml

8.6.1 Preparation of ICV Standards:

- **1660 ICV:** is prepared by diluting
 - 50uL 1016 Standard (AccuStandard, Cat. #C-216S-H-10X)
 - 50uL 1260 Standard (AccuStandard, Cat. #C-260S-H-10X)
 - 320uL of PCB CCAL Surrogate Stockto 20ml of Hexane to achieve concentration of
 - 1016 and 1260 at 2.5ug/mL
 - TCMX at 0.16ug/mL and DCB at 0.32ug/mL
- **For LVI,** this solution is diluted 10X more, achieving a concentration of
 - 1016 and 1260 at 0.25ug/mL
 - TCMX at 0.016ug/mL and DCB at 0.032ug/mL
- **2154, 4268, 3262 and 1248 ICVs** are prepared from their respective Stock Standards according to the formula used for 1660 ICV.

8.7 Internal Standard Solution: 1-Bromo-2-nitrobenzene (Ultra, Cat. #PPS-351) is used as the internal standard and is added to all single-component calibration standards and sample extracts to achieve a concentration of 0.25µg/mL. For LVI, this solution is diluted 10X more, achieving a concentration of 0.025µg/mL.

8.8 Surrogate Standards: Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB) are used as surrogates for Aroclor analysis. They are added to the calibration standards at the concentrations listed in Table 1, Continuing Calibration Standards and are spiked into all samples and QC samples prior to extraction.

- **ICAL Surrogates Stock:** is prepared by diluting of 500uL of Pesticides Surrogates Standard Spiking Solution (Ultra, Cat. #ISM-320-1) and 500uL of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20mL of Hexane to achieve concentration of TCMX at 5ug/mL and DCB at 10ug/mL. For LVI, this solution is diluted 10X more, achieving a concentration of 0.5ug/mL for TCMX and 0.1ug/mL for DCB.
- **CCAL Surrogates Stock:** is prepared by diluting of 1mL of TCMX&DCB (Accustandard, Cat. #CLP-032-R) and 1mL of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20mL of Hexane to achieve concentration of TCMX at 10ug/mL and DCB at 20ug/mL. For LVI, this solution is diluted 10X more, achieving a concentration of 1 ug/ml for TCMX and 2 ug/mL for DCB.
- **Extraction Surrogates Stock:** is prepared by diluting of 10mL of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 1000mL of Acetone to achieve concentration of TCMX and DCB at 2ug/mL. For LVI, this solution is diluted 10X more, achieving a concentration of 0.2 ug/mL for both TCMX and DCB.
- **PCB Caulking Surrogates Stock:** is prepared by diluting of 10mL of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 100mL of Acetone to achieve concentration of TCMX and DCB at 20ug/mL.
- **PCB Oil Surrogate Stock:** is prepared by diluting of 5mL of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 500mL of Hexane to achieve concentration of TCMX and DCB at 2.0ug/mL.

8.9 LCS/MS Spiking Solutions:

- **PCB Matrix Spike:** The LCS/MS spiking solution is prepared by diluting of 6.25mL of Arochlor 1016/1260 (Restek, Cat. #32039) to 500mL of Acetone to achieve

concentration of Arochlor 1016/1260 at 12.5ug/ml. For LVI, 1.25mL of the stock solution is diluted to 500mL of Acetone to achieve a concentration of Arochlor 1016/1260 at 2.5 ug/mL.

- **PCB Caulking Matrix Spike:** The LCS/MS spiking solution is prepared by diluting of 6.25mL of Arochlor 1016/1260 (Restek, Cat. #32039) to 50mL of Acetone to achieve concentration of Arochlor 1016/1260 at 125ug/mL.
- **PCB Oil Matrix Spike:** The LCS/MS spiking solution is prepared by diluting of 2.5mL of Arochlor 1016/1260 (Restek, Cat. #32039) to 200mL of Hexane to achieve concentration of Arochlor 1016/1260 at 12.5ug/mL.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A Method Blank is an aliquot of a clean reference matrix (reagent water for water samples, Ottawa sand for soil/sediment samples, or PCB free transformer oil for oil samples) that is carried through the entire analytical procedure. Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs. The extraction blank must not contain any of the reportable analytes above the reporting limit. If any reportable analytes are detected in the blank, the entire extraction batch is suspect and re-extraction of all associated samples is required, unless the associated samples are non-detect or concentration of the analyte in the samples is 10 times greater than the concentration of this analyte in the blank. The surrogate recoveries must also be within the acceptance criteria listed in Table 3. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary. Actions associated with Method Blank failures are listed in Table 5.

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD) pair is extracted with each analytical batch. The LCS/LCSD consist of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. For Arochlor analysis, the LCS/LCSD are spiked with a mixture of Arochlor 1016 and 1260 (1660) at the levels listed in Table 4. The recovery acceptance criteria are listed in Table 3. If any recovery criteria are not met, the extract may be re-analyzed. If the criteria are still not met, the **entire batch is re-extracted**, unless the recoveries are high, and the associated samples are non-detect. If this is not possible, due to insufficient sample or holding time exceedances, the analyst must narrate the failure in the LIMS for inclusion in the client report. Actions associated with LCS/LCSD failures are listed in Table 5.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike/ Matrix Spike Duplicate (MS/MSD)

Matrix Spike and Matrix Spike Duplicate are field samples spiked with known target analyte(s) at the levels listed in Table 4. Upon client request, a MS/MSD pair is extracted and analyzed with each batch of 20 or less samples. MS samples serve as a measure of extraction accuracy, by allowing the comparison of the found amount(s) of target analyte(s) with the spiked amount(s). An MS/MSD set also allows for the calculation of the extraction precision, by comparing the results of the two samples. The recovery acceptance criteria are listed in Table 3. If the recovery criteria are not met, but are met in the LCS, the failure may be attributed to sample matrix effects. Actions associated with MS/MSD failures are listed in Table 5.

For samples with a state of origin of New Jersey, a MS must be extracted for every twenty samples within a 24hr period.

9.6 Laboratory Duplicate (DUP)

Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of a sample. Upon client request, a DUP is extracted and analyzed with batch of 20 or less samples. Duplicates serve as a measure of the extraction precision, by comparing the results of the sample and duplicate. The relative percent difference (RPD) acceptance criteria are listed in Table 3. If the RPD criteria are not met, the failure may be attributed to matrix effect. Actions associated with DUP failures are listed in Table 5.

For samples with a state of origin of New Jersey, a DUP must be extracted for every twenty samples within a 24hr period.

9.7 Surrogates

All extracted samples and associated QC are spiked with surrogates at the levels listed in Table 4. The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits listed in Table 3. If the surrogate limits are not met, the extract may be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples must be re-extracted to confirm that the failure was due to sample matrix, unless the surrogate recovery is high, and the associated sample is non-detect. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

Typical Initial calibration (each level to identified with the standard lot number)

- 1.Prime
- 2.Blank
- 3.Standard Level 1
- 4.Standard Level 2

5. Standard Level 3
6. Standard Level 4
7. Standard Level 5
8. Standard Level 6
9. Initial Calibration Verification Standard (ICV)

Repeat steps 3 – 9 as needed for each Aroclor necessary for calibration.

NOTE: If multiple calibration mixtures are analyzed, it is acceptable to analyze appropriate ICVs after all calibration standards have been injected.

Typical Daily Sequence

1. 1660 Continuing Calibration Standard (**identified with the standard lot number**)
2. Extraction Blank
3. Laboratory Control Sample
4. Matrix Spike / Matrix Spike Duplicate (if requested by Client)
5. Duplicate (if included with batch QC)
6. Samples up to 16
7. Repeat 1 – 6 as needed.

10. Procedure

10.1 Equipment Set-up

10.1.1 GC Conditions:

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2. The columns are connected to an injection tee or dual injection GC, and separate electron capture detectors. Alpha typical GC conditions are listed below but may be altered as long as method performance criteria are met.

OVEN TEMP PROGRAM	
Temperature 1	150 °C
Time 1	0 min
Ramp 1	45°C/minute
Temperature 2	230°C
Time 2	0 min
Ramp 2	30°C/minute
Temperature 3	330°C
Time 3	1 min

INSTRUMENT CONDITIONS	
Injector temperature	250°C
Injector mode	Split
Split Flow	60.1 mL/min
Total Flow	78.2 mL/min
Detector temperature	350°C
Carrier gas	Hydrogen
Carrier flow	20mL/min
Carrier mode	Constant flow
Makeup gas	Nitrogen
Total detector flow	60mL/min
Injection Volume	1µL

10.2 Initial Calibration

- 10.2.1** Prepare calibration standards using the standards listed in Section 8.5 to achieve the concentrations from Table 1. Alternatively, a standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 (1660) at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing multi-point initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. Single standards of each of the other seven Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards have been used to demonstrate the linearity of the detector, these single standards of the remaining seven Aroclors also may be used to determine the calibration factor for each Aroclor when a linear calibration model through the origin is chosen. Prepare a standard for each of the other Aroclors. The concentrations should generally correspond to the mid-point of the linear range of the detector, but lower concentrations may be employed.
- 10.2.2** Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.1. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Agilent Chemstation data acquisition software. Record the calibration standard, unique lot number (PP#) and analyst's initials in the analytical sequence list.
- 10.2.3** A 1 μ L injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.
- 10.2.4** Column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a PCB standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.
- Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes.
- Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.
- 10.2.5** **Calibration Factor:** Internal Standard Calibration techniques are employed in this method.

10.2.5.1 Internal Standard Procedure. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formula listed in Alpha's Quality Manual.

Alternatively, standards of the other seven Aroclors are necessary for pattern recognition. When employing the traditional model of a linear calibration through the origin, these standards are also used to

determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1660 mixture has been used to describe the detector response.

The standards for these seven Aroclors should be analyzed before the analysis of any samples with hits above the RL. For example, an Aroclor 1254 standard should be analyzed before a sample with a hit of Aroclor 1254.

10.2.6 Initial Calibration Criteria

- If the **RSD for an analyte is < 20%**, then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results.
- If the **RSD for any analyte is > 20%**, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done by the use of **linear regression** or **quadratic regression** (minimum of six ICAL points are needed and regression must be weighted inversely proportional to concentration) as long as the correlation coefficient is **>0.990**. If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated, and a new calibration curve must be analyzed. If quadratic regression is used for calibration, this must be noted in the laboratory narrative.
- **MCP (Massachusetts Contingency Plan) requirement:** minimum of five unique peaks must be evaluated for Aroclors 1016 and 1260.
- **MCP requirement:** Minimum of five standards (or six if non-linear regression used) must be used.
- **Percent Relative Error (%RE):** For linear and quadratic fit compounds, the %RE must be calculated for the standard at or near the midpoint and at the lowest level of the initial calibration. The %RE between the calculated and expected concentration of an analyte must be **≤ 30%**, otherwise recalibration is necessary.

10.2.7 Initial Calibration Verification

An initial calibration verification standard must be run immediately after each initial calibration, near the midpoint of the curve. The standard must be prepared using a second source that is different than the source used for the initial calibration. (Standards listed in Section 8.6). The **%D** has to be within **20% (15% for CT RCP)** when compared to the mean response factor from the initial calibration.

10.2.8 Retention Time Window

- 10.2.8.1** The retention time window used for the identification of target analytes is ± 0.07 minutes. These criteria have been adopted from the EPA CLP Statement of Work (OLM04.2). It has been found that these limits work well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results.

- 10.2.8.2 The windows listed above are used as guidance; however, the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically.

10.3 Sample Processing

The determination of PCB Aroclors is accomplished by comparing the sample chromatogram to that of the most similar Aroclor standard. The use of PCB overlays is extremely helpful, either by using hardcopies of chromatograms or by utilizing the Enviroquant software. A choice must be made as to which Aroclor is most similar and whether that standard is truly representative of the PCB in the sample. Both retention time and pattern are important when determining PCBs in a sample.

Samples that contained weathered PCB present special analytical challenges. Weathering could alter the Aroclor pattern to the extent that different peaks have to be selected for quantitation. Samples that contained more than one Aroclor present similar problems. For these samples, the Analyst may have to consider selecting the earlier eluting peaks for the lower boiling Aroclor and selecting the later eluting peaks for the higher boiling Aroclors to minimize overlapping peaks.

Minimum of 3 peaks must be chosen for each Aroclor. In these instances, the Analyst may need request the assistance of someone with more expertise in determining the presence of PCB Aroclor.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

- The laboratory must report the **HIGHER** of the two results unless obvious interference is present on of the columns.
- The **Relative Percent Difference (RPD)** should be $\leq 40\%$. If the RPD exceeds 40, this will be denoted with a P flag. If the RPD exceedance is due to interference, the lower of the dual column values can be reported with I and P flags.

10.4 Continuing Calibration

- 10.4.1 Verify calibration each **12-hours** shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than **once every twenty injections**.

A bracketing CCV is not required with the use of internal standard calibration (Method 8082A 11.6.8) with the exception of samples ran under CT RCP method.

For Aroclor analysis, the calibration verification standard should be a mixture of Aroclor 1016 and 1260 (1660). The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition

(Method 8082A 11.6.2). However, if the one-point calibration is used for the seven other Aroclor, a calibration standard must be analyzed before the sample for any hits.

- 10.4.2** The **Response Factor** (for internal standard compounds) for each analyte to be quantitated must not exceed a **$\pm 20\%$ difference** when compared to the initial calibration curve (**$\pm 15\%$ for CT RCP**). The Enviroquant data processing software automatically calculates the %D for all analytes according to the formulae in Alpha's Quality Manual.
- 10.4.3** A **Retention Time shift >30 seconds** for the internal standard necessitates reanalysis of all affected samples.

10.5 Internal Standard

The use of internal standard calibration does not require that all sample results be bracketed with CCV standard. However, when internal standard calibration is used, the retention times of internal standards and the area response of internal standards should be checked for each analysis (10.4.3).

- 10.5.1** **IS in CCAL** – The measured area of the internal standard must be no more than **$\pm 50\%$** different from the average area calculated during initial calibration (**-50 to 150%**).
- 10.5.2** **IS in samples** - The measured area of the internal standard must be no more than **-50% to +100%** different from the area calculated from opening CCV (**-50 to 200%**)

Retention time shifts of more than 30 sec from the retention time of the most recent calibration standard are cause for concern and must be investigated.

10.6 Preventive Maintenance

- 10.6.1** **Preventive Maintenance:** Routine preventive maintenance is performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals.
- 10.6.2** **Other Maintenance:** ECD detectors may become contaminated, requiring bake out at elevated temperatures, (no greater than 375C) or repair by the manufacturer.

11. Data Evaluation, Calculations and Reporting

11.1 Quantitation of Aroclors

Per Method 8082A, quantitation is based on the use of a minimum of 3 of the major peaks present in the analyte, although the use of 5 of the major peaks is recommended. Each of these peaks is individually calibrated with a **minimum of five calibration points** based on average response factors. The %RSD must meet the criteria of $\leq 20\%$ for the ICAL. The five major peaks are calculated as described below. After individual calculation meets criteria, the average of the peaks selected for quantitation is used to determine the final concentration.

11.1.1 Aqueous samples

$$\text{Concentration } (\mu\text{g/L}) = \frac{C \times DF \times V_f \times 1000}{V_o}$$

where:

C = Extract concentration ($\mu\text{g/mL}$), **NOTE:** ng on column = ng/injection volume = ng/ μL = $\mu\text{g/mL}$
DF = Dilution factor
Vf = Final extract volume (mL)
Vo = Sample volume (mL)

11.1.2 Soil/sediment samples

$$\text{Concentration } (\mu\text{g/Kg, dry weight}) = \frac{C \times DF \times V_f \times 1000}{W \text{ (gm)}} \div \%S$$

where:

C = Extract concentration ($\mu\text{g/mL}$), **NOTE:** ng on column = ng/injection volume = ng/ μL = $\mu\text{g/mL}$
DF = Dilution factor
Vf = Final extract volume (mL)
W = Weight of the sample extracted (10g for high, 30g for low)
%S = Percent solids, as a decimal value

11.1.3 Reporting Results

11.1.3.1 After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

11.1.3.2 Reporting Results for PCBs in Caulking Samples

If in the screen sample Aroclor concentration as calculated above is $\geq 20000\text{ppm}$, the Client is contacted by a Customer Service Representative and these results are sent to the LIMS and reported to the Client.

If the sample concentration as calculated above for any Aroclor is $< 20000\text{ppm}$, the sample is sent for re-extraction by Method 3540C (Alpha SOP/1954).

11.1.3.3 Summation Rules

“TOTAL” concentrations are calculated for **ALL samples and Quality Control Samples** (i.e. LCS, MS, DUP, BLK).

TOTAL = sum of “reportable” Aroclors

Reportable- all Aroclors reported for associated project.

For dual-column analysis, Total is reported as part of column “A” data, unless all individuals are reported from “B” column. “Total” is calculated based on the associated “Report List”. See Work Instruction #14335 for details.

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

Holding time exceedance and/or improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written into the LIMS by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan
SOP/1732 MDL/LOQ Generation
SOP/1739 IDC/DOC Generation
SOP/1728 Waste Management and Disposal SOP

16. Attachments

Table 1: STANDARD SOLUTIONS
Table 2: PCB ICAL PREPARATION
Table 3: QC ACCEPTANCE CRITERIA
Table 4: SPIKING LEVEL
Table 5: QC FAILURE ACTION PLAN

TABLE 1
STANDARD SOLUTIONS – Suggested Concentrations

STANDARD SOLUTIONS	Stock solution (ug/mL)	Level 1 (ug/mL)	Level 2 (ug/mL)	Level 3 (ug/mL)	Level 4 (ug/mL)	Level 5 (ug/mL)	Level 6 (ug/mL)
PCB							
Aroclor 1016/1260	100	0.1	0.5	1	2.5	5	10
Aroclors 1221, 1232, 1242, 1254, 1262, 1268	100	0.1	0.5	1	2.5	5	10
LVI		0.01	0.05	0.1	0.25	0.5	1
Internal Standard							
1-Bromo-2-Nitrobenzene	5000	0.25	0.25	0.25	0.25	0.25	0.25
LVI		0.025	0.025	0.025	0.025	0.025	0.025
Surrogates:							
Tetrachloro-m-xylene	2.0	0.0064	0.032	0.064	0.16	0.32	0.64
Decachlorobiphenyl	2.0	0.0126	0.064	0.128	0.32	0.64	1.28
LVI – 10X less							

LVI is spiked 10X lower

TABLE 2
PCB ICAL PREPARATION:

Aroclors: 1660, 2154, 4268, 3262, 1248				
Level	Conc [ug/L]	Spike	Spike [uL]	Hexane [mL]
6	10000	L6	1000	0
5	5000	L6	500	500
4	2500	L6	250	750
3	1000	L6	100	900
2	500	L6	50	950
1	100	L6	10	990

TABLE 3
QC ACCEPTANCE CRITERIA

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

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

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TABLE 4
SPIKING LEVEL

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

TABLE 5
QC FAILURE ACTION PLAN

Blank Failures	Failure Situation	Action(s)
Surrogate Failure	1 of 4 high or low	Report as is
	2 or more high	Report as is
	2 or more low, samples are non-detect with passing surrogates	Report as is
	2 or more low, samples have hits and/or failing surrogates	Reviel/rerun Re-extract if still failing
IS Failure	IS not present (less than 10% recovery)	Add IS Rerun
	IS out high or low	Reviel/rerun Re-extract if still failing
Contamination	Below RL; sample(s) non-detect, below RL, or concentration 10 times or more above the blank	Report as is
	Below RL, sample(s) concentration above RL	See team leader/manager
	Above RL, sample(s) concentration 10 times or more above the blank	Report as is
	Above RL with non-detect sample(s)	Report as is
	Above RL, sample(s) concentration less than 10 times above the blank and volume available	Reviel/rerun Re-extract if contamination still present
	Above RL, sample(s) concentration less than 10 times above the blank and no volume available	Reviel/rerun B-flag affected data

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LCS/LCSD Failures	Failure Situation	Action(s)
Surrogate Failure	1 or more high or low	Report as is
IS Failure	IS not present (less than 10% recovery)	Add IS Rerun
	IS out high or low	Reval/rerun Re-extract if still failing
Spike Failure	Spike compounds out high and sample(s) are non-detect with passing surrogates	Report as is
	Spike compounds out high and sample(s) have hits	Reval/rerun Re-extract if still high
	Spike compounds out low	Reval/rerun Re-extract if still low
	RPD failure	Report as is

MS/MSD/DUP Failures	Failure Situation	Action(s)
Surrogate Failure	(MS/MSD) 1 or more high or low	Report as is
	(MS/MSD) 2 or more high or low	Report as is
	(DUP) 1 or more high, parent sample non-detect	Report as is
	(DUP) 1 or more low, parent sample matches	Report as is
	(DUP) 1 or more low, parent sample doesn't match	Reval/rerun Re-extract if still failing
IS Failure	IS not present (less than 10% recovery)	Add IS Rerun
	IS out high or low	Reval/rerun Re-extract if still failing
Spike Failure (MS/MSD only)	Compounds out high or low due to sample matrix or hits	Report as is
	Compounds out high or low due to extraction error	Reval/rerun Re-extract if still failing
	RDP failure	Report as is

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Ignitability of Solids

Reference Method: **Method 1030**, Rev. 0, December 1996 (Modified)

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

1. Scope and Application

Matrices: Solids including pastes, powders, granular material, soils, clay and solids that can be cut into strips.

Definitions: See Alpha Laboratories Quality Manual Appendix A

This method may be used to meet certain regulatory applications; with respect to the characteristic of ignitability in CFR 261.21, this method may be used, but is not required, to determine whether a solid waste "when ignited, burns so vigorously and persistently that it creates a hazard." If it is impractical to perform the test because the physical form of the sample, generator knowledge should be used to determine the ignitability hazard posed by the material.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by completing the record of training.

2. Summary of Method

In a preliminary test, the test material is formed into an unbroken strip or powder train 250mm in length. An ignition source is applied to one end of the test material to determine whether combustion will propagate along 200mm of the strip within a specified time period. Materials that propagate burning along a 200mm strip within the specified time period are then subjected to a burning rate test. Materials that do not ignite or propagate combustion as described above do not require further testing.

In the burning rate test, the burning time is measured over a distance of 100mm and the rate of burning is determined. Lab doesn't perform Burning rate test and will proceed to Flashpoint analysis by Flash test EPA 1010. SOP 2227

2.1 Method Modifications from Reference

A high temperature gas torch is utilized as the flame source. Lab is using modified method. Lab doesn't proceed to the burning rate test if sample ignites during preliminary test.

3. Reporting Limits

There is no reporting limit for this analysis. Results are reported as either Negative or Positive. Refer to Section 11 for data reporting.

4. Interferences

Particle size of test material can affect not only the burning rate, but also the ignition of the material. Therefore particle size of the test material should be the same for each test run. Report the particle size of the test material in a simple descriptive format (e.g. fine powder, sand, coarse granular).

Temperature of some test material such as sulfur powder affects the burning rate. For reproducible results, all tests must be performed at approximately the same initial temperature (ambient room or laboratory temperature).

All tests must be carried out inside a fume hood with the test apparatus situated perpendicular (90°) to the direction of the airflow. Airflow parallel (0°) to the test apparatus results in non-reproducible burning rates.

The rate of airflow through the fume hood affects the burning rate. Too high an airflow will distort the flame and retard its horizontal propagation. The optimum airflow is within the range of 0.7 – 1 meter per second.

Materials that are moisture sensitive (i.e. readily absorb moisture from the air) should be tested as quickly as possible after removal from the sample container. All materials should be tested as received by the laboratory.

5. Health and Safety

Each sample should be treated as a potential health hazard. From this viewpoint, exposure must be reduced to the lowest possible level by whatever means available. References to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. These practices include, but are not limited to, the use of: laboratory coat, safety glasses, and latex or nitrile gloves, and/or heat resistant gloves. Use caution when operating the high temperature torch. Also use caution in handling the ceramic plate after heating the sample, as it remains hot.

Prior to starting the preliminary test, all sample materials must be tested to determine if that material is explosive or extremely flammable. Use a very small portion of material (1 gram or less). **If the sample displays explosivity or extreme flammability, do not continue this test.**

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample containers should be completely filled and tightly sealed to preserve sample integrity.

6.2 Sample Preservation

No sample preservation is required.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

All samples are tested as received unless requested otherwise. Sample aliquots should be tested as soon as possible after removal from the sample container (i.e. samples must not be allowed to dry or absorb moisture for excessive periods or to lose volatiles). Allow samples to equilibrate to the ambient laboratory temperature in the sample container.

7. Equipment and Supplies

7.1 Ceramic Tile: Low heat conducting, non-combustible and impervious with marks indicating 80mm, 180mm, 200mm and 250mm along the test path. Any material capable to withstand high temperatures that have marks indicating 80mm, 180mm, 200mm and 250mm can be used.

7.2 Powder Train Mold: For molding powdered and granular materials for the burn rate test. The material of construction can be aluminum, brass, stainless steel or plastic. The mold is 250mm in length and has a triangular cross-section, with a width of 20mm, and a depth of 10mm as measured from the bottom of the triangular opening to where the sides meet. On both sides of the mold, in the longitudinal direction, two sheets are mounted as lateral limitations that extend 2mm beyond the upper edge of the triangular cross-section.

7.3 High temperature gas torch: A 6 to 7cm flame, with a minimum diameter of 5mm capable of attaining a temperature of at least 1000 °C. MAPP gas is its fuel source.

7.4 Stop watch.

7.5 Thermocouple: To measure the temperature of the gas flame

7.6 Thermometer: To measure initial temperature of material (i.e. room temperature).

7.7 Anemometer: To measure airflow in the fume hood.

7.8 Fume Hood.

8. Reagents and Standards

No special reagents are required to conduct this test.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

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9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Not applicable

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Perform preliminary screening test:
 - Prepare test strip or powder train
 - Light flame and measure temperature
 - If it does not burn within specified time, sample is considered a negative, nonflammable solid. Test is complete.
 - If it does burn within specified time, sample is considered Positive for the preliminary screening test. Stop analysis.
- Contact supervisor or team leader; in order to switch method from Ignitability to Flash point.
- Refer to SOP 2227 for Flash point analysis

10. Procedure

10.1 Equipment Set-up

Safety Test: Prior to starting the preliminary test, all sample materials must be tested to determine if that material is explosive or extremely flammable. Use a very small portion of material (1 gram or less). **If the sample displays explosivity or extreme flammability, do not continue this test.**

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Preliminary Screening Test

- 10.3.1.1 The preliminary ignitability test is conducted on all waste materials.
- 10.3.1.2 Place a platform (brick, stone, or other flame-resistant tile) in a fume hood about 20cm (or 8 inches) from the front of the hood in an area of laminar airflow. Position the sample perpendicular to the airflow. The airflow across the perpendicular axis of the sample should be sufficient to prevent fumes from escaping into the laboratory and should not be varied during the test. The air velocity should be approximately 0.7 meters/second. Measure and record the air velocity by using the anemometer (Section 7.7).
- 10.3.1.3 On the platform, prepare the test material in its "as received" form by forming an unbroken strip or powder train of sample 250mm long by 20mm wide by 10mm high on the platform. Position the sample perpendicular to the airflow. Use the mold (Section 7.3) to form the material as in Section 10.3.2.1 if appropriate.

- 10.3.1.4** Light the gas torch. Measure and record the temperature of the flame (tip of the flame) by a thermocouple (Section 7.6). The temperature of the flame must be at least 1000 °C.
- 10.3.1.5** Apply the tip of the flame to one end of the sample strip. Measure the propagation of combustion with the ceramic tile (Section 7.1). The test period will depend on the sample matrix as follows:
- 10.3.1.5.1 Non-Metallic Waste, Soils, Clays:** Hold the flame tip on the sample strip until the sample ignites or for a maximum of 2 minutes. If combustion occurs, begin timing with a stopwatch and note whether the combustion propagates up to the 200mm mark within the 2-minute test period.
- 10.3.1.5.2 Metal or Alloy Powders:** Hold the flame tip on the sample strip until the sample ignites or for a maximum of 5 minutes. If combustion occurs, begin timing with a stopwatch and note whether the combustion propagates up to the 200mm mark within the 20-minute test period.
- 10.3.1.6** If waste does not ignite and propagate combustion by either burning with open flame or by smoldering along the 200mm of sample strip within the 2 minute test period (or 20 minute test period for metal powders), the waste is not considered flammable and no further testing is required.
- 10.3.1.7** If the waste propagates burning of 200mm of the test strip within the 2-minute test period (20 minute test period for metals), test should be stopped and analysis should be switched to Flashpoint test (SOP 2227)

10.4 Continuing Calibration

Not applicable.

10.5 Preventive Maintenance

The ceramic plate and train mold must be cleaned after each sample.

11. Data Evaluation, Calculations and Reporting

Include in the log book the following information for each sample:

Test Material Information:

- Date of test:
- Description of material: e.g. powder or paste, metallic or non-metallic
- Particle size: e.g. fine powder, granular, sand, etc.
- If the preliminary screening test (Section 10.3.1) was negative, the result for the sample is Not Ignitable (reported as "NI") and no further data is required.

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

Holding time exceedence and improper preservation are noted on the Sample Delivery Group Form and/or Nonconformance Report form. Project manager also have to be notified.

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If sample cannot be analyzed using Ignitability method, project manager has to be notified and customer has to be contacted. If matrix of the sample is not appropriate for Ignitability analysis then LIMS product may be changed to Flashpoint product in order to provide customer with accurate data. Project manager/customer have to be notified and project manager has to approve this change.

Supervisor and project manager have to be notified if there is not a sufficient amount of sample provided for analysis.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

This study is not applicable to this method.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

These studies are not applicable to this method.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

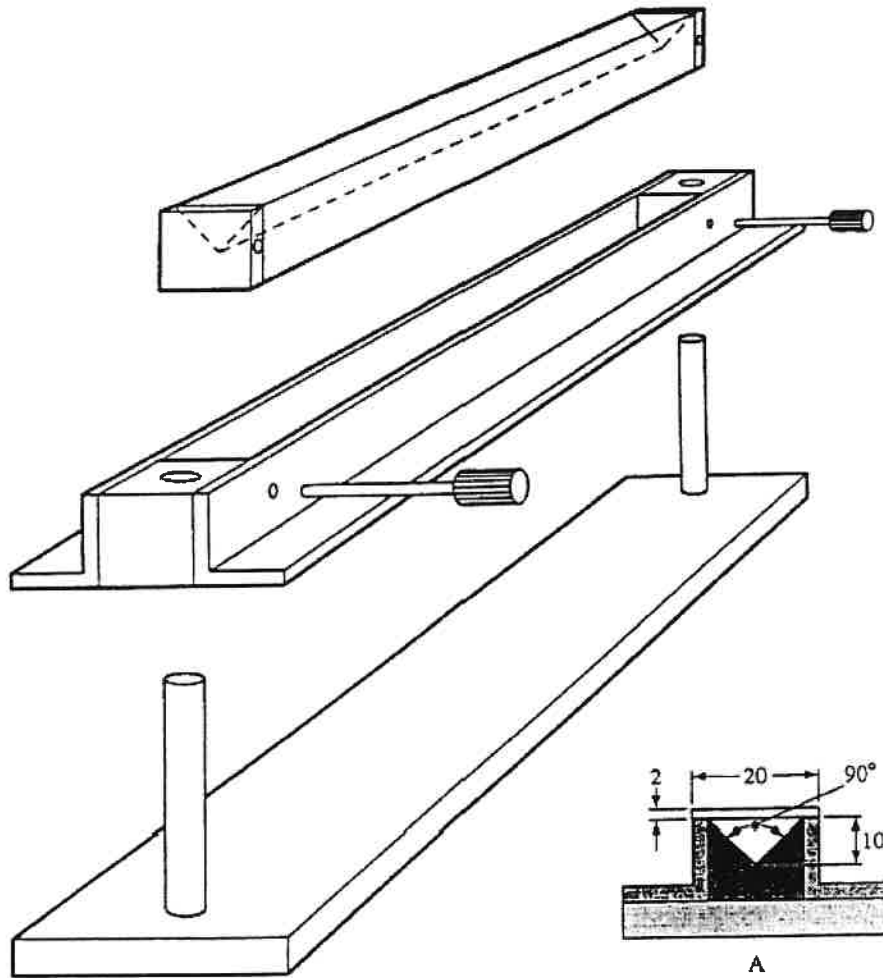
- 2121 Chemical Hygiene Plan
- 1732 MDL/LOD/LOQ Generation
- 1739 IDC/DOC Generation
- 1728 Waste Management and Disposal SOP
- 2227 Flashpoint

16. Attachments

- FIGURE 1: Powder Train Mold
- Flowchart

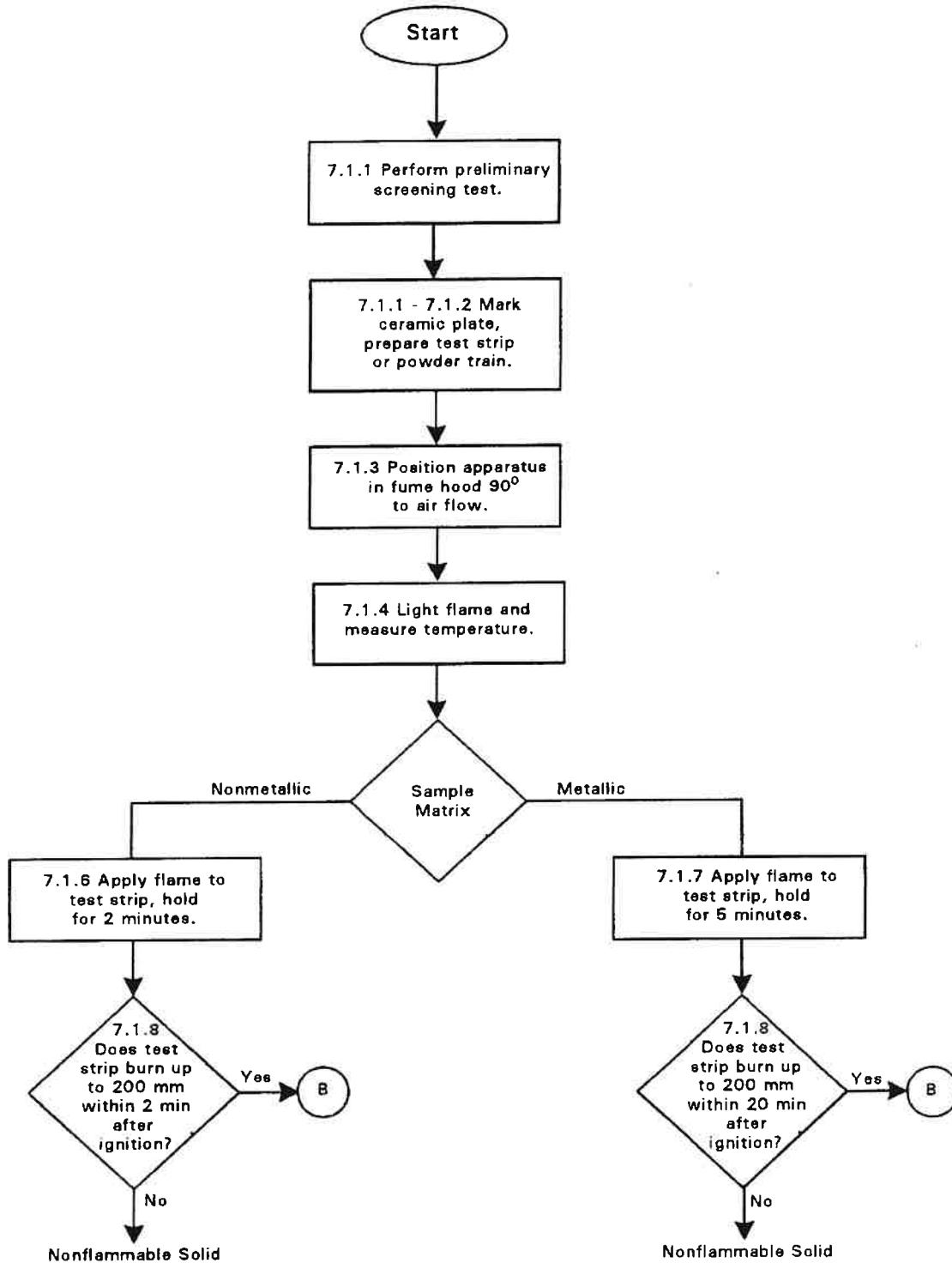
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FIGURE 1
Powder Train Mold



(A) Cross-section of 250 mm long mould

Flowchart – Ignitability of Solids Method 1030



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pH, Soil and Waste

References: Method 9045D, Soil and Waste pH, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 4, November 2004.

Method 9040C, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 3, November 2004.

NJDEP Site Remediation Program, Data of Known Quality Protocol, Version 1, April 2014

1. Scope and Application

Matrices: Soils and wastes, including solids, sludges, or non-aqueous liquids.

Definitions: See Alpha Laboratories Quality Manual Appendix A

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the pH meter and in the interpretation of pH data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured.

2.1 Method Modifications from Reference

The pH meter that is utilized will compensate for the temperature of the sample. Therefore, the temperature is not reported with the data. However, the sample is analyzed at room temperature. Only one aliquot is used for measurement.

3. Reporting Limits

None.

4. Interferences

4.1 Errors will occur when the electrode becomes coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or (3) be cleaned per the manufacturer's instructions.

4.2 Samples with a very low or very high pH may give incorrect readings on the meter.

For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode.
Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

4.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1:10) may be necessary to remove any remaining film.

4.4 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference should be controlled with instruments having temperature compensation. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. However, prior to analysis the samples are brought to room temperature (20 – 25 °C).

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in 4-ounce glass jars.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are to be analyzed as soon as possible after sampling. Samples are stored at $4 \pm 2^\circ\text{C}$. Samples that are to be run in conjunction with NJ-Hex DAK acceptance criteria must be analyzed within 24 hours of Hex sample preparation.

7. Equipment and Supplies

- 7.1 pH Meter:** “Black” Orion Research, expandable ion analyzer EA 940. Laboratory benchtop model. “White” Hanna pH temperature bench meter (HI5521-01). “White 3” Hanna pH temperature bench meter (HI2002-01) Or equivalent.

- 7.2 **pH electrode:** Accuphast electrode with automatic temperature compensation Fisher Catalog #13-620-296. Incorporates measuring and referenced functions; filled with AgCl solution. Hanna (HI 1131B) Glass body, refillable, combination electrode for "white 3".
- 7.3 **Beakers:** 50mL glass or plastic
- 7.4 **Magnetic Stirrer**
- 7.5 **Teflon-coated stirring bar**
- 7.6 **Analytical Balance:** Capable of weighing 0.1g
- 7.7 **Kimwipes**

8. Reagents and Standards

- 8.1 **Reagent Water:** All references to water in this method refer to reagent water.
- 8.2 **pH Buffers:** Commercially available pH 4 (or 4.01), pH 7, pH 10 (or 10.01). In addition, an alternate source of pH 7 Buffer is necessary. All Buffers must have been validated by comparison to NIST standards. Certificate of analysis is required. Buffers are stored at room temperature and expire upon manufacturer's specified date.

9. Quality Control

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

One LCS is analyzed with each batch of 20 samples or less. It is a pH 7 buffer of a different source than the calibration buffer. Results must be within ± 0.05 .

9.3 Initial Calibration Verification (ICV)

Refer to LCS Section 9.2.

9.4 Continuing Calibration Verification (CCV)

A CCV of pH 7 is analyzed at the end of the analytical run. Results must be within ± 0.2 units.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

One duplicate sample is analyzed per batch of 20 samples or less

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Calibration

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- Calibration Verification (LCS)
- Sample analysis
- Duplicate Analysis
- Calibration Verification (CCV)

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

In a 50mL beaker, weigh 20 grams of soil; record the weight. Add 20mL of reagent water, cover and continuously stir the suspension for 30 minutes, using a teflon stir bar on a magnetic stirrer. Let samples stand for 1 hour before measurement.

Additional dilutions are permissible if working with hygroscopic soils, salts or other problematic matrices.

NOTE: If the sample is hygroscopic and absorbs all the reagent water, begin again using 20 grams of sample and 40mL of reagent water.

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous base. The electrode may need to be cleaned (Section 4.1) if it becomes coated with an oily material.

10.2 Initial Calibration

The pH meter is calibrated on a daily basis using three pH buffers (Section 8.1). Follow manufacturer's instructions for a 3-point calibration of the pH meter. The results of the calibration must be recorded in the pH Calibration Log.

10.3 Equipment Operation and Sample Processing

10.3.1 Allow the sample suspension to stand for about 30 minutes to allow suspended material to settle out from the suspension.

10.3.2 Immerse the pH electrode just below the suspension and allow pH meter to stabilize. Note and record the sample pH in the Laboratory notebook. Report sample temperature at the time of measurement.

10.3.3 Rinse the electrode thoroughly between samples, using reagent water.

10.4 Continuing Calibration

Prior to sample analysis, the calibration is initially verified by using a pH 7 buffer from a source other than the source used for calibration. The results must be within ± 0.2 . If this criterion is not met, the meter must be re-calibrated before sample analysis can begin.

10.5 Preventative Maintenance

The pH probe is rinsed with DI and gently dried with a KimWipe between sample readings.

11. Data Evaluation, Calculations and Reporting

pH is read directly from the pH meter. No calculations are necessary.

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For pH readings of less than 1, the reported result is: pH <1.

For pH readings greater than 10, report result in 3 significant figures.

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

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the maintenance logbook.

The pH electrode is replaced as necessary.

Review of standards for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS or CCV recovery falls outside the designated acceptance range, the laboratory performance for the parameter is judged to be out of control, and the problem must be immediately identified and corrected. Re-calibration of the meter is necessary prior to sample analysis. All samples analyzed since the last acceptable QC standard must be reanalyzed following re-calibration of the meter.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

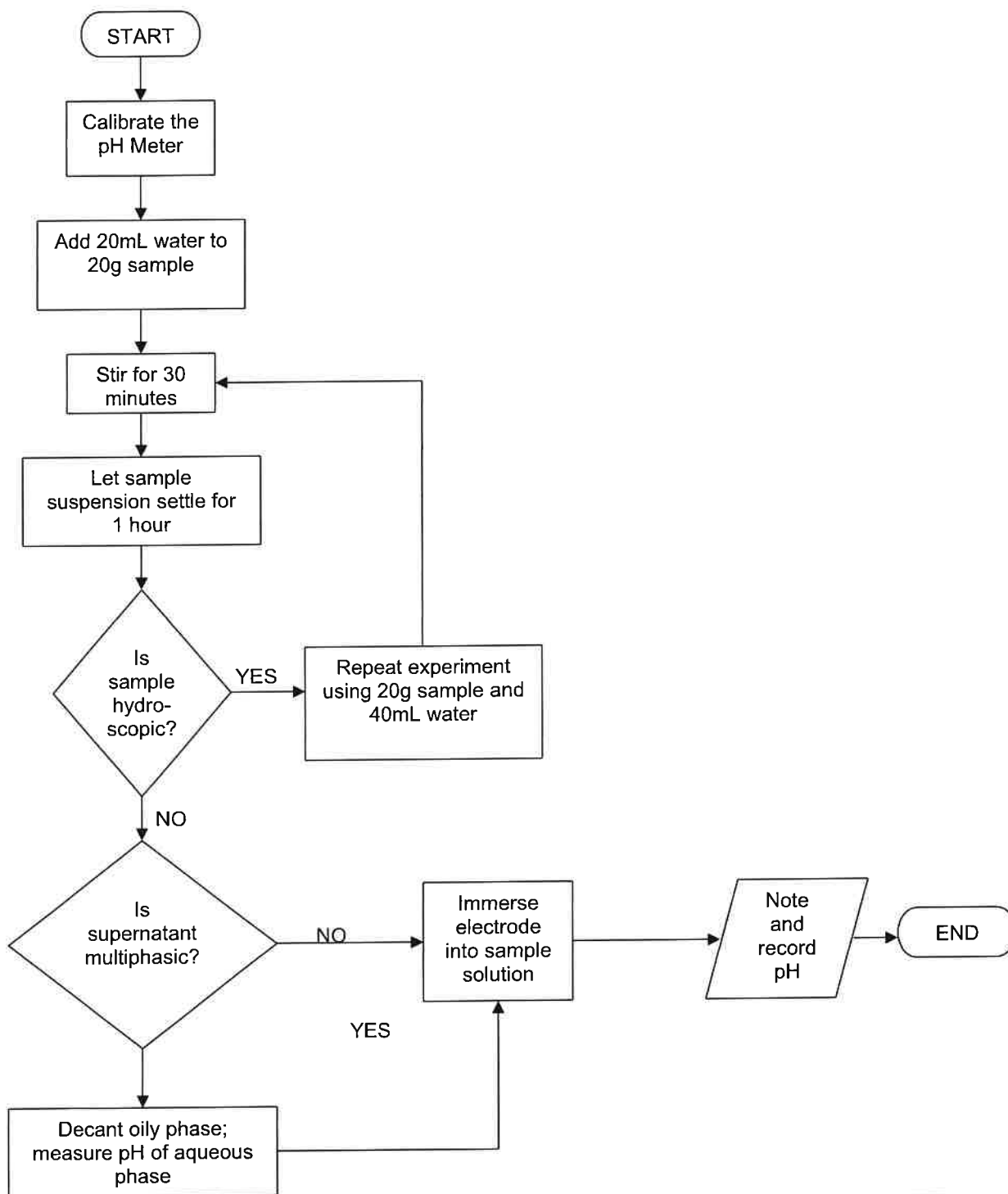
SOP/1728 Waste Management and Disposal SOP

16. Attachments

Flow Chart: Soil and Waste pH

Flow Chart:

Soil and Waste pH



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Inductively Coupled Plasma - Mass Spectrometry 6020B

Reference: Method 6020B, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision V, July 2014.

1. Scope and Application

Matrices: Groundwaters, aqueous samples, industrial waste, soils, sludges, sediments, and other solid wastes.

Definitions: Refer to Alpha Analytical Quality Manual.

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub µg/L concentrations of a large number of elements in water samples, waste extracts or digestates.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Elements for which EPA has determined the acceptability of Method 6020 in a multi-laboratory study on solid wastes are listed below and in Table 1.

If method 6020 is used to determine any analyte not listed in Table 1 below, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

Use of this method is restricted to spectroscopists who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS.

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^6Li , ^{45}Sc , ^{89}Y , ^{103}Rh , ^{115}In , ^{159}Tb , ^{165}Ho , and ^{209}Bi . The Lithium internal standard must have an enriched abundance of ^6Li , so that interference from Lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards. The internal standard used in this method is as follows: ^6Li , ^{45}Sc , ^{74}Ge , ^{103}Rh , ^{115}In , ^{159}Tb , ^{175}Lu , ^{209}Bi .

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the ICP-MS and in the interpretation of ICP-MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Table 1

Parameter	CAS	Parameter	CAS
Aluminum	7429-90-5	Magnesium	7439-95-4
Antimony	7440-36-0	Manganese	7439-96-5
Arsenic	7440-38-2	Lithium	7439-93-2
Barium	7440-39-3	Molybdenum	7439-98-7
Beryllium	7440-41-7	Nickel	7440-02-0
Cadmium	7440-43-9	Potassium	7440-09-7
Calcium	7440-70-2	Selenium	7782-49-2
Chromium	7440-47-3	Silver	7440-22-4
Cobalt	7440-48-4	Sodium	7440-23-5
Copper	7440-50-8	Thallium	7440-28-0
Iron	7439-89-6	Vanadium	7440-62-2
Lead	7439-92-1	Zinc	7440-66-6

2. Summary of Method

When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial waste, soils, sludge's, sediments, and other solid wastes for which total (acid-leachable) elements are required.

Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into the mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratio and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate a problem. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

2.1 Method Modifications from Reference

This method is performed in a 1% Nitric Acid matrix for the calibration curve and standards and a 2% Nitric Acid matrix for the rinse.

3. Reporting Limits

Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, instrumentation and operating conditions. In relatively simple matrices, detection limits will generally be below 20 µg/L. Table 2 contains the list of Reporting Limits.

4. Interferences

4.1 Isobaric Elemental Interference

Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for

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another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Since commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height, very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. Although this type of interference is uncommon, it is not easily corrected, and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, or analysis using another verified and documented isotope, or use of another method.

4.2 Isobaric Molecular and Doubly Charged Ion Interference

Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Examples include ArCl^+ ions on the ^{75}As signal and MoO^+ ions on the Cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundances from the literature, the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standing solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}\text{Ar}^{37}\text{Cl}^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}\text{Ar}^{35}\text{Cl}^+$ signal):

Corrected arsenic signal (using natural isotopes abundances for coefficient approximations) = $(m/z$ 75 signal) – (3.13) (m/z 77 signal) + (2.73) (m/z) (82 signal), (where the final term adjust for any selenium contribution at 77 m/z)

Note: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}\text{Se}^+$, (e.g. $^{81}\text{BrH}^+$ from bromine waste)

Similarly, corrected cadmium signal (using natural isotopes abundances for coefficient approximations) = $(m/z$ 114 signal) – (0.027) (m/z 118 signal) – (1.63) (m/z 108 signal), (where the last two terms adjust for any tin or MoO^+ contributions at m/z 114).

Note: Cadmium values will be biased low by this type of equation when $^{92}\text{ZrO}^+$ ions contribute at m/z 108, but use of the m/z 111 for Cd is even subject to direct ($^{94}\text{ZrOH}^+$) and indirect ($^{90}\text{ZrO}^+$) additive interferences when Zr is present

Note: As for the arsenic equation above, the coefficients in the Cd equation are for only illustrative purposes. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting precision.

The accuracy of these types of equations is based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the “parent” ion have not been found to be reliable, e.g. oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent –to-oxide ion intensities, the correction must be adjusted for the degree of outside formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferant. This type of correction has been reported for oxide-ion corrections using ThO^+/Th for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas has been shown to greatly reduce molecular interferences. These techniques can be used provided that method detection limits, accuracy, and precision requirements for analysis of the samples can be met.

4.3 Physical Interference

Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for the physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by the matrix change. When the intensity level of an internal standard is less than 30 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

4.4 Memory Interference

Memory interferences can occur when there are large concentration differences between samples or standards which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences which are observed. The rinse period between samples must be long enough to eliminate significant memory interferences.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Only Polyethylene or fluorocarbon (PFA or TFE) containers are recommended. Alpha uses polyethylene bottles. 0.5L is the recommended size.

6.2 Sample Preservation

Samples for total metals are preserved with (1:1) Nitric Acid to a pH<2.

Samples for soluble metals must be preserved with (1:1) Nitric Acid to a pH of <2 **after** filtration through a 0.45 um filter.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Samples that are to be analyzed for soluble metals, and have not been field filtered, must be filtered through a 0.45um filter as soon as possible. Samples are then preserved with 1:1 Nitric Acid to a pH<2, and then held for 18 hours. After the 18 hours the pH must be re-checked. If

after 18 hours the pH is still >2, then the sample must be re-acidified and held again for 18 hours. If the pH is still >2 after this 18 hour period, then the Inorganics Manager must be told.

Samples preserved with Nitric Acid to a pH<2, and are not being analyzed for Mercury, have a hold time of 6 months.

Samples are stored at room temperature

7. Equipment and Supplies

7.1 Thermo Q and RQ ICP-MS:

The MS features dual extraction lenses and a compound ion lens system that ensures a mass range from Li-U (masses 6-240). It also features the enhanced RAPID (Right Angle Positive Ion Deflection) lens technology (90° ion optics done right) for separation of ions and neutrals. Simultaneous analog/PC detector with real time multi-channel analyzer electronics provides >9 orders of dynamic range suitable for both steady state and transient signal analysis. The software is Qtegra which provides integrated plug-ins for autosamplers, autodiluters and one click instrument set up: with a single mouse click, the iCAP Q is taken into operate mode, tested and, where necessary intelligently autotuned and/or calibrated to meet the protocol.

7.2 CETAC ASX-510 Autosampler: Delivers sample and internal standard to the torch

7.3 Thermo X-series II ICP-MS

The Innovative Protective Ion Extraction and Infinity II ion optics, based upon a hexapole design with chicane ion deflector, provides low background specification coupled with a userinterchangeable Xt interface and Xs interface. High-performance quadrupole analyzer is pumped by a novel split flow turbo pump backed by a single rotary. Simultaneous analog/PC detector with real time multi-channel analyzer electronics provides >8 orders of dynamic range suitable for both steady state and transient signal analysis. Instrument and accessories are fully computer controlled by the PlasmaLab software. Peltier spray chamber cooling provides enhanced performance for outstanding signal /background.

7.4 Edwards E2M28 and Sogevac 40 Rotary Pumps

7.5 Neslab M-75 Chiller: Cools the torch and the MS

7.6 Eppendorf pipets: Accurate means to make trace standards

8. Reagents and Standards

8.1 Nitric Acid (HNO₃), Trace metals grade: 18M Concentrated Redistilled

8.2 1% Nitric Acid (v/v): 10mL of 18M HNO₃ diluted to 1L using Type I water

8.3 Type I De-Ionized Water

8.4 Calibration Standard: Multiple Element Standard, purchased (Table 4) and diluted. Store at room temperature. Expires upon manufacturer's specified date. Prepare as needed if confirmed with the ICV solution made fresh daily.

8.5 Internal Standard: Multiple Element Standard, purchased (Table 8). Store at room temperature. Expires upon manufacturer's specified date.

- 8.6 Tune Stock:** Multiple Element Standard, purchased (Table 9). 100.0 ug/L solution of each ${}^7\text{Li}$, ${}^9\text{Be}$, ${}^{58}\text{Ce}$, ${}^{59}\text{Co}$, ${}^{115}\text{In}$, ${}^{137}\text{Ba}$ and ${}^{208}\text{Pb}$, and ${}^{238}\text{U}$.
- 8.7 ICV:** Multiple Element Standard, second source from calibration standard (Section 8.4) purchased and diluted (Table 10). Concentrations are near but not at the mid-point of the calibration Prepare fresh daily.
- 8.8 CCV:** Multiple Element Standard, purchased (Table 11) and diluted. Store at room temperature. Concentrations are at or near the mid-point of the calibration. Expires upon manufacturer's specified date.
- 8.9 SIC:** High-Purity Standards Cat. #ICP-MS-ICS-2 A stock. Dilute 5 mL to 50 mL (Table 12). Store at room temperature. Prepare fresh weekly.
- 8.10 LDR Check Solution:** A multiple element or single element solution run at a point above the highest calibration standard under the same calibration used to quantify the associated sample data. The LDR check must be within +/-10% of the true value of each element of interest and be within +/- 50% of the sample concentration to be consider valid.
- 8.11 Lower Limit of Quantitation (LLOQ) check standard:** The laboratory should establish the LLOQ as the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve. See section 12.16.
- 8.12 Argon Gas:** 0.9995 or better grade. High capacity tank plumbed into the lab.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Three types of blanks are required for the analysis. The calibration blank, which is also the ICB/CCB, is used in establishing the calibration curve. The ICB/CCB is used to monitor carryover, signal noise, and drift. The method blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards. See Section 12.3 for Corrective Action.

9.1.1 The Calibration blank, Initial Calibration Blank and the Continuing Calibration Blank consists of the same concentration of the same acid used to prepare the final dilution of the calibrating solutions of the analytes. This is a 1% HNO_3 solution (v/v) in Type I deionized water along with the selected concentrations of internal standard element for each of the analytes. The Calibration Blank is analyzed before the standards are analyzed. The Initial Calibration Blank must follow the Initial Calibration Verification standard (ICV) and the Continuing Calibration Blank must follow the Continuing Calibration Verification standard (CCV). These must be analyzed at a frequency of every 10 or less samples and at the end of the analytical run. The ICB must be no greater than $\frac{1}{2}$ the LLOQ for any analyte; The CCB must be no greater than the LLOQ for any analyte See Section 12.11 for Corrective Action.

9.1.2 The method (or reagent) blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the sample solutions. Method blanks are generally considered to be acceptable if target analyte

concentrations are less than the LLOQ or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e. targets are not present in samples or sample concentrations are $\geq 10X$ the blank). Other criteria may be used depending on the needs of the project. See Section 12.8 for Corrective Action.

- 9.1.3** The rinse blank consists of HNO₃ (1% or 2%) (v/v) in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

9.2 Laboratory Control Sample (LCS)

The Laboratory Control Sample (LCS) must be analyzed for each analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the test samples. One LCS must be prepared and analyzed for each sample batch at a frequency of one LCS for each 20 samples or less. Aqueous LCS recoveries must be 80-120%, and soil LCS recoveries must be within the SRM limits. See Sections 12.9 and 12.10 for Corrective Actions.

9.3 Initial Calibration Verification (ICV)

The ICV must have a recovery that is within $\pm 10\%$ of the true value. The ICV must contain all of the elements that are calibrated. The ICV is an independent source other than those standards used for the calibration of the instrument. The ICV must be analyzed following the calibration.

9.4 Continuing Calibration Verification (CCV)

The CCV must have a recovery that is within $\pm 10\%$ of the true value. The CCV must be analyzed at a frequency of 10 samples or less and at the end of the analytical run.

9.5 Matrix Spike

Analyze one matrix spike per twenty or less analytical samples. The recovery of the matrix spike must be between 75 – 125%. Calculate percent recovery using Section 11.2.

- 9.5.1 Dilution Test:** If the analyte concentration is sufficiently high (minimally, a factor of 25 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution must agree within $\pm 20\%$ of the original determination. Elements that fail the dilution test are reported as estimated values.

- 9.5.2 Post Digestion Spike Addition:** If the sample concentrations are insufficient to perform a dilution test a post digestion spike added to a portion of a prepared sample, or its dilution for the elements failing the matrix spike recoveries must be run, recovery limits equal to 75% to 125% of the known spike value. If the spike is not recovered within the specified limits If the post-digestion recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values

9.6 Laboratory Duplicate

Analyze one duplicate sample for every matrix in a batch at a frequency of one matrix duplicate for every 20 samples. Calculate RPD under section 11.3.

A control limit of 20% RPD must not be exceeded for analyte values greater than 100 times the instrument detection limit (Section 13.3). See Section 12.14 for Corrective Action.

9.7 Method-specific Quality Control Samples

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9.7.1 Internal Standards

The intensities of all internal standards must be monitored for every analysis. If the intensity of any internal standard applied to a sample falls below 70% of the intensity of that internal standard in the initial calibration standard, a significant matrix effect must be suspected.

Under these conditions, the established lower limit of quantitation has degraded and the correction ability of the internal standardization technique becomes questionable. The following procedure is followed -- First, make sure the instrument has not drifted by observing the internal standard intensities in the nearest calibration blank.

If the low internal standard intensities are also seen in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilution of the affected sample. The sample should be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. If the first dilution does not eliminate the problem, this procedure must be repeated until the internal-standard intensities rise to the minimum 70% limit. Reported results must be corrected for all dilutions.

9.7.2 Interference Check Standards

Verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the SIC check (8.9). Refer to Section 4.0 for a discussion on interferences and potential solutions to those interferences if additional guidance is needed. SIC must be below 2X LLOQ for all non-spiked elements of interest. See Section 12.8 for Corrective Action

9.7.3 Low Level Verification Standard (LLV)

The low level standard analyzed should quantitate to within 80-120% of the true value.

9.7.4 Mid Level Verification Standard (MLV)

The mid-level standard analyzed should quantitate to within 90-110% of the true value.

9.8 Method Sequence

Performance Check Solution / Tuning Solution
Calibration of instrument
Initial Calibration Verification Standard
Initial Calibration Blank
Low Level Verification Standard
Mid- Level Verification Standard
Spectral Interference Check Solution
Continuing Calibration Verification Standard
Continuing Calibration Blank
Samples (10)
Continuing Calibration Verification Standard

Continuing Calibration Blank
Samples (10)
Continuing Calibration Verification Standard
Continuing Calibration Blank

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

Prior to analysis, samples which require total (acid-leachable) values must be digested using appropriate sample preparation methods, such as Methods 3005A, 3015, 3051 and 3050B.

10.1.2 Tuning the instrument on from Stand By

- Tighten the peristaltic pump windings for the sample, Internal standard and spray chamber drain.
- Turn on the Argon gas supply and chiller.

Thermo Q and RQ:

- Turn on the computer and monitor. Start the Qtegra software. On the front instrument tab select the ON button, select OK. The fully automatic system will advance to ignition, torch box alignment and start the sample introduction system.
- Allow the instrument to warm up for **30 minutes**.

Thermo X-series II:

- Turn on the computer and monitor. Start the Plasmalab software. On the front instrument tab select the ON button. A message box will ask if to advance from vacuum to start up, select OK. The fully automatic system will advance to ignition, torch box alignment and start the sample introduction system.
- Allow the instrument to warm up for **30 minutes**.

10.1.3 Tuning

After the 30 minute warm up period has passed, a daily tune must be performed and a report generated.

Thermo X-series II and Thermo Q:

Select autotune, introduce the Tune A solution The Instrument stock tune solution (Table 9) is a 10 µg/L, Tune A is a 10 fold dilution. Run the autotune after a minimum 40 second uptake delay.

From the drop down menu select Run Performance Check (x –series), icon (Q) with the sample probe in Tune A solution. After completion of the performance check, select print and the file will be save to the archive. Upon successfully passing the performance check the instrument is ready for calibration.

Typical values of tuning parameters

Parameter	Typical Conditions	Adjustment
RF Power (W)	1300	1200 to 1600
Sampling Depth (mm)	6	4 to 8
Carrier gas (L/min)	1.2	0.8 to 1.3
Makeup gas (L/min)	0	0 to 0.4
Peri-pump 1 (rps)	0.1	0.06 TO 0.15
S/C Temp (°C)	2	Normally used at 2 °C
Extraction	-150	-200 to -100
Extraction	-70	-150 to -10
Einzel 1.3 (V)	-100	-130 to -40
Einzel 2(V)	7	-20 to +70
Omega Bias (V)	-35	-40 to 0
Omega (+)(V)	5	0 to +30
RF Power (W)	1300	1200 to 1600

Typical value of Sensitivity and RSD (Using the normal torch)

Mass	Counts / 10ppb Integ. Time = 0.1 sec	RSD
⁷ Li	>6400	<5%
⁸⁹ Y	>16000	<5%
²⁰⁵ Tl	>9600	<5%

10.2 Initial Calibration

Mixed calibration standard solutions are prepared by diluting the stock-standard solutions to levels within the linear range for the instrument in a solvent consisting of 5% HNO₃ (v/v) in reagent water. The calibration standard solutions must contain a suitable concentration of an appropriate internal standard for each analyte. Internal standards are added at the time of analysis using a second channel of the peristaltic pump and an appropriate mixing manifold. Generally, an internal standard must be no more than 50amu removed from the analyte. Alpha employs the following internal standards: ⁶Li, ⁴⁵Sc, ⁷⁴Ge, ¹⁰³Rh, ¹¹⁵In, ¹⁵⁹Tb, ¹⁷⁵Lu, ²⁰⁹Bi. Lithium is excluded when analyzing for ⁶Li/⁷Li.

Prior to preparing the mixed standards, each stock solution must be analyzed separately to determine possible spectral interferences or the presence of impurities. Care must be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to freshly acid-cleaned HPLC bottles for storage. Fresh mixed standards must be prepared as needed with the realization that concentrations can change upon aging. Calibration standards must be initially verified using a quality control standard (ICV), and monitored daily for stability.

Calibrate the instrument for the analytes of interest (recommended isotopes for the analytes in Table 1 are provided in Table 3), using the calibration blank (Section 9.1.1) and the set of 4 standards (see Table 4.). The analytical range brackets an RDL of 0.2 µg/L to 120 µg/L with the exceptions of Fe, K, Na, Ca and Mg which bracket the range of 10µg/L to 12,000µg/L. All solutions

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and standards are prepared in a 1% Nitric Acid matrix. The calibration is defined as the calibration blank and four standards. The only standards that may be discarded in the calibration are the first standard (the low standard) and the fourth standard (the high standard). The only instance when the standard may be discarded is if the linearity of the element in question does not meet the correlation coefficient acceptance criteria of 0.995 or greater. No mid-level standards are ever discarded. The LDR is defined by the LDR standard run within the same calibration and recovered with +/- 10% of the true value of each element and within +/- 50% of the sample concentration, no sample is to exceed 90% of the top LDR standard or a dilution must be performed. All standards, QC samples and samples are to be integrated three times and then averaged. The calibration block is part of the analytical sequence and includes an ICV, ICB, LLV, MLV immediately following the analysis of the calibration standards. The calibration block can also contain a pause so that the analyst can evaluate the calibration prior to sample analysis. The routine calibration sequence is as follows:

Blank #1
Cal blank
0.2 µg/L std (10 µg/L Fe, K, Na, Ca, Mg)
1.0 µg/L std (100 µg/L Fe, K, Na, Ca, Mg)
10.0 µg/L std (1000 µg/L Fe, K, Na, Ca, Mg)
120 µg/L std (12,000 µg/L Fe, K, Na, Ca, Mg)

The quality control standard is the Initial Calibration Verification solution (ICV), which must be prepared in the same acid matrix as the calibration standards. This solution must be an independent standard near but not at the midpoint of the instrument calibration. An independent standard is defined as a standard composed of analytes from a source other than that used for the standards for instrument calibration.

Immediately after the calibration has been established, the calibration must be verified and documented for every analyte by the analysis of the Initial Calibration Verification solution (ICV) (Section 8.7). The calibration is verified if the solution is within the 10% of the true value (Table 10) for each element. See Section 12 for corrective action if the ICV fails. The ICB (9.1.1) is analyzed immediately following the ICV. The ICB must be no greater than the ½ LLOQ for any analyte. See Sections 12 for Corrective Actions.

10.3 Equipment Operation and Sample Processing

10.3.1 Spectral Interference Check Solution

The interference check solution (SIC) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interference and provide an adequate test of any corrections.

Chloride in the SIC provides a means to evaluate software corrections for chloride-related interferences such as $^{35}\text{Cl}^{16}\text{O}^+$ on $^{51}\text{V}^+$ and $^{40}\text{Ar}^{35}\text{Cl}^+$ on $^{75}\text{As}^+$.

Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese.

Molybdenum serves to indicate oxide effects on cadmium isotopes.

The other components are present to evaluate the ability of the measurement system to correct for various molecular-ion isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits. The ICSA solution (Section 8.8) contains 20,000mg/L of Cl^- , 3,000 mg/L of Ca, 2,500 mg/L of Fe and Na, 2,000 mg/L of C, 1,000 mg/L of Al, K, Mg, P, S and 20 mg/L of Mo and Ti.

The non-target analytes for the SIC should have a recovery less than 2x LLOQ. Narrate nonconformance.

10.3.2 Sample Analysis and Continuing Calibration Verification

The default sample table on the instrument will include the ICV, ICB, LLV, MLV, SIC, samples and CCV and CCB. After the initial Instrument QC has been analyzed and passed, then 10 samples will be analyzed. After the tenth sample has been analyzed, a CCV and a CCB will be analyzed. Analysis of standards and samples must only take place when the instrument has come to equilibrium (typically after 30 minutes after plasma ignition). All masses which could affect data quality must be monitored to determine potential effects from matrix components on the analyte peaks. The recommended isotopes to be monitored are listed in Table 3. When the analysis is ready to start, flush the system with the rinse blank solution (Section 9.1.3) until the signal levels return to the method's level of quantitation (usually about 30 seconds) before the analysis of each sample. Nebulize each sample until a steady-state signal is achieved (usually in about 30 seconds) prior to collecting the data. Analyze the Continuing Calibration Verification solution (CCV) (Section 8.7) and the continuing Calibration blank (Section 9.1.1) at a frequency of at least once per ten analytical samples, and at the end of the analytical run. Dilute and reanalyze samples that are more concentrated than the linear range (or species needed for a correction) or measure an alternate less-abundant isotope if calibrated. The linearity at the alternate mass must be confirmed by appropriate calibration.

10.4 Preventive Maintenance

The scheduled maintenance is automatically tracked by the run time log for the following parameters. A message will appear on the computer screen reminding the analyst that a piece of maintenance must be performed. An electronic log, along with a maintenance notebook is kept. Below is a list what needs to be performed:

1. Check rough pump oil
2. Replace rough pump oil
3. Replace mist filter 4
4. Clean the extraction lenses
5. Clean the skimmer cone
6. Clean the sampling cone
7. Clean the nebulizer

11. Data Evaluation, Calculations and Reporting

The quantitative values are reported in appropriate units directly from the instrument: micrograms per liter ($\mu\text{g/L}$) for aqueous samples and milligrams per kilograms (mg/Kg) for solid samples. If dilutions were performed, the appropriate corrections must be applied to the sample values.

11.1 It is required that results for solids be reported on a dry weight basis as follows:

A separate determination of percent solids is performed by the Wet Chemistry Department and the result is loaded in the LIMS. To retrieve the result in the LIMS, go to "Status" and type in the sample ID. Click the sample number that is part of that sample ID, and click the product that says **TS-S**. The percent solids for the sample is there. LIMS will automatically

correct the sample for percent solids after the metals analysis has been **Final Metals Reviewed**. This is the calculation:

$$\text{Concentration (dry weight) (mg/kg)} = \frac{C \times V}{W \times S}$$

Where:

C= Digest Concentration (mg/L)

V= Final volume in Liters after sample preparation

W= Weight in Kg of wet sample

S= $\frac{\% \text{Solids}}{100}$

Calculations must include appropriate interference corrections (see Section 4.1 for examples), internal-standard normalization, and the summation of signals at 206, 207, and 208 m/z for Lead (to compensate for any differences in the abundances of these isotopes between samples and standards).

11.2 Calculate Percent Recovery for the Matrix Spike corrected for concentrations measured in the unfortified sample. Percent recovery is calculated using the following equation:

$$\% \text{ Recovery} = \frac{(C_m - C)}{S} \times 100$$

Where:

C_m = measured in the fortified sample

C = measured native sample concentration

S = concentration equivalent of spike added to sample

11.3 Calculate the Relative Percent Difference (RPD) for each Duplicate of the initial quantitated concentration (IC) and duplicate quantitated concentration (Dc) using the following formula:

$$\text{RPD} = \frac{|(IC - Dc)|}{\{(IC + Dc) / 2\}} \times 100$$

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

Holding time exceedances, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form

12.1 The performance check standard is included in the tuning solution. Ce and Ba are included with the tuning solution and monitor the formation of oxides and the effect of doubly-charged ions

- respectively. If the solution fails >3% (Section 10.1.3) then instrument maintenance must be performed and the solution re-run.
- 12.2** For the Tune to pass, the mass calibration for the three tune elements (Section 10.1.3) must not differ by more than 0.1 amu from the true value and the resolution must be <0.9 amu full width at 10% peak height. Furthermore the RSD for the three tune elements must be <5%. If any of these criteria fail then the mass calibration must be adjusted to the correct value. This is done by re-optimizing the instrument conditions and re-tuning.
- 12.3** The results of the calibration blank (Section 9.1.1) must be less than the LLOQ for each element. If this is not the case, the reason for the out-of-control condition must be found and corrected, and affected samples must be reanalyzed. If the laboratory consistently has concentrations greater than 3 times the LLOQ, the LLOQ may be indicative of an estimated LLOQ and must be re-evaluated.
- 12.4** Calibration is performed after the tune passes. A five point calibration is performed (Section 9.3). If more than three points are to be used for the calibration, then the resultant curve must have a correlation coefficient (cc) of 0.995 or greater. If the cc is <0.995, then the instrument must be re-optimized and re-calibrated. The calibration must define the working linear range of the curve. The reporting RL must be the low standard, and the upper linear range must be defined by the high standard.
- 12.5** The ICV (Section 9.3) is performed immediately after an acceptable calibration has been produced. The acceptable range for this standard is 90-110%. If this standard fails then the analysis must be stopped and the instrument re-calibrated, and the ICV re-analyzed. Analysis cannot continue until this standard passes.
- 12.6** The CCV (Section 9.4) is performed at a frequency of 10 samples or less and at the end of the analytical run. The acceptable range for this standard is 90-110%. If this standard fails it can be rerun once. If it continues to fail the instrument is re-calibrated, and the CCV re-analyzed. Analysis cannot continue until this standard passes. All samples run after the last passing CCV must be rerun for the CCV failures.
- 12.7** The ICB (Section 9.1.1) must be performed immediately after the ICV. The ICB must be $\frac{1}{2}$ LLOQ. If the ICB fails then the analysis is terminated, sources of contamination are checked for, and the instrument is re-calibrated and the ICV and ICB are re-analyzed.
- 12.8** The Spectral Interference Check Solution (Section 9.7.2) monitor how well the system is correcting for interference. The target non-spiked elements in SIC must be below 2X LLOQ for those elements in question. If the recovery of this solution is outside of the control limits, the non-compliance must be narrated. There is no corrective action required because instrument corrections are based on natural isotope abundances that cannot be changed. If the IS is in compliance then the data is acceptable.
- 12.9** The Method (Preparation) Blank (Section 9.1.2) must be less than the LLOQ for all of the elements that are being analyzed. If the element in question is non-detect, and the method blank is positive then the corrective action is a narration on the final report. If the samples associated with the method blank have "hits", and they are 10x greater than the method blank, then the corrective action is a narration on the final report. If the method blank is not less than the RL, and the associated samples have results that are greater than the RL but less than 10x the method blank, then the samples must be re-digested and re-analyzed.
- 12.10** If the LCS (Section 9.2) fails, then all samples associated with that batch must be re-digested and re-analyzed. (Massachusetts recognizes that if the MS passes and the LCS fails then the data can be accepted. Narrate then non-compliance. This is only for MCP projects).

- 12.11** Failure of the CCB (9.1.1) rerun once, if continued failure evaluate the data; if associated sample results are greater than 10x CCB level then the results are acceptable. Otherwise, recalibrate and re-analyze all samples since last compliant CCB.
- 12.12** If the dilution test (Section 9.5.1) fails, then an interference may be suspected. There is no corrective action to be applied. Report all elements as estimated on the final report if the sample concentration is 25x LLOQ.
- 12.13** If the Post-Digestion Spike (Section 9.5.2) fails, then matrix interference may be suspected and a dilution should be performed to confirm. Report all elements as estimated on the final report.
- 12.14** Failure of the duplicate sample (Section 9.6) must be investigated. If the sample is found to be non-homogeneous or less than 5 x the LLOQ then the non-compliance must be narrated on the final report.
- 12.15** If the Internal Standards (Section 9.7.1) fail the acceptance criteria, then dilute the samples until the IS passes. If the criteria are still not met, then terminate the analysis, re-calibrate, verify the new calibration, and reanalyze all of the affected samples. If IS continues to fail at dilutions, seek an alternate technology to analyze the affected samples.
- 12.16** The LLOQ is initially verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be +/- 35% of the true value and RSD should be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.
- Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

13. Data Evaluation, Calculations and Reporting

- 13.1** If dilutions were performed, the appropriate factors must be applied to sample values. All results must be reported with up to three significant figures.

13.2 Soil samples

Soil samples are calculated as follows:

$$A = \frac{\text{Sample weight (grams)}}{\text{Final Volume (mL)}}$$

$$B \text{ (concentration in mg/Kg)} = \frac{\text{Concentration of analyte (mg/L)}}{A}$$

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13.2.1 Dry weight correction

The LIMS calculates the dry weight correction, however it is calculated as follows:

$$\text{Final concentration in mg/Kg dry weight} = \frac{B}{\% \text{ Solids}}$$

13.3 Liquid samples

Liquid samples are calculated as follows:

$$\text{Dilution Factor} = \frac{\text{Final Volume (mL)}}{\text{Sample Volume (mL)}}$$

Final concentration in mg/L = Concentration of analyte (mg/L) x Dilution Factor

13.4 Calculations for Hardness

The method for determining hardness is to compute it from the results of separate determinations of Calcium and Magnesium on aqueous samples.

13.4.1 Total Hardness

Total Hardness, mg equivalent CaCO₃/L = [2.497 (Ca, mg/L)] + [4.118 (Mg, mg/L)]

13.4.2 Calcium Hardness

Calcium Hardness, mg equivalent CaCO₃/L = [2.497 (Ca, mg/L)]

14. Method Performance

14.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732 unless superseded in the SOP. These studies performed by the laboratory are maintained on file for review

14.2 Demonstration of Capability Studies

14.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

14.2.2 Continuing (DOC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples

Printouts of this document may be out of date and should be considered uncontrolled. To accomplish work, the published version of the document should be viewed online.

14.3 Instrument Detection Limits (IDLs)

Instrument detection limits (IDLs) are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation, however, it should be understood that the lower limit of quantitation needs to be verified.

Instrument Detection limits (in $\mu\text{g/L}$) can be estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e. each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least once using new equipment, after major instrument maintenance such as changing the detector, and/or at a frequency designated by the project and a record of time and date kept in the instrument maintenance log book.

15. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

16. Referenced Documents

DOC ID 2124 Chemical Hygiene Plan

SOP ID 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

SOP ID 1739 Demonstration of Capability (DOC) Generation SOP

SOP ID 1728 Hazardous Waste Management and Disposal SOP

17. Attachments

Table 2: Reporting Limits

Table 3: Interference Check Solution Concentrations

Table 4: Calibration Standards

Table 5: Recommended Isotopes for Selected Elements

Table 6: Precision and Accuracy Acceptance Criteria

Table 7: Metals LCS Concentrations

Table 8: Internal Standard

Table 9: Tune Solution

Table 10: ICV Solution

Table 11: CCV Solution

Table 12: Interference Check Solutions

Table 13: Interference Correction Equations

Table 2
Reporting Limits

Element	Atomic Symbol	Mass (m/z)	Aqueous (µg/L)	Soil / Solid (ug/Kg)
Beryllium	Be	9	0.5	20
Sodium	Na	23	100	4000
Magnesium	Mg	24	100	4000
Aluminum	Al	27	10	400
Potassium	K	39	100	4000
Calcium	Ca	44	100	4000
Vanadium	V	51	0.50	20
Chromium	Cr	52	0.50	20
Manganese	Mn	55	0.50	20
Iron	Fe	57	50	2000
Cobalt	Co	59	0.50	20
Nickel	Ni	60	0.50	20
Copper	Cu	65	0.50	20
Zinc	Zn	66	5.0	100
Arsenic	As	75	0.50	20
Selenium	Se	82	0.50	20
Molybdenum	Mo	98	0.50	20
Silver	Ag	107	0.50	20
Cadmium	Cd	111	0.50	20
Antimony	Sb	121	0.50	20
Barium	Ba	137	0.50	20
Thallium	Tl	205	0.50	20
Lead	Pb	208	0.50	20
Lithium ¹	Li	7	N/A	100
Internal Standards				
Lithium	Li	6		
Scandium	Sc	45		
Germanium	Ge	74		
Indium	In	115		
Bismuth	Bi	209		

¹ Lithium is performed only by request for limited client projects with project specific reporting limits.

Calculated Method Detection Limits are on file in the QA Department.

Table 3
Interference Check Solution

Solution Component	Solution A Concentration (ug/L)
Al	100,000
Ca	300,000
Fe	250,000
Mg	100,000
Na	250,000
P	100,000
K	100,000
S	100,000
C	200,000
Cl	2,000,000
Mo	2,000
Ti	2,000

Table 4 Calibration Standards

Multi-element standard (Section 8.4) containing the following:

High Range Elements (HR): 1000 mg/L of the following: Fe, K, Ca, Na, Mg, Sr

Low Range Elements (LR): 10 mg/L of the following: Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Th, U

All Calibration Standards are purchased in 5% HNO₃, Tr HF (v/v).

Lithium is analyzed with a single standard curve matching the levels listed below for the routine curve.

Standard #1 (0.2 µg/L LR, 20 ug/L HR): Dilute Standard #3: 1.0 mL to 50mL final volume 1% HNO₃ (v/v).

Standard #2 (1.0 µg/L LR, 100 ug/L HR): Dilute Standard #3: 5.0 mL to 50mL final volume 1% HNO₃ (v/v).

Standard #3 (10.0 µg/L LR, 1000 ug/L HR): Dilute 0.05 mL to 50mL final volume 1% HNO₃ (v/v).

Standard #4 (60.0 µg/L LR, 1000 ug/L HR): Dilute 0.3 mL to 50mL final volume 1% HNO₃ (v/v).

Standard #5 (120.0 µg/L LR, 12000 ug/L HR): Dilute 0.6 mL to 50mL final volume 1% HNO₃ (v/v).

Table 5: Recommended Isotopes for Selected Elements

<u>Mass</u>	<u>Element of Interest</u>
<u>27</u>	Aluminum
121, <u>123</u>	Antimony
<u>75</u>	Arsenic
138, 137, 136, <u>135</u> , 134	Barium
<u>9</u>	Beryllium
<u>209</u>	Bismuth (IS)
<u>114</u> , 112, <u>111</u> , 110, 113, 116, 103	Cadmium
42, 43, <u>44</u> , 46, 48	Calcium (I)
35, 37, (77, 82) ^a	Chlorine (I)
<u>52</u> , <u>53</u> , <u>50</u> , 54	Chromium
<u>59</u>	Cobalt
<u>63</u> , <u>65</u>	Copper
165	Holmium (IS)
<u>115</u> , 113	Indium (IS)
<u>56</u> , <u>54</u> , <u>57</u> , 58	Iron (I)
139	Lanthanum (I)
<u>208</u> , <u>207</u> , <u>206</u> , 204	Lead
6 ^b , 7	Lithium (IS)
24, <u>25</u> , <u>26</u>	Magnesium (I)
<u>55</u>	Manganese
98, 96, 92, <u>97</u> , 94, (108) ^a	Molybdenum (I)
58, <u>60</u> , 62, <u>61</u> , 64	Nickel
<u>39</u>	Potassium (I)
103	Rhodium (IS)
45	Scandium (IS)
80, <u>78</u> , <u>82</u> , <u>86</u> , <u>77</u> , 74	Selenium
<u>107</u> , <u>109</u>	Silver
<u>23</u>	Sodium (I)
159	Terbium (IS)
<u>205</u> , 203	Thallium
<u>51</u> , <u>50</u>	Vanadium
120, <u>118</u>	Tin (I)
89	Yttrium (IS)
64, <u>66</u> , <u>68</u> , <u>67</u> , 70	Zinc

NOTE: Method 6020 is recommended for only those analytes listed in Table 1. Other elements are included in this Table because they are potential interferents (I) in the determination of recommended analytes, or because they are commonly used internal standards (IS). Isotopes are listed in descending order of natural abundance. The most useful isotopes are underlined and in boldface, although certain matrices may require the use of alternative isotopes.

^a These masses are also useful for interference correction (Section 4.2)

^b Internal standard must be enriched in the 6Li isotope. This minimizes interference from indigenous Li

Table 6 Precision and Accuracy Acceptance Criteria *

Element	Aqueous % Recovery LCS		Soil % Recovery LCS or SRM limits		Duplicate	
	Lower Control Limit	Upper Control Limit	Lower Control Limit	Upper Control Limit	Aqueous %RPD	Soil %RPD
Aluminum	80	120	70	130	20	20
Antimony	80	120	70	130	20	20
Arsenic	80	120	70	130	20	20
Barium	80	120	70	130	20	20
Beryllium	80	120	70	130	20	20
Cadmium	80	120	70	130	20	20
Calcium	80	120	70	130	20	20
Chromium	80	120	70	130	20	20
Cobalt	80	120	70	130	20	20
Copper	80	120	70	130	20	20
Iron	80	120	70	130	20	20
Lead	80	120	70	130	20	20
Magnesium	80	120	70	130	20	20
Manganese	80	120	70	130	20	20
Molybdenum	80	120	70	130	20	20
Nickel	80	120	70	130	20	20
Potassium	80	120	70	130	20	20
Selenium	80	120	70	130	20	20
Silver	80	120	70	130	20	20
Sodium	80	120	70	130	20	20
Thallium	80	120	70	130	20	20
Vanadium	80	120	70	130	20	20
Zinc	80	120	70	130	20	20

*These are default limits. The limits are re-evaluated and updated as necessary pending compilation of the minimum number of data points

Table 7 Metals LCS Concentrations

Analyte	Liquid Concentration (mg/L)
Antimony	0.5
Arsenic	0.12
Barium	2.00
Beryllium	0.05
Cadmium	0.051
Chromium	0.20
Copper	0.25
Lead	0.51
Nickel	0.50
Selenium	0.12
Silver	0.05
Thallium	0.12
Zinc	0.50
Iron	1.00
Manganese	0.50
Calcium	10.0
Magnesium	10.0
Potassium	10.0
Sodium	10.0
Aluminum	2.00
Cobalt	0.50
Vanadium	0.50

Table 8 Internal Standard

Multiple Element Standard, purchased (Section 8.5), 100mg/L of ⁶Li, ⁴⁵Sc, ⁷⁴Ge, ¹⁰³Rh, ¹¹⁵In, ¹⁵⁹Tb, ¹⁷⁵Lu, ²⁰⁹Pb

Working Internal Standard (IS) Solution (1 mg/L solution) Dilute 10.0 mL to 1L final volume 1% HNO₃ (v/v).

Table 9 Tune Solution

Tune Solution: 100 ug/L multi-element standard, purchased for ⁷Li ⁹Be, ⁵⁸Ce ⁵⁹Co, ¹¹⁵In, ¹³⁷Ba and ²⁰⁸Pb, ²³⁸U in 1% HNO₃ (v/v) (Section 8.6).

Working Tune Solution: Dilute 1.0 mL of 100 ug/L multi-element standard for ⁷Li ⁹Be, ⁵⁸Ce ⁵⁹Co, ¹¹⁵In, ¹³⁷Ba and ²⁰⁸Pb, ²³⁸U with 1% HNO₃ (v/v) to 100mL final volume. This will give a final concentration of these metals at 1.0µg/L.

Table 10 ICV Solution

Multiple Element Standard, purchased (Section 8.7):

1000mg/L of K, Na, Ca, Mg, Fe, Sr

10mg/L of Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, An, Th, U

Working ICV Solution: Dilute 1.0 mL with 0.25% HNO₃ (v/v) to 50mL final volume. This will give the following concentrations:

5000µg/L of K, Na, Ca, Mg, Fe, Sr

50µg/L of Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, An, Th, U

Table 11 CCV Solution

Multi-element standard (Section 8.4) containing the following:

High Range Elements (HR): 1000 mg/L of the following: Fe, K, Ca, Na, Mg, Sr

Low Range Elements (LR): 10 mg/L of the following: Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Th, U

All Standards are purchased in 5% HNO₃, Tr HF (v/v).

60.0 µg/L LR, 1000 µg/L HR: Dilute 0.3 mL to 50mL final volume 1% HNO₃ (v/v).

Table 12 Spectral Interference Check Solutions

SIC, High-Purity Standards Cat. #ICP-MS-ICS-2 A (Section 8.8):

20000 mg/L of Cl⁻ **3000 mg/L Ca**

2500 mg/L Fe, Na

2000 mg/L of C

1000 mg/L of Al, K, Mg, P, S

20 mg/L of Mo, Ti

Working SIC Solution: Dilute 5 mL of High-Purity Standards Cat. #ICP-MS-ICS-2 A, with 1% HNO₃ (v/v) to 50mL final volume. This will give the following concentrations:

2000 mg/L of Cl⁻

300 mg/L Ca

250 mg/L Fe, Na

200 mg/L of C

100 mg/L of Al, K, Mg, P, S

2 mg/L of Mo, Ti

Table 13 Interference Correction Equations

Correction equations are in the form: corrected signal for mass X=(signal from mass X) ± (signal from mass Y)*(correction factor).

$$6 = (6) - ((7) * 0.082)$$

$$44 = (44) - ((88) * 0.015)$$

$$51 = (51) - ((53) * 3.0) + ((52) * 0.34)$$

$$66 = (66) - ((69) * 0.00141)$$

$$75 = (75) - ((77) * 2.9) + ((82) * 2.23) - ((83) * 2.23)$$

$$82 = (82) - (83)$$

$$98 = (98) - ((99) * 0.146)$$

$$111 = (111) - ((108) * 1.073) + ((106) * 0.712)$$

$$114 = (114) - ((118) * 0.027)$$

$$115 = (115) - ((118) * 0.016)$$

$$208 = (208) + (206) + (207)$$

Mercury in Liquid Waste (Automated Cold-Vapor Technique)

Reference Method No.: **EPA 7470A**

Reference: SW-846, Test Methods for Evaluating Solid Waste:
Physical/Chemical Methods, EPA SW-846, Update II, September
1994.

1. Scope and Application

Matrices: Method 7470 is a cold-vapor atomic absorption procedure approved for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. (Method 7470 can also be used for analyzing certain solid and sludge-type wastes; however, Method 7471 is usually the method of choice for these waste types.) All samples must be subjected to an appropriate dissolution step prior to analysis.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Mercury Analyzer and in the interpretation of Mercury data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Prior to analysis, the liquid samples must be prepared according to the procedure discussed in this method.

Method 7470, a cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.1 Method Modifications from Reference

- 2.1.1** A smaller sample sized is prepared, and therefore proportionately less reagent volumes are used.
- 2.1.2** The original method does not address the automated instrument procedure.

3. Reporting Limits

The typical reporting limit for Mercury is 0.0002mg/L. This satisfies Massachusetts, GW1 and GW 2 criteria. Connecticut mobility criteria for SPLP is 0.0004mg/L, Rhode Island is 0.002mg/L, and the Drinking Water reporting limit is 0.0002mg/L.

4. Interferences

Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL). Both inorganic and organic mercury spikes have been quantitatively recovered from seawater by using this technique.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analysis is conducted under a laboratory exhaust hood. The analyst must wear chemical resistant gloves when handling concentrated mercury standards.

The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Therefore, the acidification of samples is to be conducted under a laboratory exhaust hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in either glass or plastic containers.

6.2 Sample Preservation

Samples are preserved with HNO₃ to a pH of <2.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are stored under refrigeration at $4 \pm 2^{\circ}\text{C}$ and analyzed as soon as possible after collection. The samples have a 28-day holding time from the time of collection.

7. Equipment and Supplies

Instrumentation:

Perkin Elmer FIMS 100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. The PE FIMS is designed specifically for the measurement of mercury using the cold-vapor technique with BOC (background offset correction) performed by a survey scan prior to each sample introduction. PE S10 autosampler is coupled to the instrument.

Cetac M-6100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. This instrument employs a reference cell off-set correction and full automation through the CETAC software. A Cetac ASX-260 autosampler is coupled to the instrument.

Nippon Instrument model# RA-4300A analyzer with integrated 80 position autosampler:

The instrument adds a stannous chloride (II) solution to the sample post digestion, the divalent mercury ion (Hg^{2+}) is reduced to zero-valent metallic mercury and turns into mercury gas by bubbling. $\text{Hg}^{2+} + \text{SnCl}_2 \rightarrow \text{Hg}^0 \uparrow$

After removing the acid mist and water vapor generated by bubbling with an electronic cooling unit, the instrument measures the absorbance of mercury at 253.7 nm absorption wavelength. It measures the known mercury amount, creates a calibration curve, and then calculates the mercury amount from the absorbance.

- 7.1 **Graduated cylinder:** Rinse once with 50% HNO_3 and then rinse with reagent water prior to use.
- 7.2 **Volumetric Flasks, Class A, various volumes:** Rinse once with 50% HNO_3 and then rinse with reagent water prior to use.
- 7.3 **Heating Block:** Environmental Express HotBlock, 48 position capacity, able to maintain $95^{\circ}\text{C} \pm 3$.
- 7.4 **50 mL Digestion Tubes:** Polypropylene, graduated.
- 7.5 **50 mL Digestion Tube Rack:** 48 position, racklock
- 7.6 **Pump tubing:** Environmental Express, three stop and two in various IDs.
- 7.7 **PTFE membranes:** Whatman TE37 disks.
- 7.8 **Dilution vials:** 20mL capacity, used to prepare analytical dilutions.
- 7.9 **Low Dust Laboratory Wipes**
- 7.10 **Compressed Air**
- 7.11 **Whatman 41 filter paper or equivalent**

8. Reagents and Standards

- 8.1 Reagent Water:** Reagent water is DI water shown to be interference free. All references to water in this method will refer to reagent water unless otherwise specified.
- 8.2 Sulfuric acid (H₂SO₄), concentrated:** Reagent grade. Store at room temperature in an appropriately designated acid cabinet.
- 8.3 Hydrochloric acid, concentrated:** Trace Metal grade. Store at room temperature in an appropriately designated acid cabinet.
- 8.4 Carrier, Hydrochloric Acid, 3%:** This is the *carrier* for the Instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl (Section 8.3). Bring to volume with reagent water. Store at room temperature, prepare daily as needed.
- 8.5 Reductant, Stannous Chloride in 3% HCl:** This is the *reductant* for the Instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl and 11g SnCl₂ · 2H₂O. Mix to dissolve the solid and bring to volume with reagent water. Store at room temperature, prepare daily as needed.
- 8.6 Nitric acid (HNO₃), concentrated:** Trace metal grade of low mercury content. If a high reagent blank is obtained, it may be necessary to distill the nitric acid. Store at room temperature in an appropriately designated acid cabinet.
- 8.7 Sodium chloride-hydroxylamine hydrochloride solution:** Dissolve 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride in reagent water and dilute to 100mL. Store at room temperature. Expires one month from date of preparation.
- 8.8 Potassium permanganate, mercury-free, 5% solution (w/v):** Dissolve 5 g of potassium permanganate in 100 mL of reagent water. Store at room temperature. Expires one month from date of preparation.
- 8.9 Potassium persulfate, 5% solution (w/v):** Dissolve 5 g of potassium persulfate in 100 mL of reagent water. Store at room temperature. Expires one month from date of preparation.
- 8.10 Mercury Stock Standard, 1000ppm:** Purchased from a commercial source with a certificate of analysis. Purchase three different sources. Store at room temperature. Expires upon manufacturer's specification.
- 8.11 Mercury Stock Calibration Standard, 10ppm:** To a 100mL volumetric flask, add 5mL of concentrated HNO₃, 2.5mL of concentrated H₂SO₄ and 1000ppm Mercury Stock Standard (Section 8.10, use one source). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.
- 8.12 Mercury Working Calibration Standard / Matrix Spike Solution, 0.1ppm:** To a 100mL volumetric flask, add 5mL of concentrated HNO₃, 2.5mL of concentrated H₂SO₄ and 1mL of 10ppm Mercury Stock Standard (Section 8.11). Bring to volume with reagent water. Store at room temperature. Make fresh daily.
- 8.13 Mercury Calibration Standards:** All calibration standards are prepared daily.
- 8.13.1 0 ppm Calibration Standard:** Add 10 mL of reagent water to a polypropylene digestion vessel. This aliquot may be used for the CCB. Another separate aliquot is prepared in the same manner for use as the ICB and diluent for sample dilutions.

- 8.13.2 0.0002ppm Calibration Standard:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 0.05 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.
- 8.13.3 0.001ppm Calibration Standard:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 0.25 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.
- 8.13.4 0.002ppm Calibration Standard** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 0.5 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.
- 8.13.5 0.005ppm Calibration Standard/Continuing Calibration Verification Standard:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 1.25 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.
- 8.13.6 0.010ppm Calibration Standard / Post Analytical Spike Solution:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 2.5 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.
- 8.13.7 0.020ppm Calibration Standard / Post Analytical Spike Solution:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 5.0 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.
- 8.14 Mercury Stock LCS Standard, 10ppm:** To a 100mL volumetric flask add 25mL of reagent water and 5mL of concentrated HNO₃ (Section 8.6). Add 1mL of 1000ppm Mercury Stock Standard (Section 8.10). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.
- 8.15 Mercury Working LCS Standard, 0.1ppm:** To a 100mL volumetric flask add 25mL of reagent water and 5mL concentrated HNO₃ (Section 8.6). Add 1mL of 10ppm Stock LCS Standard (Section 8.14). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.
- 8.16 Mercury LCS Standard, 0.001ppm:** Prepare daily with each batch of samples. To a 50mL digestion vessel add 10mL of reagent water Add 0.25 mL of 0.1ppm Working LCS Standard (Section 8.15). Bring to a final volume of 25mL and carry through entire digestion process as in Section 10.1.1.
- 8.17 Mercury Stock ICV Standard, 10ppm:** To a 100mL volumetric flask add 25mL of reagent water and 5mL of concentrated HNO₃ (Section 8.6). Add 1mL of 1000ppm Mercury Stock Standard (Section 8.10-use alternate source than that used for both the calibration standards and the LCS Stock Standard). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.
- 8.18 Mercury Working ICV Standard, 0.3ppm:** To a 100mL volumetric flask add 25mL of reagent water and 5mL of concentrated HNO₃ (Section 8.6). Add 3mL of 10ppm Stock ICV Standard (Section 8.5). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.

8.19 Mercury ICV Standard, 0.003ppm: Prepare daily with each batch of samples. To a 25mL digestion vessel add 10mL of reagent water. Add 0.25mL of 0.3ppm Working ICV Standard (Section 8.18). Bring to a 25mL final volume with reagent water and carry through entire digestion process as in Section 10.1.1..

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

The ICB, CCB, and Method Blank: A 25mL aliquot of 0ppm calibration standard brought through the preparation procedure as outlined in Section 10.1.1. Blank results must be <RL. See Section 12.1 for corrective action. An ICB is analyzed after the initial calibration or re-calibration. The CCB is analyzed at every 10 sample injection interval. Method Blank is analyzed once per batch of samples; batch consists of 20 samples.

9.2 Laboratory Control Samples (LCS)

The LCS Standard consists of a 0.001ppm Mercury LCS Standard (Section 8.16). This standard is brought through the preparation procedure as outlined in Section 10.1.1. The LCS Standard must be recovered within $\pm 20\%$ of the true value. See Section 12.3 for corrective action. The LCS Standard is analyzed once per batch of samples. A batch consists of 20 samples.

9.3 Initial Calibration verification (ICV)

The ICV Standard consists of a 25mL aliquot of 0.003ppm Mercury ICV Standard (Section 8.19). The ICV must be recovered within 10% of the true value. See Section 12.2 for corrective action.

9.4 Continuing Calibration Verification (CCV)

The CCV Standard consists of a 0.010ppm calibration standard (Section 8.13.3). The CCV must be recovered within 20% of the true value. See Section 12.2 for corrective action.

9.5 Matrix Spike

A matrix spike is analyzed once per batch of samples. A batch consists of 20 samples for monitoring wells, surface waters, influents and effluents. Prepare the matrix spike at 0.005ppm by adding 1.25mL of 0.1ppm Mercury Stock Standard (Section 8.3) to 25mL of the selected QC sample. The final concentration of the matrix spike is 0.005mg/L.

The matrix spike sample is brought through the preparation procedure as outlined in Section 10.1. A matrix spike is analyzed once per batch of samples. A batch consists of 20 samples for monitoring wells and surface waters. The recovery of the matrix spike must be between 75 – 125% (using the calculation in Section 11.2).

If the recovery of the matrix spike is out of range, a post-analytical spike is analyzed. Prepare the post analytical spike by adding 5mL of 0.010ppm Calibration Standard / Post Analytical Spike Solution (Section 8.1.6) and 5mL of the sample digestate to a 50mL centrifuge tube for a final concentration of 0.005mg/L. Analyze the post spike as outlined in Section 10.3.5.

Calculate the post spike concentration as follows:

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Post Analytical Spike Sample Concentration (mg/L) =

$$[\text{Sample Concentration (mg/L)} \times (0.5)] + 0.005\text{mg/L}$$

The percent recovery of the post-analytical spike must be between 75 – 125%. See Section 12.4 for corrective action.

9.6 Laboratory Duplicate

A sample is analyzed in duplicate once per batch of samples. A batch consists of 20 samples for monitoring wells and surface waters. The RPD between the sample and its duplicate must be 20% or less (using the calculation in Section 11.3), See Section 12.5 for corrective action.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Calibration Blank
- 0.0002 ppm Calibration Standard
- 0.0005 ppm Calibration Standard
- 0.001 ppm Calibration Standard
- 0.002 ppm Calibration Standard
- 0.010 ppm Calibration Standard
- 0.020 ppm Calibration Standard
- ICV
- ICB
- Ten analytical samples
- CCV
- CCB
- Ten analytical samples
- CCV
- CCB
- Etc.

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation and Digestion

Samples, Standards and All Batch QC

Transfer 25mL of well-homogenized sample (or an aliquot of the sample diluted to 25mL with reagent water) or standards (Sections 8.13.1 through 8.13.7, 8.16 and 8.19) to a 50mL centrifuge tube.

Add 1.25mL of concentrated H₂SO₄ (Section 8.2), 0.625mL of concentrated HNO₃ (Section 8.6), Add 3.75mL of Potassium Permanganate Solution, shake and add additional portions of potassium permanganate solution (if necessary) to all samples and QC, until the purple color persists for at least 15 min. (Section 8.8). Add 2mL of Potassium Persulfate Solution (Section 8.9), and heat samples for 2 hours in a 95°C +/-3

heating block. Cool, and add 1.5mL of Sodium Chloride-Hydroxylamine hydrochloride solution (Section 8.7).

Filter the sample if needed to remove any sediment or particulate.

Analyze samples and the digested calibration standards (Sections 8.13.1 through 8.13.7) are used in Section 9.2 to generate a calibration curve.

10.2 Initial Calibration

Construct a calibration curve by plotting the absorbances of prepared standards (Section 10.1.1) versus micrograms of mercury. Determine the peak height of the unknown from the absorbance maxima on the spectrometer, and read the mercury value from the standard curve. (See Section 11.)

The curve correlation coefficient (cc) must be greater than or equal to 0.995 in order for the curve to be linear. If the correlation coefficient is less than 0.995, find and correct the problem. When the problem has been corrected, re-analyze either the previous standards or new standards. When the curve has generated an acceptable cc then the analysis can continue with the ICV/ICB.

10.3 Equipment Operation and Sample Processing

Sample and standard analysis:

10.3.1 Instrument Setup

10.3.1.1 Turn the instrument on. The autosampler will initialize itself.

10.3.1.2 Choose the instrument software from the desktop menu. The autosampler will initialize again.

FIMS 100 NOTE: The instrument must be turned on before the application is started. Otherwise, an error message will result.

10.3.1.3 Enter the appropriate fields for sample identification, and data storage.

10.3.1.4 Fill the carrier and reductant bottles.

10.3.1.4.1 The Carrier is 3% HCl (Section 8.6).

10.3.1.4.2 The Reductant is 1.1% SnCl₂ in 3% HCl (Section 8.5).

10.3.1.5 Allow the instrument to warm up while clearing samples. Samples that are cloudy or with particulate present after clearing must be filtered through Whatman 41 filter paper (Section 7.11) before analysis.

10.3.1.6 Place carrier uptake line and reductant uptake line.

10.3.1.7 Load carrier and reductant lines into pump magazines

10.3.1.8 Load the two waste lines into the pump magazines below the roller.

10.3.1.9 Lock the magazines into place.

FIMS100 only:

10.3.1.10 Remove the cap from the liquid/vapor separator and wipe dry with a Lab Wipe (Section 7.9). Compressed air (Section 7.10) through the vapor transfer line to dry it out.

- 10.3.1.11 Place a PTFE membrane (Section 7.7), rough side up, in the liquid/vapor separator; replace the cap and reattach the vapor transfer line to the sample absorption cell.
- 10.3.1.12 Adjust the gas flow by turning the black knob below the air flow meter to obtain a reading of just over 50.

10.3.2 Calibration and Sample Analysis

- 10.3.2.1 The instrument will now run the calibration standards; verify a CC of 0.995 or better before proceeding with the ICV and ICB. Ten analytical samples, a CCV and CCB, ten analytical samples, CCV, CCB, etc. The CCBs and CCVs must be recovered within the proper ranges (Sections 9.4 and 9.1.3) for analysis to continue.
- 10.3.2.2 If the sample result is beyond 90% of the concentration of the highest point on the calibration curve or LDR study used to establish the linear range, dilute the sample extract with a portion of one of the prepared blanks (ICB, CCB or PBS) to produce an analytical result that is within the range.

10.3.3 Instrument Shut Down

- 10.3.3.1 When analysis is complete place reagent uptake lines in a beaker of reagent water.
 - 10.3.3.1.1 Continue to run the pumps for several minutes to flush reagents out of the lines.
 - 10.3.3.1.2 Continue to run the pumps for several minutes to flush reagents out of the lines.
- 10.3.3.2 Pull the reagent uptake lines out of the reagent water beaker to allow the pump to draw air through the lines.
- 10.3.3.3 Unlock the top and bottom pump magazines and remove tubing from the magazines.
- 10.3.3.4 Exit from the software application.
 - 10.3.3.4.1 Dump the samples and instrument waste in the Metals/WetChem waste drum located in the transfer room.

10.4 Continuing Calibration

Continuing calibration verification samples are analyzed after every 10 samples in the sample run, as outlined in Section 10.3.5.

10.5 Preventative Maintenance

Preventative maintenance is conducted per the manufacturer's instructions. All preventative maintenance is recorded in the Instrument Maintenance Logbook.

11. Data Evaluation, Calculations and Reporting

11.1 Calculate Mercury concentration

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Calculate Mercury concentration from the daily calibration curve. The curve is generated utilizing a straight-line equation defined as:

$$A = k_1 + k_2C$$

Where:

A = Average peak height of the sample/standard integrations
C = Sample/Standard Concentration, $\mu\text{g/L}$
 k_1 = y-intercept
 k_2 = slope

The instrument will plot peak height against concentration ($\mu\text{g/L}$). The result is generated in $\mu\text{g/L}$. This value is divided by 1000 to convert the units to mg/L . If the sample is diluted (DF), the result is multiplied by the DF to generate the final result.

$$\text{Result, mg/L} = (\text{concentration, } \mu\text{g/L}) \times (1\text{mg}/1000\mu\text{g}) \times (\text{DF})$$

11.2 Matrix Spike Calculation

Calculate percent recovery for the Matrix Spike corrected for concentrations measured in the unfortified sample. Percent recovery is calculated using the following equation:

$$\% \text{ Recovery} = \frac{(C_m - C)}{S} \times 100$$

Where:

C_m = measured Mercury in the fortified sample
 C = measured native mercury sample concentration
 S = concentration equivalent of spike added to sample

11.3 Relative Percent Difference (RPD) Calculation

Calculate the Relative Percent Difference (RPD) for each Duplicate of the initial quantitated concentration (IC) and duplicate quantitated concentration (Dc) using the following formula:

$$\text{RPD} = \frac{|(IC - Dc)|}{\{(IC + Dc) / 2\}} \times 100$$

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

12.1 Method Blank Failure: When a prep blank mercury level constitutes 10% or more of analyte level determined for any sample in the batch, or is greater than 2.2x the MDL value (whichever is greater), the associated samples in the batch must be redigested (Section 10.1).

For method blanks that have concentrations greater than the RL, the data is rejected and the associated samples sent back for redigestion unless the associated sample concentrations are greater than 10x the blank concentration. In this case the blank is narrated and the results are reported without qualification.

12.2 ICV / CCV Failure: If the ICV %Recovery is outside of acceptance criteria, the ICV is reinjected. If the %Recovery is outside the acceptance criteria, the analysis is terminated until the problem is found and corrected. If the CCV %Recovery is outside of acceptance criteria, the CCV is reinjected. If the % Recovery is still outside the acceptance criteria, all samples analyzed since the last acceptable CCV must be reanalyzed following correction of the problem.

12.3 LCS Failure: If the LCS is not recovered within acceptance criteria, the LCS is reinjected. If the % Recovery is still outside the acceptance criteria, either recalibrate and rerun or the associated batch and another LCS must be redigested (Section 10.1).

12.4 Matrix Spike / Post Digestion Spike Failure: If the recovery of the matrix spike is outside of the acceptance criteria, a post digestion spike is performed (Section 9.54). If the post digestion spike is outside of 75 – 125%, a narrative must be included with the data. (Section 10.1).

12.5 Duplicate Failure: If the RPD between the sample and its duplicate is greater than 20%, visually evaluate the sample matrix. If the matrix appears problematic, the sample digestate may be diluted and reanalyzed, or a narrative included with the data to explain the matrix problem.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

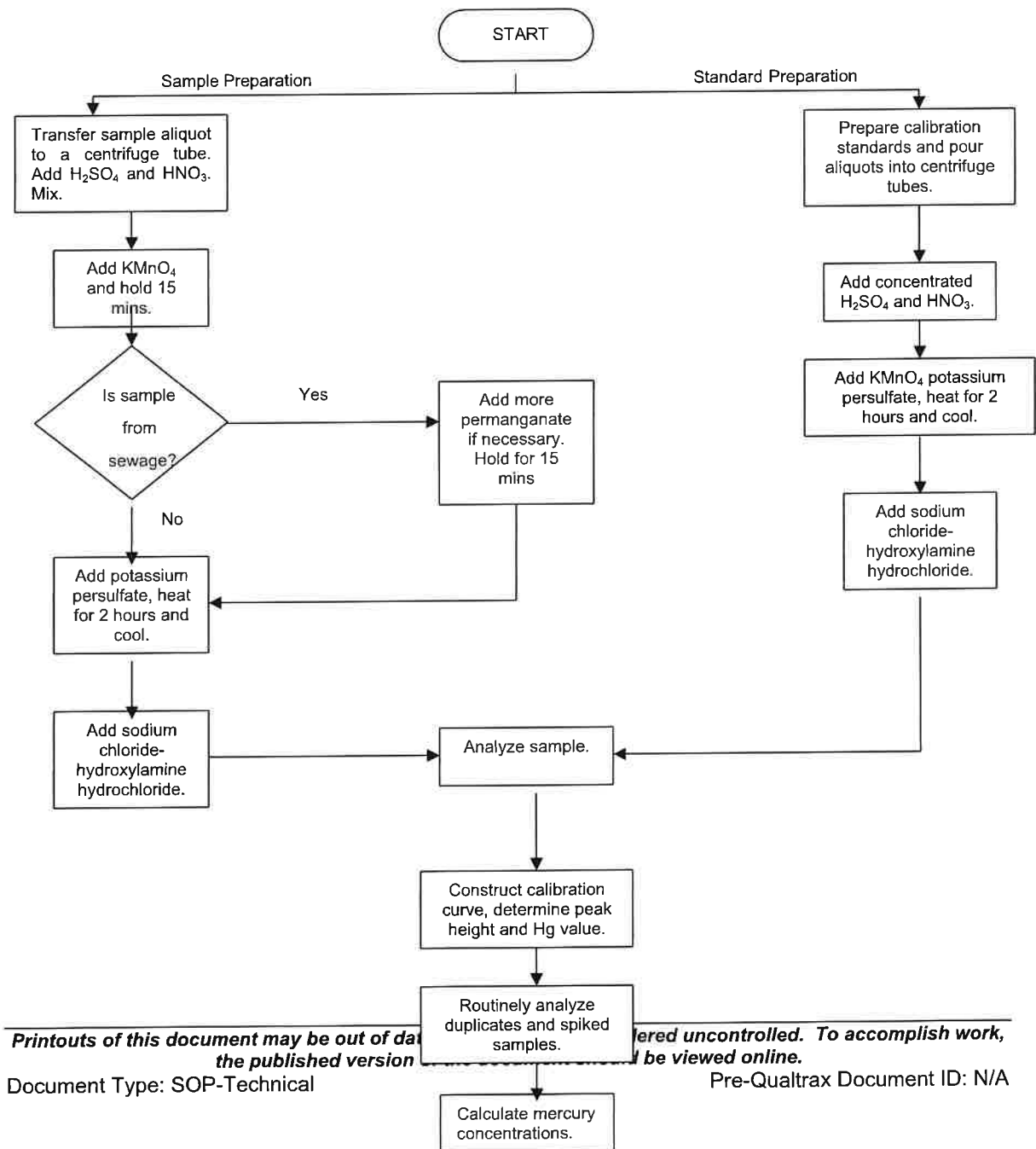
Chemical Hygiene Plan
SOP/1732 DL/LOD/LOQ Generation
SOP/1739 IDC/DOC Generation
SOP/1797 Waste Management and Disposal SOP

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16. Attachments

Figure 1: Method 7470A Flow Chart

Figure 1
Method 7470A Flow Chart



Alpha Analytical, Inc.
Facility: Mansfield, MA
Department: Metals Analysis
Title: Mercury in Liquid Waste (Automated Cold-Vapor Technique) EPA 7470

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Revision 2
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Page 13 of 13

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APPENDIX E

LaBella SOPs



Soil Identification and Description

Standard Operating Procedure



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<i>Revision</i>	<i>Effective Date</i>	<i>Prepared By</i>	<i>Description of Changes</i>	<i>Affected Pages</i>
0	2/21/2022	K. Truong	New Procedure	
1	2/24/2022	K. Truong	Formatting	All



1.0 INTRODUCTION

Soil description and identification is a subjective practice based on the individual performing the classification. The goal of this SOP is to establish a consistent method for soil classification despite each person's differing background and experience in soil characterization. This SOP describes LaBella's preferred method of soil classification to be used by field personnel: the modified Burmister system. The Burmister system is based on standardized nomenclature to define physical characteristics such as manual/visual description of soils, and includes nomenclature to describe the texture, color, mineralogy, and geological origin. Note that this SOP describes the procedure of identifying and describing soils and does not include soil logging during drilling (i.e., recording blow counts, etc.).

2.0 SUBSURFACE DEFINITIONS

There are different subsurface materials that are typically encountered during subsurface investigations; including but not limited to: bedrock/rock, soil, and historic fill. Although there are different classifications of bedrock/rock and soil based on geologic or engineering applications, the definitions outlined in this SOP will apply the engineering lens. A general definition of fill is used for this SOP, however the NYSDEC-10 Technical Guidance has its own definition

- **Bedrock:** Defined as relatively hard, naturally forming solid mass consisting of various minerals whose formation is from any number of physical and chemical processes. Rock is encountered in masses which require great efforts to break down into smaller particles. The physical properties of rock differ from soil and require different design and construction.
- **Fill:** non-native material, historically deposited in the general area by man. Fill materials can be contaminated or not depending on the materials deposited. The following are examples of some fill materials:
 - Gravel, sand, or topsoil backfill
 - Coal ash, wood ash, municipal solid waste incinerator ash, construction and demolition debris, dredge sediments, railroad ballast, refuse and land clearing debris
- **Soil:** used in this SOP is considered to be any unconsolidated natural material composed of solid particles with the pore spaces occupied by water, gas, or liquid. The term soil is not limited by depth below ground surface or the origin of the material. It may also include materials that can be classified as sediments.



3.0 METHOD SUMMARY

This SOP will include the procedures for:

- **Identification:** the process of determining which components are present in a soil sample, such as gravel, sand, silt, clay, etc.
- **Description:** the process of estimating the relative percentages of each component, when to use modifiers, and their definitions.

Soil identification and description is accomplished through primarily manual/visual means but supported by other field methods also covered in this SOP.

The main attributes of soils that should be identified and described are as follows:

- Soil type or lithology (sand, silt, clay)
- Color
- Soil relative density or consistency
- Moisture content
- Presence of Fill Material
- Odors/Staining/Impacts

Other attributes that should be included if observed are:

- Shape
- Sorting
- Structure

Soil logs should also include the method of sample collection, location, and depth.

3.1 Interferences and Potential Challenges

Due to the nature of field identification of soil, results may vary based on experience, weather conditions, and method of sample collection. Common challenges complicating soil classification include:

- When collecting samples via direct push or other methods that involve retrieving tooling from the borehole (i.e., not keeping the borehole “open”), “fall back” of overburden material into the borehole can occur. Such material might be re-collected as the borehole advances to greater depths and should be omitted from subsequent characterization, if identifiable.
- Accurate assessment of whether a soil is “native” can be difficult if the geological history of the Site is not known, or if limited recovery occurs.
- Differing sampling and drilling methods can also introduce some biases during the soil retrieval.

3.2 Equipment Needed

Equipment typically needed for soil classification includes:

- Pocket knife
- Soil boring log/field notebook
- Color Chart
- Grain Size Chart
- Tape measure



4.0 SOIL IDENTIFICATION AND DESCRIPTION PROCEDURES

Several subsurface investigations require soil logs which include soil identification and descriptions. In general, soil logs should note the following:

- Soil Boring Number/Test Pit Number/Surface Soil Number
- Location of soil boring/test pit/surface soil
- Total Depth of soil boring/test pit/surface soil

These notes can be logged in a soil classification log or field notebook. When looking at a soil boring/test pit/surface soil hole – note the depth of any clear changes in soil strata. Each strata should be identified and described using the following flow chart:



4.1 Identify Soil Name or Type

The bulk of the material should be noted with one of the following three main soil types and can be broken down into groups defined by sieve sizes:

- Granular (Non-plastic)
- Fine Grained (plastic or non-plastic)
- Organic

The difference between silt and clay cannot be determined visually, but a field test for plasticity can determine the soil type by doing the following:

- Roll a thread of the fine-grained soil (should be moist-wet)
- The smallest diameter that can be rolled determines the identity

Soil type	Soil Material	Size
Granular Soil Type	Gravel	Coarse Gravel: 3" to 3/4"
		Fine Gravel: No. 3/4" to No. 4
	Sand	Coarse Sand: No.4 to No. 10
		Medium Sand: No. 10 to No. 40 Fine Sand: No. 40 to No. 200
Soil type	Soil Material	Size of thread Rolled/Plasticity Index
Fine Grained	Silt	Cannot be rolled/0
	Clayey SILT	1/4" / 1 to 5
	SILT & CLAY	1/8" / 5 to 10
	CLAY & SILT	1/16" / 10 to 20
	Silty CLAY	1/32" / 20 to 40
	CLAY	1-64" / > 40
Soil type	Soil Material	Description
Organic Soil Type	Topsoil	- Typically, black/dark brown - Light weight, spongy, can contain roots, intact organic matter, found near the top of the deposit



4.2 Soil Density or Consistency Classification (only if SPTs are done)

Soil density or consistency is determined through Standard Penetration Resistance, can be described depending on the type of soil

- Resistance in granular soils (cohesionless) = relative density
- Resistance in cohesive soils = soil consistency

Standard Penetration Resistance can be determined in the field through the Standard Penetration Test (SPT) which requires a drill rig and typically used in Geotechnical work. See Tables 1 and Tables 2 for additional information.

4.3 Color Description

The color of the soil should be described with a primary color with a maximum limit of a 2-word description for tints or shades of the color

- Primary colors: Black, brown, gray, white, yellow, red
- Other terms that can be used to describe color are:
 - Mottled: Soil is marked with spots of color, normally associated with periodic wetting
- Ex. “Light-Brown”, “Olive-Brown”, “Dark Gray”
- Avoid lengthy combinations: “Reddish dark brown/gray”

4.4 Identification and Description of Major and Minor Soil Components

Major Constituents

- Always CAPITALIZED the major constituent
- For granular soil grain sizes, list the largest to smallest
- Ex. “medium to fine SAND” or “coarse GRAVEL”

Minor Constituents

- Should be listed in order of decreasing percentages, use the following table

Percentage	Label
0-10	Trace
11-20	Little
21-35	Some
36-50	And

- Only the first letter should be capitalized
- “With” can be used for unspecified amounts, (e.g. with organics or with possible cobbles)
- Ex. “some fine to medium Sand, little Silt”



4.5 Other Descriptions

- Moisture content
 - Important for showing groundwater

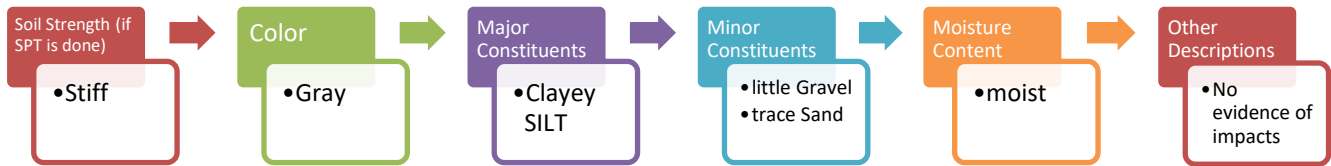
Dry	No moisture to touch
Dry to moist	Slight hand staining
Moist	Stains hands easily
Moist to wet	Stains hands, feels greasy
Wet	Free water in sample
Saturated	Water flows from sample

- Fill Materials
 - Note any fill materials (can be historic)
 - Brick, glass, ash/cinders, wood, etc.
 - Man-made materials have specific particle size descriptions (blocks=boulders, pieces=cobbles, fragments=gravel, particles=sand, specks=finer)
- Odors/Staining
 - Note any type of odors/staining/product or lack of
 - Can be organic or chemical
 - Petroleum odors/Chlorinated odors
 - Black staining
 - Product (oil, free product)
 - Can use short hand such as “NEI – no evidence of impacts”
- Shape
 - Describes the overall shape of the soil (can only be used for granular soil types)
 - Descriptions
 - Well rounded, rounded, sub-rounded, sub-angular, and angular
- Sorting
 - Describes the overall sorting of the soil (can only be used for granular soil types)
 - Descriptions
 - Poorly sorted, moderately sorted, well sorted
- Structure
 - Description of undisturbed soil samples can provide important information on their origin; see table 3 for descriptions



4.6 Naming Convention

Soil descriptions and identification should be constructed in the following order:



- Stiff, gray clayey SILT, little fine Gravel, trace coarse to fine Sand. Moist, no evidence of impact”
- Loose, brown coarse to fine SAND and coarse to fine Gravel, trace Silt. Moist, no evidence of impact.
- Short hand/refs can be used for field notes but should be fully typed out in report logs
 - SR to SA should typed out to “sub-rounded to sub-angular”
 - If the logger has the same description for soils in two different borings or at two different depths, field references can be used such as “see boring 1 or see S₁ (with S₁ noted at a certain description)”. While these are used in the field for reporting, logs should not include any short hand or references.

4.7 Description Tables

Table 1. Compactness of Cohesionless Soils	
Relative Density	SPT (blows per foot, bpf)
Very loose	0-4
Loose	5-10
Medium Dense	11-30
Dense	31-50
Very Dense	> 50

Table 2. Consistency of Cohesive Soils	
Consistency	Bpf
Very soft	0-2
Soft	3-4
Medium Stiff	5-8
Stiff	9-15
Very Stiff	16-30
Hard	>30

Table 3. Structure Descriptions	
Term	Definition
Homogeneous	Uniform properties throughout
Heterogeneous	Dissimilar properties
Stratified	Alternating layers of different types or color of soil
Laminated	Alternating layers less than 1/8 to 1/4 inch thick
Blocky	Easily broken into smaller angular lumps which are difficult to further break down
Lensed	Containing thin, discontinuous beds of different materials
Caliche	Secondary calcium carbonate forming a horizon that is typically very hard or well cemented



Subsurface Soil Sampling

Standard Operating Procedure



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1.0 INTRODUCTION

1.1 Applicability

The purpose of this Standard Operating Procedure (SOP) is to establish uniform procedures for the collection of subsurface soil samples via direct-push methods (i.e., Geoprobe® or similar drilling equipment). Adherence to this SOP will promote consistency in sampling methods and if properly followed will ensure sample representativeness.

1.2 Background

Direct-push sampling involves the hydraulic pushing and/or percussive hammering of a sampling tube into the subsurface. The inside of the sampling tube is generally lined with a sleeve or liner (typically made of acetate, stainless steel, plastic, Teflon, etc.), that encapsulates the soil during the samplers advancement. The sampler typically includes a cutting shoe, and may also include an internal locking piston (or similar device) that seals the sampling tube until it is unlocked at the top of a specific depth to facilitate the collection of soils from a discrete interval. The sampling tube is threaded onto direct-push rods. The rods and tooling are driven into, and subsequently pulled from the subsurface with the hydraulic/percussive direct-push equipment. The direct-push “drill rig” or “rig” may be mounted on wheels so that it can be manually moved by personnel. More typically, however, the rig is track-mounted, attached to a skid-steer, or on the back of a pick-up truck so that it can be easily moved from location to location across a site.

Direct-push sampling methods are generally applicable to unconsolidated soil/fill materials to a maximum recommended depth of approximately thirty (30) feet below ground surface (bgs). Soils may be obtained using this method for visual classification, field screening for contamination, as well as for physical and/or chemical analysis. Sampling is continuous throughout the length of the boring.

The ability to drive the sample tooling to a desired depth (as well as the ability to retrieve the sampling device from the subsurface) depends on the density and composition of the soil and the power of the hydraulic equipment. Additionally, sample recovery is somewhat dependent on grain size and density. Coarse gravel, cobbles, and boulders may plug a small diameter sample tube, preventing material from entering, or may cause refusal of the tooling altogether.

Soil types that might be encountered and background site information (accessibility, surface conditions, etc.) should be considered to decide whether direct-push methods are appropriate for a site, and to determine the specific tooling best suited for subsurface characterization.

It is noted that specific state and/or federal agencies may maintain specific guidelines and procedures that require deviation from this SOP. Such deviation should be identified prior to sampling (ideally during the work plan/sampling plan development) and should be explained in the project-specific work plan/sampling plan, when applicable.



2.0 RESPONSIBILITIES

2.1 Drilling Contractor

Direct-push drilling / sampling is an intrusive subsurface exploration method. By law, the clearance of underground utilities must be performed prior to the initiation of any intrusive subsurface activities. The drilling contractor performing the direct-push activities are responsible for notifying *Dig Safely New York* prior to initiating drilling / sampling activities.

Safety is of critical importance when working with and around hydraulics, drill rigs / drilling equipment, heavy machinery, etc. The drill contractor / crew must be aware of the safety requirements of working with and around such equipment. Prior to the start of a project, the drill crew should conduct a tailgate meeting / toolbox talk to ensure safe completion of activities.

The drilling contractor is responsible for providing the necessary equipment for obtaining subsurface soil samples. This generally includes the track-mounted, truck-mounted, or ATV-mounted Geoprobe® (or similar percussion/probing rig) and one or more sampling tubes (multiple diameters) in good operating condition, appropriate liners, and other necessary equipment for borehole preparation and sampling. Equipment decontamination materials should also be provided by the drilling contractor and should meet project specifications. Finally, materials for cleanup are required (i.e., sand, bentonite, asphalt cold-patch, brooms, etc.)

2.2 Project Manager

Typically, the Project Manager prepared the scope of work, work plan, and/or sampling plan (including the project proposal). The Project Manager must fully understand all elements of the applicable project documents and provide / communicate project-specific pertinent information to the Drilling Contractor and Project Geologist / Scientist (i.e., number and location of proposed sampling locations, analytical requirements, etc.).

The Project Manager is responsible for coordinating appropriate site access with applicable parties (property owner, tenant(s), client, etc.) and scheduling appropriate access and field activities with the Drilling Contractor and Project Geologist / Scientist. **The Project Manager should reconfirm the Drilling Contractor made the *Dig Safely New York* notification.** The Project Manager should also communicate specific safety concerns / requirements and Task Hazard Analysis (THA) forms.

Open and clear communication between the Project Manager, Drilling Contractor, Project Geologist / Scientist, and Client is a key component of the successful completion of direct-push / subsurface soil sampling projects. The Project Manager is responsible for maintaining these lines of communication.



2.3 Project Geologist / Scientist

The Project Geologist / Scientist is responsible for conducting subsurface soil sampling in a manner consistent with this SOP. The Project Geologist / Scientist will observe all sampling activities to ensure that the SOP is properly followed and record all pertinent data and information on appropriate forms, logs and/or in the project field notebook. Data recording may also include photo documentation.

It is the Project Geologist / Scientist's responsibility to review and understand the project work plan / sampling plan, and to communicate pertinent elements of the plan to the drilling contractor during activities. The Project Geologist / Scientist should be able to indicate the specific targeted sampling depth or sampling interval to the drilling contractor on-site. **The Project Geologist / Scientist should reconfirm the Drilling Contractor made the *Dig Safely New York* notification.** The Project Geologist / Scientist should also confirm the Drilling Contractor conducted an appropriate on-site tailgate meeting / toolbox talk prior to the initiation of work activities, and sign-off that such meeting occurred on the Contractor's form.

The Project Geologist / Scientist is also responsible for the collection of representative soil samples once the sampling device has been retrieved from the subsurface, disassembled, and liner removed. Additional sample collection responsibilities include labeling, handling, and storage of samples using standard chain-of-custody procedures.

3.0 EQUIPMENT/MATERIALS

In addition to the equipment and materials provided by the drilling contractor, materials to be furnished by LaBella field personnel (i.e., the Project Geologist / Scientist) typically include the following:

- Project-specific documents (proposal / scope of work, Health and Safety Plan (HASP), QAPP, Sampling Plan, etc.)
- Boring logs, field notebook
- Personal Protective Equipment (PPE) (as required by applicable HASP, Work Plan, Task Hazard Analysis Form, or Toolbox Talk)
 - Typical PPE required includes Hi-Visibility Safety Vest, Steel-Toe Boots, Safety Glasses, Hard Hat, Hearing Protection, work gloves, and Nitrile Gloves.
- Stainless steel spoons, collection / mixing pans, etc.
- Ziploc-type bags
- Sampling supplies (jars, labels, chain-of-custody records, tape, cooler)
- Ice (for sample preservation)
- Tape measure
- Field screening equipment (i.e., Photoionization Detector (PID), etc.)
- Phone / Camera
- GPS



4.0 PROCEDURES

4.1 General

Site-specific characteristics and project-specific requirements such as sampling depth will dictate the preferred type of sampling equipment to be used. In addition, the analytical program requirements will define the volume of sample needed, which will also influence the selection of the appropriate sampling equipment (i.e., sampling for semi-volatile organic compounds requires a larger soil volume and thus may require a wider diameter sample core than that necessary for volatile organic compound sampling via terracore). The project work plan / sampling plan should define specific requirements and equipment required for the given site. Sampling personnel should be equipped with a variety of sampling equipment to address deviations from anticipated sampling situations, including extra sample jars / containers in case of loss, damage, inadvertent contamination, or change in scope.

4.2 Equipment Decontamination

Sample tooling and components that may come in contact with soil must be decontaminated prior to their initial use and following the collection of each sample. Site specific decontamination might also be outlined in the sampling / work plan. If site-specific decontamination procedures are not stipulated, the procedures described in LaBella SOP – Equipment Decontamination, shall be followed. *Note: the level of decontamination will depend on whether soils are being sampled for laboratory analysis, field screening, or simply for visual classification.*

4.3 Typical Direct-Push Sampling Procedure

1. Don required PPE.
2. Decontaminate sample tooling and components that may encounter soil during sampling.
3. Drilling contractor / crew prepares the surface for direct-push sampling. Direct-push tooling can generally penetrate several inches of asphalt and/or crushed stone surface materials. If several inches of concrete are present at the location, core-drilling or another method of coring the concrete would be necessary to penetrate the surface pavement.
4. Drill contractor / crew assembles the sampling tube including the liner, discrete sample tooling (if appropriate), etc.
5. The direct-push rig operator will thread a push/drive cap on the top of the device and advance the sample tube into the ground.
6. The direct-push rig operator removes the push/drive cap, replaces it with a pull-cap, and pulls the sampler from the ground with the machine hydraulics.
7. The sample tube is then opened, to allow the soil-filled liner to be removed so that it can be cut open to allow for soil logging, field-screening, sampling for laboratory analysis, etc.
8. The sampling tube and components that contact soil during the sampling process are decontaminated, re-assembled, a new disposable liner inserted, and the process is repeated. The advancement of the sampling tube to depth is achieved through the addition of drive-rods, each of which is typically the same length as the sampling tube (commonly 3, 4, or 5 feet in length).
9. Upon completion of activities, the borehole is backfilled with soil cuttings, sand, and/or granular bentonite, or is completed as a piezometer or monitoring well.



4.4 Exposing Soils for Characterization and/or Sampling for Laboratory Analysis

Upon extraction of the liner from the direct-push sampling tube, the liner must be opened so as to expose the soils for visual classification/description, field screening, and/or sampling for laboratory analysis. This is preferably accomplished through the use of a liner cutting system, typically comprising a liner holder, and a liner cutter. The liner holder is a trough-like device that holds the liner securely in place so that it can be cut open.

The liner cutter is a tool affixed with two parallel hook-shaped blades that is drawn along the liner to cut a lengthwise opening in the liner for easy access and viewing of the sampled material. Liner cutters come in one-handle and two-handle varieties.

1. Place the soil-filled liner into the soil holder. Be sure that the liner holder is placed on a solid surface such as a sturdy work table, tailgate, etc.
2. Install the liner in the liner holder. Adjust the stop on the liner holder to secure the liner tightly in the holder.
3. Wearing leather work gloves, grasp the cutter by the handle(s) (avoid accidental contact with the blades) and place the cutter on the liner. The liner holder will usually have a bent bar that secures the liner in place, which provides resistance against the draw of the liner cutter. Begin the cut at the end of the liner opposite this bar. Be sure that blades are positioned just beyond the end of the liner to initiate the cut.
4. With slight downward pressure on the cutter, draw the cutter slowly and smoothly along the liner. If excessive force is required to open the liner, the cutter blades may be dull and should be replaced.
5. When the cutter has been drawn the entire length of the liner, the cut section of the liner may be removed to access the sampled material.

The equipment described above is standard practice for most drilling contractors and is recommended by this SOP. Alternate methods of cutting sample liners open (i.e., holding a liner with one hand and using a hook-blade utility knife with the other to open the liner) can result in severe cuts and nasty infections, and their use should be avoided whenever possible. If the use of a hook-blade is necessary, don cut-resistant work gloves and use exceptional caution by cutting away from you and others.

4.5 Screening and Sampling Soils for Environmental Laboratory Analysis

Target locations, depths, and/or intervals to be sampled are typically specified in the work plan or sampling plan, although they are sometimes subject to the findings of field screening/characterization and/or the discretion of the Project Geologist / Scientist. If the sampling program includes laboratory analysis for volatile organic compounds (VOCs), the VOC sampling shall be performed before any other activity (see *Volatile Organic Samples*, below).

Once the liner has been opened, the soils contained within can be sampled for laboratory analysis and classified. Materials from the liner can be removed using clean decontaminated/disposable spoons, etc. Except for soils to be sampled for volatile organic compound analysis (see below), the soils should be placed into a sample collection pan and homogenized or placed directly into the appropriate sample container(s). Note that samples for VOCs and PFAS are almost always to be collected as “grab” samples, while samples for other parameters (such as SVOCs, metals, etc.) may be collected as “grab” or “composite” samples. Grab samples are collected from a specific and discrete location, while Composite samples are generally collected from 3- to 5- locations and mixed into one sample jar(s).



Once filled, the sample container(s) should be properly capped, cleaned and labeled, and placed into a cooler with ice in preparation for delivery to the laboratory. Log the samples in field notebook, chain of custody and other required documentation. Handle samples for shipment to the laboratory in accordance with LaBella Sample Packaging, and Shipping.

If more soil is needed to meet sample volume requirements, additional soil cores may be collected from an immediately adjacent location. Decontaminate sampling tools prior to reuse.

4.5.1 Volatile Organic Samples

In order to minimize the loss of VOCs during the sampling process, VOC samples should be collected into laboratory-supplied glassware as soon as possible after retrieving the sampler from the subsurface. Other tasks (classification, sampling for other parameters, field-screening, equipment decontamination, etc.) should either be performed by others, or be completed after collecting samples for VOC analysis.

Upon filling the sample container, clean and label the container and place it into a cooler immediately. Residual sample may then be used to meet other sample quantity requirements.

When using direct-push methods for collecting soil samples for VOC analysis, the drilling contractor should not retrieve more than one subsequent sampler from the subsurface while the Project Geologist / Scientist collects samples from a previous interval.

4.5.2 Per- and Polyfluoroalkyl Substances (PFAS) Samples

Because PFAS can be present in a variety of common materials, the required detection limits are extraordinarily small (parts per trillion), and PFAS is considered an Emerging Contaminant (EC) by the NYSDEC, special sampling precautions are necessary when collecting soil samples for analysis of PFAS. A sample collected for PFAS analysis should be collected first (before collecting samples to be analyzed for other parameters), is required to be collected into specific laboratory-supplied bottleware, and should be collected according to the NYSDEC's *Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances Under NYSDEC's Pat 275 Remedial Programs – June 2021*, found online at:

https://www.dec.ny.gov/docs/remediation_hudson_pdf/pfassampanaly.pdf

4.6 Soil Classification

Soils should be visually classified using the Modified Burmeister Soil Classification System, unless alternate methods are required by project specifications. Refer to the Soil Identification and Description SOP.

4.7 Ground Surface Restoration at the Boring / Sampling Location

Upon completion of sampling activities, backfill the sampling / borehole location and restore the surface to as close to pre-sampling conditions as possible to eliminate surface hazards (i.e., trip hazards) or preferred path for contaminant migration (unless the borehole is intended to be outfitted with a groundwater monitoring well). The sampling / work plan may specify requirements for backfilling and surface restoration, and/or locations that require finishing as a groundwater monitoring well.



4.8 Field Screening Equipment / Procedures

Photoionization Detectors (PID)

When conducting soil sampling, the most commonly used field instrument is the PID. The PID allows for the rapid detection of VOCs while conducting work in the field or at any given site. Specific operating instructions for using and handling the PID are documented in the Owner's Manual that accompanies the instrument; however, it is useful to also be aware of the following items regarding proper use and handling of the PID for obtaining accurate field-screening data, and how to interpret the data collected from a PID:

- PIDs should be routinely calibrated per manufacturer's recommendations and the requirements of any project-specific work plans.
- In most outdoor environments, a properly calibrated PID will read 0.0 ppmV when on and reading the "open-air". If the PID is not reading 0.0 and you suspect it should be (i.e., that there are not any nearby sources of VOCs), this is an indication that the instrument requires calibration.
- Protect the PID from excess moisture. Moisture or high-humidity environments can damage the sensor and cause inaccurate readings.
- Whenever possible, use a "pre-filter" on the end of the PID's nozzle so that soil / debris does not enter and damage the inner components of the instrument. Keep the pre-filter clear of obstruction and replace as-needed to ensure accurate readings.
- Make sure that the PID is fully charged prior to bringing it to the project site. Not all batteries will last a full 8-hour shift, especially if they are not fully charged. The use of 4-AA alkaline battery packs with certain PIDs is appropriate as a temporary fix, but should not be relied upon for typical routine /everyday use.
- PIDs are expensive and should be handled with care. Do not leave PIDs unattended for extended periods of time, or in the vicinity of other contractors or activities that could result in damage to the instrument.
- When screening soil with a PID, there two primary methods of collecting data: "open air" and "headspace".
- Open air readings are collected by pointing the inlet nozzle of the PID <1" from the exposed soil targeted for screening. Point the PID at the soil immediately after exposing a 'fresh' surface (i.e., Dig into the soil and then use the PID. Do not use the PID on soil that has been exposed to the open air for an extended period of time as most VOCs will have already dissipated once exposed to the open air).
- "Headspace" readings are obtained by collecting freshly exposed soil directly into a sealed jar or Ziploc bag, then allowing the soil to be exposed to sunlight and heat to 'volatize' potential VOCs. The headspace reading is collected by inserting the PID nozzle into the jar or bag after an approximately 1 - 5 minute period of being allowed to volatize. *NOTE: soil used to collect a headspace reading should not be used for laboratory analysis. Collect a fresh soil sample for lab analysis of VOCs.*



5.0 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

Quality control requirements for direct-push / subsurface soil sample collection are dependent on project-specific sampling objectives which may be outlined in the site-specific Quality Assurance Project Plan (QAPP), if applicable, or may be included in the site-specific work / sampling plan. This information will include requirements for sample preservation and holding times, container types, sample packaging and shipment, as well as requirements for the collection of various quality assurance samples such as trip blanks, matrix spike/matrix spike duplicates, field blanks/equipment blanks, and field duplicates. The Project Manager is responsible for assuring that the QA/QC objectives are specified and communicated to individuals responsible for collecting the samples.

6.0 DOCUMENTATION

Documentation of sample collection, handling and shipping is required, and takes a variety of forms including:

- Field Log Book
- Soil Boring Logs
- Sample Collection Records
- Sample Container Labels
- Chain-of-Custody Forms
- Shipping Labels

The field log book will be maintained as an overall log of all samples collected during a project. Sample collection records are generated for each sample collected during a project and must include:

- Project Number and Location / Address
- Sampling point location / ID
- Date and time that sample was collected
- Name of collector
- Equipment used to collect the sample (when applicable)
- Number of sample containers, sizes, preservatives
- Specific Sample ID
- Depth
- Soil type (when applicable)
- Analysis Requested
- Shipping ID Number/Tracking ID Number (when applicable)

Soil boring logs provide visual and descriptive information for each sample collected and are often the most critical form of documentation generated during a direct-push / subsurface soil sampling program. The field log book is kept as a general log of activities and should not be used in place of the boring log. Occasionally, sample collection records are used to supplement boring logs, especially for environmental samples which have been collected for laboratory analysis.

Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes. These may be LaBella-specific or be provided by the laboratory providing analytical services for the project. Shipping labels are required if sample coolers are to be transported to the laboratory by a third-party (courier service). Original and/or copies of these documents must be retained in the appropriate project files.



7.0 TRAINING AND QUALIFICATIONS

Direct-push / subsurface soil sampling is a moderately complex task requiring some general training and experience that is usually earned by shadowing and assisting experienced field staff. Individuals conducting direct-push / subsurface soil sampling for the first time will be supervised/assisted by experienced personnel. Personnel collecting samples that might contain petroleum compounds, heavy metals, or other potentially hazardous materials will be trained and certified in accordance with the requirements of 29 CFR 1910.120 (OSHA's HAZWOPER standard).



Groundwater Sampling

Standard Operating Procedure



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1.0 GROUNDWATER SAMPLING

Groundwater sampling is typically conducted following monitoring well development and/or purging, depending on whether the well to be sampled is newly constructed or previously existing at the Site. Newly constructed monitoring wells are typically developed prior to purging and sampling. If there are existing wells at a Site that haven't been sampled in a long period of time, well development may be completed prior to purging and sampling, at the discretion of the Project Manager. The groundwater in newly installed monitoring wells will be allowed to stabilize for at least 24-hours following development, or as specified in the site-specific work plan prior to purging and sampling. Section 1.1 below describes well development activities.

Prior to initiating purging and sampling activities at each well, the static water level will be measured to the nearest 0.01 of a foot using a water level meter and recorded in the field notes. It is best to collect and record water level measurements from all wells on a Site on the same day to help generate accurate groundwater contouring data and avoid groundwater elevation fluctuation resulting from weather events and seasonal changes.

Groundwater sample collection is typically accomplished using either active or passive sampling techniques, as described in Section 1.2 and below:

1.1 Active Purging and Sampling:

Active purging and sampling includes the use of well pumping equipment and/or bailers to evacuate groundwater from the well by one of the following three (3) methods:

- Low Stress (low-flow) Purging and Sampling Procedure For the Collection of Groundwater Samples From Monitoring Wells (US EPA Region 1 EQASOP-GW4); this is typically conducted using a submersible bladder pump
- Modified low-flow purging and sampling by use of a peristaltic pump
- Purging and Sampling by Disposable Bailer (LDPE or HDPE)

1.2 Passive Sampling:

Passive sampling is typically conducted using the following equipment:

- Passive Diffusion Bag (PDB); purging is not required prior to sample collection



2.0 WELL DEVELOPMENT

Well development refers to the removal of fine-grained sediment that has settled out of solution inside a monitoring well casing during well installation, and to the extent possible, evacuating drilling fluids used to install the well (i.e. recirculation water used during bedrock coring or roller-bitting). Well development should be performed on newly installed monitoring wells and existing wells that haven't been purged or developed in a significant period of time, as specified in the Site-specific work plan.

Accumulated sediment that is not removed from inside a well can negatively influence groundwater sample analysis. Removing sediment and drilling fluids prior to purging and sampling helps ensure that the sample quality is most representative of groundwater aquifer conditions. If a newly installed well has been completed with grout, development should not occur until 24 hours after grouting has taken place.

Well development is typically accomplished using a pump, bailer, or surge block to remove accumulated sediments and to clean the pore spaces in the sand pack. It is generally not possible to over-develop a well. The more it is developed, the more representative of your sample will be. No dispersing agents, acids, disinfectants, or other additives will be used during development or introduced into the well at any time.

It is noted that if the well is to be sampled for PFAS, do not introduce any non-PFAS free equipment into the well at any time, including pumps, tubing, bailers, twine or water level meters.

General Procedure for Well Development

- 1) Regardless of what equipment is used for development, it should be lowered to the bottom of the well and surged up and down to help get sediment that has accumulated in the well into solution so that it can be evacuated from the well.
- 2) Aggressively surge the well for a few minutes and then evacuate the well using a pump (i.e. whale pump or other submersible pump designed to pump sediment) or bailer.
- 3) Development should continue until:
 - removal of 110% of the water lost during drilling is accomplished (i.e. water used during coring),
 - at least three (3) to five (5) well volumes are removed,
 - the hard PVC cap at the bottom of the well screen can be felt with the equipment being used for development and/or a water level meter,
 - or as specified in the Site-specific work plan or by the Project Manager.
- 4) The Site-specific work plan will indicate whether or not ground water quality parameters should be collected periodically during development. At a minimum, turbidity is typically measured, monitored and recorded during development.
 - Turbidity should decrease over time as the sediment is evacuated from the well.



- 5) If limited groundwater recharge does not allow for the recovery of:
 - o All drilling water lost in the well during installation or does not allow for evacuation of three (3) well to five (5) well volumes,
- 6) The well will be allowed to stabilize to conditions deemed representative of groundwater conditions, per the work plan or Project Manager. Stabilization periods will vary by Site and will often be discussed with NYSDEC prior to sampling, depending on the type of work being performed.
- 7) Development water will either be properly contained (i.e. 55-gallon drum(s)) and treated as waste until results of the chemical analysis of samples are obtained, or discharged on Site as determined by the Site-specific work plan and/or as directed by the Project Manager.

3.0 GROUNDWATER PURGING PRIOR TO SAMPLE COLLECTION

3.1 Active Purging and Sampling:

For active sampling methods including use of well pumps or bailers, monitoring wells are typically purged first to ensure stabilization of select groundwater quality parameters has been achieved prior to sample collection, as specified in the Site-specific work plan or as directed by the Project Manager.

Stabilization of water quality indicates the water being tested is representative of groundwater conditions at the well location. Prior to purging, the static water level in the well will be measured to the nearest 0.01 of a foot and recorded on the groundwater sampling log/field notes. There are different methods and equipment used to purge monitoring wells, each with their own advantages and disadvantages.

Equipment/Method	Advantages	Disadvantages
Bailers/Grab	- Inexpensive	-Time consuming/labor intensive - Transfer of water from bailer to sample jars can cause aeration and release VOCs - Requires complete removal of stagnant water in casing
Bladder Pump/Low Flow	- Presumes isolation of water from the screened well -Optimal for VOC sampling	- Careful measurements of pumping rate and drawdown -Rental fees can be costly
Peristaltic Pump/Low Flow	- Presumes isolation of water from the screened well -Fewer equipment -Optimal for VOC sampling	- Careful measurements of pumping rate and drawdown -Rental fees can be costly



3.1.1 Purging by Bladder Pump:

Bladder Pump Equipment:

- Bladder pump
- Bladders/Grab Plates (for each well)
- Twine (or cable)
- Compressor
- Battery (for compressor)
- YSI or Horiba Water Quality Meter (including turbidity)
- Water level meter
- Bucket to contain & measure volume of purge water removed
- Knife or cutting tool
- Tubing (typically 0.25-inch diameter; will need tubing for airline and water line; replace tubing between each well sampled)

General Procedure for Well Purging via Bladder Pump

- 1) When purging a well by use of a bladder pump, make sure of the following:
 - The bladder pump and any other equipment being introduced into the well (i.e. water level meter) have been properly **decontaminated**
 - New HDPE bladder and hoisting plate (aka grab plate) has been installed in the pump prior to lowering it into the well.
- 2) Given the depth of the well and depth to groundwater, the pump will be connected to the appropriate length of 0.25-inch diameter air and water tubing, and lowered into the well with twine or cable tied to the pump tether until the pump intake is positioned approximately at the midpoint of the screened interval.
 - Once the pump has been placed at the desired depth, secure the twine or cable so that the depth of the pump intake doesn't change.
 - Sometimes depth to groundwater (i.e. partially submerged screen) or other conditions (i.e. continuous drawdown during purging) will require the pump to be lowered to a depth greater than the midpoint of the screened interval for purging and sampling.
 - The depth of the pump intake should be recorded on the sampling log.
- 3) Once the pump is positioned in the well, the tubing should be connected to the airline attached to the compressor (activates bladder) and to the flow-through cell between the pump and the discharge point of the tubing so water quality parameters can be continuously monitored during purging.
- 4) The air compressor is then connected to the battery.
- 5) The water quality multi-meter (i.e. YSI, Horiba) connects to the flow through cell so it can continuously measure water quality parameters as the purge water passes through the flow through cell.
- 6) Place the discharge tubing from the flow through cell to a bucket to collect any discharge
- 7) Water quality parameters will be recorded at approximate 5 minute intervals until stabilization of parameters has been achieved and sampling can be completed.



- Water quality parameters should be measured from the flow-through cell, not from within the container (i.e. 5-gallon bucket) being used to capture the discharged purge water, since measurements from the bucket will not be representative of purge water conditions in the well at the time they are recorded.
- 8) The pumping rate of the bladder pump should be adjusted by the compressor during purging to produce the minimum drawdown possible, per the EPA method.
- To determine the flow rate of the pump, measure the amount of water collected over a set period of time (i.e. how much water is discharged into a container of known volume in one (1) minute).
 - Make sure to record the depth to water each time groundwater quality parameters are recorded so drawdown of the well can be frequently monitored and the flow rate of the pump can be adjusted as necessary to minimize drawdown.
- 9) At a minimum, the entire pump apparatus should be decontaminated with an alconox and water solution and rinsed with DI water, and the bladder and hoisting plate should be changed between each well sampling event.
- The twine or cable used to lower and raise the pump to its desired vertical position in the well should also be changed or decontaminated between each well sampled.
 - The flow-through cell, water quality instrument and water level meter should also be decontaminated between each well purged and sampled. Re-calibrate the water quality meter as necessary.

3.1.2 Purging by Peristaltic Pump:

Peristaltic Pump Equipment:

- Peristaltic pump
- String (or cable)
- Compressor
- Battery (for compressor)
- YSI or Horiba Water Quality Meter (including turbidity)
- Water level meter
- Flexi Tubing (need 3-inches per well)
- Bucket to contain & measure volume of purge water removed
- Knife or cutting tool
- Tubing (typically 0.25-inch diameter and surgical tubing in pump; replace tubing between each well sampled)

General Procedure for Well Purging via Peristaltic Pump

- 1) When purging a well by use of a peristaltic pump, make sure to use new tubing and decontaminate any equipment being introduced into the well (i.e. water level meter, flow-through cell, water quality meter) before lowering it into the well.
- 2) Given the depth of the well and depth to groundwater, the peristaltic pump tubing will be lowered into well until the intake end of the tubing is positioned approximately at the midpoint of the screened interval.
 - Sometimes depth to groundwater or other conditions (i.e. partially submerged screen, or continuous drawdown during purging) will require the tubing to be lowered to a depth greater than the midpoint of the screened interval for purging and sampling.
 - The depth of the intake tubing should be recorded on the sampling log.



- 3) The pumping rate of the peristaltic pump should be adjusted during purging to produce the minimum drawdown possible, per the EPA method.
 - To determine the flow rate of the pump, measure the amount of water collected over a set period of time (i.e. how much water is discharged into a container of known volume in one (1) minute).
 - Make sure to record the depth to water each time groundwater quality parameters are recorded so drawdown of the well can be frequently monitored and the flow rate of the pump can be adjusted as necessary to minimize drawdown.
- 4) All tubing used in the peristaltic pump should be replaced between each well purged and sampled.
 - The flow-through cell, water quality instrument and water level meter should also be decontaminated between each well purged and sampled. Re-calibrate the water quality meter as necessary.

3.1.3 Purging by disposable bailer:

Bailer Equipment:

- Types of bailers: LDPE for non-PFAS sampling; PVC/HDPE for PFAS sampling
- String/twine (PFAS-free if PFAS sampling)
- Water level meter (PFAS-free if PFAS sampling)

General Procedure for Well Purging via Bailer

- 1) Cut a length of string/twine to the appropriate length to allow the bailer to reach the bottom of the well, including the stickup length of the well casing, if applicable.
- 2) Attach the twine to the bailer and begin purging.
- 3) Discharge the purge water to a 5-gallon bucket (or similar container) so the water is containerized, and purge volumes can be measured.
- 4) Purge water will periodically (every +/- 5 minutes) be poured out of the bailer and into the container provided with the multi-meter so groundwater quality parameters can be measured, monitored for stabilization, and recorded.
- 5) Water quality parameters should not be measured from within the container (i.e. 5-gallon bucket) being used to capture the discharged purge water, since measurements from within the bucket will not be representative of purge water conditions in the well at the time they're recorded.

Purge water will typically be transferred from the 5-gallon bucket into a 55-gallon steel drum, as necessary during purging. The purge water will be treated as waste until results of the chemical analysis of groundwater samples are obtained, or discharged on Site as determined by the Site-specific work plan and/or as directed by the Project Manager.



3.2 Achieving Stabilization of Groundwater Quality Parameters:

As previously mentioned, groundwater quality parameter measurements should be recorded approximately every 5 minutes during purging. The tolerance for achieving stability of each groundwater quality parameter is listed on the low-flow sampling log. The goal for turbidity level prior to sample collection is <50 NTU (or lower for metals analysis). The lower the turbidity, the better.

Once all groundwater parameters achieve stability for three (3) consecutive readings, groundwater samples can be collected. If stability doesn't occur within the amount of time specified in the Site-specific work plan or within a reasonable amount of time, discuss the appropriate time for purging and sample collection with the Project Manager. Some wells stabilize fairly quickly but it is not uncommon for it to take 45 minutes to an hour to achieve stabilization of all parameters.

4.0 RECORD KEEPING OF PURGING AND SAMPLING DATA:

Purging and sampling information, including groundwater quality parameters that are typically measured, monitored and recorded during purging, is recorded on the Labella low-flow groundwater sampling log, and includes the following:

- Date
- Weather
- Well ID
- Static water level (including measurement point reference)
- Depth of well including measurement point reference (typically feet below top of PVC well casing)
- Well construction details (screen interval, total well depth)
- Pump type (i.e. bladder vs peristaltic pump, watera pump) and depth of pump intake
- Purge start time
- Pump rate (may be adjusted during purging)
- Gallons purged
- Temperature (°C)
- Dissolved oxygen (mg/L)
- Conductivity (mS/cm)
- pH
- Redox (mV)
- Turbidity (NTU)
- General observations (i.e. odor, changes in turbidity during purging, presence of NAPL and, if any, approximate or measured thickness)
- Purge end time
- Final static water level after purging
- Total water volume purged (typically recorded in gallons)
- Sample ID (including QC sample references if collected)



5.0 GROUNDWATER SAMPLING

5.1 Active Groundwater Sampling

As previously described, low-flow sample collection can commence once stabilization of groundwater quality parameters has been achieved through purging. Low-flow groundwater sampling is typically conducted using a bladder pump. There are times when “modified” low-flow sampling is conducted by use of a peristaltic pump. Consult the Site-specific work plan and/or Project Manager to determine which type of pump is best suited for your sampling job.

The following link provides the EPA sampling methodologies and procedures for low-flow sample collection:

<https://www.epa.gov/sites/default/files/2015-06/documents/lwflw2a.pdf>

5.1.1 Groundwater Sample Collection by Bladder Pump:

Once sufficient stabilization of groundwater quality parameters has been achieved and purging is complete, groundwater sample collection can be completed.

- 1) Prior to sample collection, disconnect the flow-through cell from the pump’s discharge tubing.
- 2) Collect the groundwater sample directly from the discharge tubing by filling the appropriate sample containers as specified in the Site-specific work plan.
- 3) At a minimum, the bladder and hoisting plate should be changed between each well sampling event, and the entire pump apparatus should be decontaminated with an alconox and water solution and rinsed with DI water.
- 4) The string used to lower and raise the pump and all tubing should be replaced between each well sampled.

5.1.2 Sample Collection by Peristaltic Pump:

Once sufficient stabilization of groundwater quality parameters has been achieved and purging is complete, groundwater sample collection can be completed.

- 1) Prior to sample collection, disconnect the flow-through cell from the pump’s discharge tubing.
- 2) Collect the groundwater sample directly from the discharge tubing by filling the appropriate sample containers as specified in the Site-specific work plan.
- 3) All tubing should be replaced between each well sampling event.
 - The flow-through cell, water quality instrument and water level meter should also be decontaminated between each well purged and sampled.
- 4) Re-calibrate the water quality meter as necessary.



5.1.3 Groundwater Sampling by Bailer

Once sufficient stabilization of groundwater quality parameters has been achieved and purging is complete, groundwater sample collection can be completed.

- 1) Pour the groundwater from the bailer directly into the appropriate sample containers as specified in the Site-specific work plan using the sample tip/port provided with the bailer.
- 2) New string/twine and a new bailer should be used for each well sampled.
 - Water quality instrument and water level meter should also be decontaminated between each well purged and sampled.
- 3) Re-calibrate the water quality meter as necessary.

5.2 Passive Groundwater Sampling

5.2.1 Passive Groundwater Sampling by Passive Diffusion Bag

Passive groundwater sampling methods are typically only used for collecting samples to be analyzed for Volatile Organic Compounds (VOCs) and do not require purging of the monitoring well prior to sample collection.

Passive sampling involves placement of a Passive Diffusion Bag (PDB) into a well where it is allowed to stabilize for a minimum of two (2) weeks after deployment before extracting the PDB from the well to collect the sample. PDBs operate by diffusion of contaminants across their polyethylene (LDPE) membrane and are typically pre-filled with laboratory grade (ASTM Type II) deionized water and sealed at both ends.

- 1) Each PDB is hung from a cable or rope, and positioned within the well screen interval until equilibrium has taken place between the water in the sampler and surrounding groundwater.
 - The PDBs also act as a filter, so field filtering is not required.
- 2) Once the PDB is deemed ready for sampling, it is retrieved from the well, cut open and the groundwater is poured into the appropriate sample container(s).
 - PDBs come in several different sizes and volumes.
 - Once the sample has been collected, the empty PDB should be properly disposed of.



Some reminders for passive sampling by PDB:

- Pre-filled PDBs will not be stored for longer than 30 days prior to deployment and will be kept stored at room temperature in a sealed plastic bag until ready to use.
- PDBs filled in the field will be used immediately and not stored for future use.
- PDB samplers will only be used to collect groundwater samples which will be analyzed for VOCs.
- Mesh covers will be utilized for open rock holes (so the PDB is not punctured or broken by abrasion) and will be secured to the bag using zip-ties.
- PDB samplers will be deployed by hanging in the well at the depth(s) specified in the project-specific work plan. The PDB samplers will be deployed at least 14 days prior to sampling.
- When transferring water from the PDB to sample containers, care will be taken to avoid agitating the sample, since agitation promotes the loss of volatile constituents;
- Gloves will be changed between collection of each PDB and tools used to open the PDB will be decontaminated with an alconox and potable water solution between each PDB;
- Any volume not used will be treated as investigation derived waste;
- Any observable physical characteristics of the groundwater (e.g., color, sheen, odor, turbidity) at the time of sampling will be recorded; and
- Weather conditions (i.e., air temperature, sky condition, recent heavy rainfall, drought conditions) at the time of sampling will be recorded.



Equipment Decontamination

Standard Operating Procedure



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1.0 INTRODUCTION

1.1 Applicability

The purpose of this SOP is to establish a uniform set of methods and procedures for decontaminating field sampling equipment. Decontamination is performed as a quality assurance measure and a safety precaution. The use of equipment that has not been properly decontaminated for collecting samples for chemical analysis can lead to erroneous data due to cross contamination. Decontamination protects field personnel from potential exposure to hazardous materials. Additionally, it prevents contamination from being transported off- site.

This SOP focuses on decontamination of non-disposable equipment used for sampling environmental media for chemical analysis. Decontamination of other materials (for example well-construction materials) are sometimes required and discussed in project-specific work plans.

It is noted that additional / other state or federal agency SOPs or requirements may exist that require deviation from this SOP. These required deviations should be identified before the sampling program begins and explained in the project-specific work / sampling plan.

1.2 Equipment/Materials

Materials for equipment decontamination typically include some or all of the following:

- Project-specific documents (proposal / scope of work, Health and Safety Plan (HASP), QAPP, Sampling Plan, etc.), as applicable
- Field notebook
- Personal Protective Equipment (PPE) (as required by applicable HASP, Work Plan, Task Hazard Analysis Form, or Toolbox Talk)
 - Typical PPE required could include disposable / washable clothing such as Tyvek, Steel-Toe Boots, Safety Glasses, Hard Hat, Hearing Protection, work gloves, and Nitrile Gloves.
- Tap / potable water
- Distilled and/or deionized water
- Phosphate-free detergent (Liquid-nox, Alconox, etc.)
- Solvents (such as dilute acids, methanol, hexane, isopropanol, etc., *only if defined by the work plan*)
- Paper towels
- Wash buckets / basins / containers
- Waste containers / trash bags
- Cleaning brushes / sponges
- Spray bottles, hoses, and/or pressure sprayers
- Plastic / poly sheeting
- Phone / Camera



2.0 PROCEDURE

2.1 Typical Equipment Decontamination Procedure

LaBella's standard equipment decontamination procedure is presented in the steps below. The procedure may be modified on a project-specific basis, as described in project specific documents (i.e., proposal, work / sampling plan, QAPP, etc.), and may include additional steps, solvents, materials, etc., depending on the quality assurance objectives for the project. Site and/or project specific documents should be referenced as appropriate.

- 1) Don PPE items appropriate to the characteristics of the contaminated material that was encountered (for example safety glasses, nitrile gloves, and disposable Tyvek garment).
- 2) Remove gross contamination, dirt, etc. from the equipment by physical methods (i.e., scraping, brushing, and/or rinsing with tap water). This step should be completed in a 5-gallon bucket or appropriately sized containment.
- 3) Wash the equipment with a phosphate-free detergent and tap water solution. This step should be completed in a separate wash bucket using brush, hose, sprayer, etc.
- 4) Rinse the equipment with potable water until all detergent has been removed. This step can be performed over an empty bucket using a squeeze bottle, hose, or pressure sprayer.
- 5) When required, triple-rinse the equipment with distilled or de-ionized water.
- 6) Allow the equipment to air dry on clean plastic sheeting. If faster drying is required, use paper towels to blot the equipment dry before reuse.
- 7) Containerize and/or manage wash water and decontamination rinseate in accordance with project-specific requirements.

When decontaminating submersible pumps used for groundwater sampling (or monitoring well development), the above-listed steps 2 and 3 may be conducted in a bucket, tube, or cylinder filled with the wash water, detergent solution, or rinse water. Turn on the pump at a low flow rate / setting for approximately five (5) minutes, allowing the wash solution to cycle through the pump's internal components. After the pump is removed from the potable water rinse cycle, the final rinse is performed with distilled/deionized water, being sure to flush through the internal components.

As previously stated, project-specific decontamination procedures may be required and will be specified in the project documents. Some project-specific modifications include the following:

- For glass and plastic sampling equipment used for sampling environmental media for metals analyses, decontamination may include a rinse with a 10% solution of nitric acid.
- For metallic sampling equipment used for sampling environmental media for metals analyses, decontamination may include a rinse with a 10% hydrochloric acid solution.
- For sampling equipment used for sampling environmental media for organic parameters (VOCs, SVOCs, pesticides, PCBs, etc.), decontamination may include an intermediate rinse with methanol, hexane, or isopropanol.



The above-listed solvents are usually hazardous materials due to their toxicity and/or corrosivity, and are specifically excluded from LaBella's standard decontamination procedure because of these properties. When the use of these (or other similar) solvents is required by project-specific documents, the project documents must also describe the additional protocols and procedures necessary for their safe use, handling, and disposal in accordance with federal, state and local requirements.

2.2 Large Equipment Decontamination

On some projects, heavy machinery and other large equipment (i.e., excavators, backhoes, truck-mounted drilling equipment, etc.) is used for sampling or site characterization activities, and may become contaminated during site activities (or may require decontamination prior to use on site). In these situations, the large equipment contractor should construct a temporary decontamination pad that typically consists of a bermed, plastic-sheet lined area where equipment and tooling can be staged for decontamination with a high-temperature high pressure washer and/or manual scrubbing. If heavy equipment decontamination is required on a project, the specifications for the decontamination pad and procedures for decontamination will be stipulated in the project documents.

2.3 Quality Assurance/Quality Control (QA/QC)

Quality control requirements for equipment decontamination are dependent on project-specific conditions and objectives typically outlined in the site-specific documents (proposal, sampling / work plan, Health and Safety Plan (HASp), and/or Quality Assurance Project Plan (QAPP)). The Project Manager is responsible for assuring that the QA/QC objectives are specified and communicated to individuals responsible for equipment decontamination.

Projects requiring specific equipment decontamination procedures usually require the collection of an equipment blank from the decontaminated equipment (typically at a rate of one per day; however the collection of equipment blanks and similar QA/QC samples is to be based on project documents that specify the type and frequency of collection of each type of quality assurance sample).

Equipment blanks are generally collected by pouring laboratory-supplied deionized water into, over, or through the freshly decontaminated sampling equipment and then transferring this water into a sample container. Equipment blanks should then be labeled as a sample and submitted to the laboratory to be analyzed for the same parameters as the associated environmental samples. Field blank sample numbers, as well as collection method, time and location should be recorded in the field notebook.



2.4 Documentation

Specific information regarding decontamination procedures should be recorded in the project notes and field notebook. Documentation should thoroughly describe the construction of each decontamination facility and the decontamination steps implemented in order to show compliance with the project documents. Decontamination events should be logged when they occur with the following information recorded:

- Date, time and location of the decontamination event
- What equipment was decontaminated
- Method(s) of decontamination
- Solvents used
- Other notable circumstances
- Date, time and location of equipment blank samples collected, and the methods / procedures used for collection
- Storage of decontamination wastes (spent wash and rinse water)

Repetitive decontamination of small items of equipment does not need to be logged each time the item is cleaned; however, a note should be made that such equipment was decontaminated as required and in accordance with this SOP, or project specific documents.

2.5 Training/Qualifications

Equipment decontamination is a relatively simple procedure generally requiring minimal training. Individuals conducting equipment decontamination for the first time will be supervised/trained by experienced personnel. Personnel exposed to sites / projects that might contain petroleum compounds, heavy metals, or other potentially hazardous materials will be trained and certified in accordance with the requirements of 29 CFR 1910.120 (OSHA's HAZWOPER standard).



Sample Packaging and Shipping

Standard Operating Procedure



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1.0 INTRODUCTION

1.1 Applicability

The purpose of this Standard Operating Procedure (SOP) is to establish uniform methods of handling, packaging, and shipping environmental samples. Adherence to this SOP will ensure that samples are received by the laboratory in good condition. This procedure will also prevent cross-contamination of samples during shipment and minimize sample container breakage, which can result in thousands of dollars lost to re-sampling efforts and project delays.

This SOP is to be used for environmental samples only. Hazardous material shipments shall adhere to USDOT requirements which are not presented in this document.

1.2 Equipment/Materials

Materials for environmental sample packaging and shipment typically include some or all of the following:

- Field notebook
- Chain of custody
- Custody seal
- Re-sealable Ziploc bags
- Bubble wrap
- Duct / packaging Tape
- Ice
- Personal Protective Equipment (PPE)
 - Includes but not limited to Nitrile Gloves
- Labels
 - Shipping Address Labels
 - 'Fragile' Labels
 - 'This Side Up' Labels

2.0 PROCEDURE

2.1 Sample Container Preparation

Each sample container (i.e., jar, bottle, vial, etc.) must be appropriately labeled with all identifying information (refer to the Sample Identification Nomenclature). Once the label is affixed, check that it is firmly adhered to the container. If there is any question about the label's 'stickiness', the label should be covered with clear packing tape which is wrapped completely around the container

If the project work / sampling plan or Quality Assurance Project Plan (QAPP) indicates that a custody seal on a container cap / lid is required, it must be placed across the container cap / lid prior to then placing clear packaging tape completely around the neck of the container.

2.2 Sample Container Packaging

Each jar, bottle, or set of VOA vials shall be placed in an appropriately sized resealable plastic bag. Care should be taken to ensure that air is removed from each bag. Properly bagging the samples protects against sample material release and cross-contamination should the sample container leak or break during shipment.

Bubble wrap should also be used to completely wrap the bagged sample container as necessary.



2.3 Cooler Inspection, Preparation, and Packaging

Each cooler to be used for sample shipment should be inspected for integrity. Lids / hinges should be inspected for their ability to create a tight seal. The walls, bottom and top of the cooler should be inspected for cracks / damage. Coolers with broken hinges and/or cracks shall not be used for sample shipment.

Each cooler shall be clean and free of any solid or liquid residue. If the cooler is equipped with a drain, duct tape shall be placed on the inside and outside of the drain to ensure that liquids cannot leak from the cooler.

Prior to the placement of any samples or ice into the cooler, it shall be lined with bubble wrap. A layer of bagged ice may be placed on the bottom of the cooler for samples requiring preservation by cooling.

Properly prepared / labeled sample containers must be placed upright in the cooler such that they are tightly arranged. If there are insufficient sample bottles to achieve a tight packing arrangement, then the samples shall be equally spaced throughout the cooler and the interstices filled with additional bubble wrap.

A layer of bagged ice shall be placed on top of the samples and bubble wrap shall also be laid over the top.

If the cooler is to be shipped via an overnight carrier (i.e. FedEx, UPS, or similar) the signed chain of custody shall be placed in a sealable plastic bag and taped to the underside of the cooler lid. Be sure retain a copy of the signed chain of custody prior to sealing the cooler!

2.4 Ice Bagging

Ice (consisting of commercially available cubed ice) shall be placed in sealable plastic bags to prevent ice melt from leaking into the cooler. The sealable plastic bags should be sized for the cooler to be used. A second resealable bag shall be placed over the first to provide a secondary containment layer. Care should be taken not to overfill the bags such that the bag is difficult to seal or at risk of rupturing during transit. A typical cooler will require four (4) gallons of ice; however, more ice may be used to fill gaps in the shipping container. Consideration for additional ice may also be prudent when shipping during the warmer months (April to November) or when using an overnight courier as coolers may spend time in transit in warmer climates and/or heated warehouses.

2.5 Cooler Packaging / Sealing and Labeling

The cooler must be able to be tightly closed and the lid secured using duct tape. Duct / packaging tape shall be placed along the entire perimeter of the lid where it meets the cooler body. The custody seal must be placed over the cooler body / lid joint and should be adhered with clear packaging tape.

“Fragile” and “This Side Up” labels may be used as appropriate. A “Fragile” label should be placed on the top / lid of the cooler. “This Side Up” labels shall have an arrow pointing upward. Clear packing tape should be placed over all labels.

An adhesive label shall be attached to the top of the cooler which has the destination information clearly shown on it. Clear packing tape shall be placed over the entire surface of the label. If shipping by FedEx, UPS, or similar, the printed airbill / tracking information shall be affixed to the top of the cooler.



3.0 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

Quality control requirements for sample handling, packaging, and shipping are dependent on project-specific conditions and objectives outlined in the site or project-specific documents (work plan and/or Quality Assurance Project Plan (QAPP)). The Project Manager is responsible for assuring that the QA/QC objectives are specified and communicated to individuals responsible for sample handling, packaging, and shipment.

Temperature blanks and Trip Blank samples are frequently used as QA/QC elements of sample handling and shipment, and should be included within the shipping container as specified by the project documents. Temperature blanks are typically provided by the laboratory supplying the cooler and should remain within the cooler at all times. Trip Blanks are provided by the laboratory supplying the cooler and should remain within the cooler at all times. Never open a Trip Blank. Make sure the Trip Blank is appropriately labeled and included on the chain of custody document.

Prior to shipment, the cooler should be inspected to ensure that it is undamaged, properly sealed, and appropriately labeled.

4.0 DOCUMENTATION

If samples are being shipped via courier or via direct delivery, then a copy of the signed chain of custody shall be retained.

If shipping via other carrier, make a copy of the chain of custody prior to sealing the container. The copy of the airbill / tracking receipt shall also be retained with the project records.

Forward any sample shipping documents to the Project Manager (via e-mail) for tracking purposes.

5.0 TRAINING & QUALIFICATIONS

Proper sample handling, packaging, and shipping is a relatively simple procedure generally requiring minimal training; however, it should not be taken for granted as lost or damaged samples / containers can incur thousands of dollars in re-sampling costs and project delays. Individuals shipping samples for the first time should be supervised/trained by experienced personnel. Anyone with questions regarding proper sample handling and shipment should contact a Project Manager.