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February 26, 2018 2011-2678-11

Mr. Peter Ramanauskas
United States Environmental Protection Agency – Region 5
Waste, Pesticides, Toxics Division
77 West Jackson Boulevard (DW-8J)
Chicago, IL 60604-3590

RE: Revised RFI Work Plan, Field Sampling Plan and QAPP
Exide Technologies, 555 Hoke Avenue, Frankfort, Indiana

Dear Peter:

On behalf of Exide Technologies, Advanced GeoServices presents the following responses to your comments (received via email on November 7, 2017) for the RFI Work Plan, Field Sampling and Analysis Plan (SAP), and QAPP that were submitted and originally dated October 12, 2017. Your comments are shown in **bold** below. Our responses follow in regular font.

A revised version of the RFI Work Plan, SAP and QAPP is provided for your review.

RFI WORK PLAN COMMENTS

Comment: 1. **Section 4.1.1 discusses the utility corridors and storm sewers. The text states that: "Exide will review its records to confirm the location(s) of stormwater inlets and piping onsite. Exide will also contact the City of Frankfort to obtain utility plans for the streets around the facility." Does Exide wish to include and sampling for stormwater sediments and water within this RFI Work Plan?**

Response: The revised RFIWP includes provisions in Section 4.1.2 to perform a video survey of those portions of the onsite stormwater piping that were not cleaned and plugged during the 2012 decommissioning. Locations of sediment accumulations, if any, will be documented and can be sampled as part of an addendum to this RIWP. Onsite inlets that are accessible during the proposed RI field work will be inspected and if sediments are present they will be sampled. All sediment samples will be analyzed for RCRA 8 metals. Section 3 of the SAP also incorporates requirements for potential sediment sampling.

Comment: 2. **Section 4.3.8, SWMU #8 – Propose adding one focused sample within the SWMU-8 footprint as was done for SWMU-6.**

Response: We have shifted the location of R-10 south by approximately 20 feet to address investigation in SWMU-8. R-10 is shown on Figure 3 and referenced in section 4.3.8.

Comment: 3. Section 4.3.10, AOC-1 – The text of this section states that "Soil samples were randomly collected at depths up to 5 feet to support determination of background lead and cadmium concentrations in soil. Lead concentrations ranged from 12 to 9,300 mg/kg, and cadmium concentrations ranged from 0.11 to 13 mg/kg." Were the samples exceeding screening levels all found within the surface samples? Were any elevated results found below the 1 foot excavation depth?

Response: The text in Section 4.3.10 has been revised to provide additional detail and a copy of the results from the 1988 AOC-1 sampling. As presented therein, the lead results for samples collected at depths below the 1 foot excavation depth ranged from 8.8 to 530 mg/kg.

Additional investigation is not proposed for this area.

Comment: 4. Section 4.4.2.2, AOC-3 (USTs) discusses 0 to 4 and 4 to 8 foot sampling intervals to be targeted to highest PID readings. Can Exide provide additional rationale for choosing those intervals and would the targeted interval then be a 6-inch subset of the 4 foot range?

Response: The 4-foot intervals were selected to coincide with typical geoprobe sample intervals. The sample collected would be from a targeted 6 inch interval with the highest PID measurements within the 4 foot range. Section 4.4.2.2 is revised to highlight this.

Comment: 5. Include the collection of surface soil samples in areas where visible runoff from site was observed during the September site visit (e.g. by the old rail spur, areas along fence line).

Response: An allocation for up to ten samples from five locations (0-6") and (6-12") with RCRA 8 metals analysis has been incorporated to the RFIWP. The exact locations will be determined in the field and documented in the RFI report. Section 4.4.2.3 has been revised to state this. Section 3 of the SAP also incorporates procedures for surface soil sampling.

Comment: 6. Section 10.4 of the Current Conditions Report states: "Additional upgradient background wells and offsite wells should be installed to determine the hydraulic gradient and contaminant isoconcentrations in order to identify the source of the chlorinated solvents." However,

in the RFI, no off-site wells are proposed. Please provide additional rationale for that. In order to determine water quality at the facility perimeter and provide a better site wide potentiometric surface map, additional wells in the southeast and northwest corner of the facility (near the former Filter Building/Baghouses) and along Hoke Avenue on the western site boundary would be beneficial. A southeast well may act as a checkpoint to see if the organic contamination in the vicinity of AOC-3 is flowing onto the site from an off-site source. Recommend moving proposed well MW-3 closer to AOC-3 and near the fenceline. Would MW-2 be better placed near the SWMU-8 area fenceline?

Response: Chlorinated compounds (TCE and degradation compounds) were identified in groundwater in the vicinity of UST-2, which is located along the western property. Three groundwater monitoring wells are proposed in the vicinity of UST-2. Based on a northerly groundwater flow direction, we are able to provide adequate coverage without the need to located wells off-site. If results of groundwater sampling determine that contaminant concentrations above the MCLs extend beyond proposed downgradient wells MW-2 and MW-3 or flow is in an easterly direction then additional wells may be required. Some of which may be situated off-site.

We agree with the assessment to incorporate a well in the southeast corner of the property. We also agree to incorporate a well along Hoke Avenue as requested by USEPA. The well network shown on Figure 4 and discussed in the RFI documents has been revised as follows:

- MW-1: no change proposed.
- MW-2: Shifted slightly southeast along property line to lie more directly north (assumed down-gradient) of SWMU-8.
- MW-3: shifted south to a location inside fence and closer to AOC-3.
- MW-4: no change proposed.
- MW-5: no change proposed.
- MW-6: well added in SE corner of property (near Kelly Avenue).
- MW-7: well added in NW corner of property (near Hoke Avenue, assumed downgradient of baghouses and filter building).
- MW-8: well added along Hoke Avenue toward center of property.

Comment: 7. **There appear to be inconsistencies between the text of Sections 2 and 4 and the Tables with respect to sampling depths and analyses. For example, Section 2.4 states that "During drilling, soil samples will be collected from the 0-4 foot bgs, 4-8 foot bgs, and 8-12 foot bgs intervals." while Table 1 identifies sample intervals down to 20 feet. Section 4.5 states monitoring wells will be installed to 15 feet while Table 1 identifies an 18 foot depth of boring with sampling intervals down to 20 feet and analyzed for TCL VOC/SVOC. Table 3 identifies VOC/SVOC/Metals testing of soils at MW boreholes, but Section 4.5.1 of the text does not.**

As an additional example, the proposed sampling identified in the text for AOC-2 does not match the analysis in Table 1 for samples F-7 to F-9. Please correct the text and tables to accurately reflect the proposed sampling and analysis. In order to get information on deeper geologic features, can Exide extend a well boring to deeper depth (e.g. 40 to 50 feet)?

Response: We have reviewed these Sections and Tables. Revisions have been made to address the inconsistencies. Well depth is expected to be 18 feet. Samples will be collected from each four foot interval to a depth of 16 feet bgs. A remaining sample will be collected from the 16-18 foot bgs interval.

Per new section 4.5.2, a VOC/SVOC sample will be collected from soils generated from the MW boreholes (split spoon) based on the depth of the highest PID reading. If no elevated PID readings are observed then the VOC/SVOC sample will be collected from the bottom of the well (see also SAP Section 3.1.4).

All previous sampling at the Site has indicated that lead/metals impacts are limited to shallow intervals (see 2014 soil boring data from CCR). This is expected given the immobility of lead and loamy soils in the region.

Some deeper impacts were observed during UST removal (depths approaching 12 feet). The proposed wells will achieve a depth of 18 feet. We do not believe that deeper borings/wells are warranted at this time.

Comment: 8. **Does Exide want to consider delineating extent of VOC/SVOC impacted soil during this mobilization since it is already known that such contamination is present at the site (i.e. include contingency for step-out sampling if you can stage the work such that you get analytical soil results back in time to do another round)?**

Response: Exide agrees to the concept of performing contingency step out sampling if warranted based on initial results. VOC/SVOC samples associated with the AOC-3 investigation can be performed as the first group of borings/samples so that results can be evaluated on an expedited basis. If data is received that indicate soil concentrations of VOC/SVOCs are present above IDEM non-residential direct contact standards, additional offset boring(s) can be performed. Sections 4.4.1 and 4.4.2.2 of the revised RFI work plan proposes a 2 day turn-around-time for the initial VOC/SVOC samples and one round of step out sampling.

Comment: 9. Table 1 footnote regarding all soil borings being screened using PID says that soil samples will be collected if <10 ppm above background. Should this be > 10 ppm?

Response: The table 1 footnote has been corrected to indicate sampling based on PID screening results greater than 10 ppm (> 10ppm).

FSAP COMMENTS

Comment: 1. Section 2.5.2 states that if a bladder pump isn't to be used, "peristaltic or electric pumps are acceptable alternatives." Please clarify 'electric pumps' as many such pumps may not be suitable for VOC sampling.

Response: This section is revised to "peristaltic pumps are acceptable". We do not think that the depth or purge rates will necessitate use of an electric pump.

Comment: 2. Section 2.5.8 mentions using passive sampling (Hydrasleeves) as an alternative. Are such passive samplers only appropriate for certain hydraulic conductivities when there is enough exchange between the well and the surrounding aquifer such that the contents in the Hydrasleeve will match the formation water quality?

Response: Hydrasleeves were mentioned as a contingency and are not currently proposed. This section is removed from the revised FSAP.

Comment: 3. Section 2.6.2 states equipment blank frequency is once per sampling event. The QAPP and Section 3 of the FSAP states once per day or per 20 samples, whichever is more frequent. Please make these two sections match (the QAPP specifies the correct equipment blank frequency).

Response: SAP has been revised to reflect equipment blank collection once per day or per 20 samples, whichever is more frequent. This is also summarized on the new table (SAP Table 4) identifying sample and analysis quantities.

Comment: 4. **Section 2.6.4/5 conflicts with the QAPP/Section 3 of the FSAP. The QAPP says one duplicate/MS/MSD per 20 samples per matrix, while the FSAP says two dupes/MS/MSDs per sampling event. The QAPP is correct. Please make these two sections match.**

Response: SAP has been revised to reflect that one field duplicate/MS/MSD will be collected per 20 samples per matrix. This is also summarized on the new table (SAP Table 4) identifying sample and analysis quantities.

QAPP Comments – review checklists specific for the QAPP were provided. The subject of the majority of the comments was providing more clear definitions for the personnel and roles of personnel that will be involved with the RFI. Advanced GeoServices communicated directly with the QAPP reviewer and has added the requested/required information to the QAPP.

Based on communication between Exide, Advanced GeoServices, and USEPA in December 2017 and January 2018, we believe that the comments in this letter and revisions made to the RFI Work Plan will be satisfactory. Upon approval from USEPA we will begin implementation of the RFI Work Plan.

If you have any questions please feel free to contact Jan Dobinsky at (610) 840-9136 or Paul Stratman at (610) 840-9122.

Sincerely,

ADVANCED GEOSERVICES CORP.

Jan S. Dobinsky
Associate Project Professional

Paul G. Stratman, P.E., P.G.
Senior Project Manager, Consultant

JSD:PGS:vm

Enclosure

cc: Brad Weaver, Exide

RCRA FACILITY INVESTIGATION (RFI) WORK PLAN
FOR THE
FORMER EXIDE MANUFACTURING FACILITY
Frankfort, Indiana

Prepared for:
EXIDE TECHNOLOGIES
Milton, Georgia

RCRA FACILITY INVESTIGATION (RFI) WORK PLAN
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Prepared for:

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Prepared by:

ADVANCED GEOSERVICES
West Chester, Pennsylvania

Project No. 2011-2678-11
February 26, 2018

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1.0 INTRODUCTION

1.1 PURPOSE

This RCRA Facility Investigation (RFI) Work Plan (WP) has been prepared by Advanced GeoServices Corp. (Advanced GeoServices), on behalf of Exide Technologies Inc. (Exide), to identify the activities that are required to perform the RFI at Exide's property located at 555 North Hoke Avenue in Frankfort, Indiana (the Site). The Site was formerly a battery manufacturing facility and during the operating period as well as after the operation ceased, various environmental investigations were performed at the Site. The Facility is located as shown on the attached Site Location Map (Figure 1).

Exide is the Respondent for a United States Environmental Protection Agency (USEPA) Administrative Order on Consent (Consent Order) under Section 3008(h) of the Solid Waste Disposal Act, commonly referred to as the Resource Conservation and Recovery Act of 1976 (RCRA), as amended by the Hazardous and Solid Waste Amendments of 1984, 42 USC 6928(h). The lead enforcement agency for the Site is the USEPA Region 5.

A summary of the available information for the Site was presented in the Current Conditions Report (CCR), which was also prepared by Advanced GeoServices for Exide and is dated July 6, 2017.

As required by Section VI, Item 11.b of the Consent Order, this RFIWP is being developed to identify the investigation process that will be used to close data gaps identified in the CCR and allow Exide and the USEPA to determine appropriate remedial measure, if any, for the Site, as well as additional investigation needs.

1.2 WORK PLAN ORGANIZATION

This RFIWP is organized into seven sections with supporting tables, figures, and appendices as follows:

- Section 1 Introduction
- Section 2 Background
- Section 3 RFI Chronology
- Section 4 RFI Objectives
- Section 5 Field Procedures
- Section 6 Quality Control/Quality Assurance
- Section 7 Reporting

2.0 BACKGROUND

2.1 FACILITY OWNERSHIP

Limited information is available regarding the exact nature of historic manufacturing operations at the Site. Based on information contained in the Consent Order and a November 28, 2011 Letter Report prepared by USEPA, Prest-O-Lite Manufacturing owned the Site during the World War II era. Prest-O-Lite was a car equipment manufacturer (including lead acid batteries), although this ownership history and specific Prest-O-Lite manufacturing operations conducted on-site could not be confirmed during preparation of this RFIWP. Based on Polk's City Directory in 1959 a telephone exchange registered to P.R. Mallory & Co. Inc. was listed for the address. P.R. Mallory & Co. manufactured electronics including dry cell batteries and eventually became Duracell. General Battery Corporation purchased the Site in 1963, which is consistent with additional information from Polk's City Directory which shows the telephone exchange for the address belonging to General Battery & Ceramic Co. Exide assumed ownership of the Site during the acquisition of General Battery Corporation in 1988. Exide currently owns the parcels that make up the Site.

2.2 FACILITY SETTING

The Site is located as shown on Figure 1 (based on USGS Topographic maps for Michigantown Quadrangle and Frankfort Quadrangle, Indiana-Clinton County 7.5 minute series). The Site is bounded by North Hoke Avenue to the west, Kelley Avenue to the east, Washington Street to the north (also referred to as Michigantown Road on some maps), and Norfolk Southern railroad tracks to the south. The Site is located in central Indiana within Clinton County, approximately 50 miles northwest of Indianapolis. Residential properties lie across the street from the Site on Hoke and Kelly Avenue; as well as on the opposite side of the railroad tracks to the south. Washington Avenue is immediately north of the Site and has several light industrial commercial properties located in proximity to the Site.

The Site consists of eighteen (18) contiguous/adjacent parcels owned by Exide which encompass approximately 13.7 acres (Figure 2). All but three of the parcels are located within a perimeter security fence. The majority of the area within the fence perimeter is paved (12.1 acres). The three parcels outside the fence are open grassy lots.

During operations approximately 4.5 acres of the Site were under roof and dedicated to battery manufacturing and other directly related operations (wastewater, emission control, offices, etc.). The remaining 7.6 acres of paved area inside the fence was utilized for parking of automobiles, trucks, and trailers. The Site buildings have been demolished and it is currently vacant.

Based on a review of historic aerial photos performed during the preparation of the CCR, the three (3) grassy parcels outside of the perimeter fence do not appear to have been used for any operational purpose by Exide. Historic facility plans dated from 1984 indicate that an alley separated the three grassy parcels from the manufacturing areas of the Site.

A topographic survey of the Site does not exist. The Site appears relatively flat with a typical ground surface elevation of approximately 850 feet MSL. However, a review of topographic maps for the area around the Site indicates that elevations are generally higher south of the Site and lower north of the Site. It has been observed that stormwater from the eastern half of the Site drains east and north to Kelley Avenue. Stormwater from the western half of the Site drains west to Hoke Avenue. Northern portions of the Site drain north to Michigantown Road.

Stormwater inlets are shown on Figure 3. Water which enters these inlets drains to stormwater pipes on Kelley Avenue or Michigantown Road. An unnamed tributary of Prairie Creek lies approximately 300 feet north of the Site; on the opposite side of Kelley Avenue. The unnamed tributary eventually drains to Prairie Creek approximately 1.5 miles downstream of the Site.

2.3 FACILITY OPERATIONS

2.3.1 Early History

Carriage manufacturing operations reportedly began on the Site in the early 1900s. Historic aerial photographs dating back to 1969 show many structures that were still present at the time of demolition in 2012. Prior to 1963, the Site was also reportedly used for cabinet manufacturing. It is possible that Prest-O-Lite, P.R. Mallory and cabinet manufacturing operations all occurred on the property between World War II and 1963.

2.3.2 Battery Manufacturing

In 1963, General Battery Corporation began the manufacture of lead-acid batteries for use in automotive, golf cart, marine and industrial applications. At its peak the facility produced over 12,000 automotive batteries per day. The Standard Industry Classification (SIC) code for the facility was 3691; "Battery manufacturing". The aerial photograph taken in 1969 shows a Facility layout that looks very similar in development to conditions observed in subsequent photographs.

The extent of paved areas is unclear in the 1977 aerial photo. The 1981 aerial photo included in the CCR clearly shows that the remaining portions of the Site, along Kelley Avenue have been paved. The New Formation portion of the facility was constructed in the late 1980s or early 1990s and is visible in the 1992 aerial photographs reviewed in the CCR. Some portions of the New Formation area may have been enclosed earlier in the 1980s.

During the battery manufacturing process, metallic lead was received at the facility, melted, and cast into grids and posts. Lead oxide paste was also manufactured at the facility and subsequently applied to the grids. Lead oxide was created by feeding molten lead into a reactor and mixing it with air to oxidize the lead. Pasted grids were placed in stacks that formed the core of the battery. The cores were placed in battery cases that were produced offsite. Then the remaining components were added. Electrolyte (dilute sulfuric acid) was then added to the

battery and a charge was applied (formation). After formation, the battery was cleaned, finished, labelled, and packaged for shipment to retailers and distributors.

Water was used to cool batteries after charging and also to wash batteries prior to shipment. Cooling water was collected in floor drains and emptied to a sump in the wastewater treatment plant (WWTP) located on the northwest corner of the Site. The sump collected wastewater from the entire plant, including sulfuric acid from the cooling and washing process, and lead from washing and dry charge operations. The corrosive wastewater (containing primarily sulfuric acid and lead) was pumped into two above ground holding tanks outside the building and then into reactor tanks located inside the WWTP building (SWMU-5).

Wastewater was treated with lime to neutralize the pH and precipitate dissolved metals. Following neutralization with lime, the wastewater flowed into a large clarifier outside. Precipitated solids settled to the bottom and was pumped to a sludge holding tank. From June 1986 until operations ceased the sludge was dewatered using a filter press. Sludge cake generated by the filter press was collected in a roll-off container for offsite disposal. Extracted water was recycled back to the clarifier. Prior to the use of the filter press, sludge was dewatered using vacuum pan filters and the filter cake was temporarily stored in an enclosure building prior to offsite disposal. Clarified water was then discharged to the City of Frankfort sanitary sewer system in accordance with a discharge permit.

Lead vapors were generated from molten lead handled in melting pots and casting machines. Plastic fumes were also generated from the battery case heat sealing process. Air handling equipment was used to vent these emissions to baghouses on the southeast side of the building. Lead oxide dust also was vented through ducting to a baghouse (SWMU-2). In general, air from the Facility was cycled through a filtering system called the OSI in the filter building (SWMU-6).

Battery manufacturing operations ceased in 1997. The Site was used by Exide for equipment storage until the demolition project in October 2012. The Site is currently vacant.

2.3.3 Decommissioning

In October 2012, Exide voluntarily performed a decontamination of the facility followed by demolition of the above grade structures. The decontamination and demolition project was completed in January 2013. The scope of work included:

- Interior pressure washing and vacuuming to remove dust;
- Removal of universal wastes (mercury bulbs and switches, PCB ballasts, miscellaneous chemicals, paints, and petroleum products, etc.);
- Dust suppression and air monitoring program;
- Demolition of above grade structures;
- Cleaning of pits, sumps, and pipes to remove sediment;
- Abandonment of piping/drains;
- Sampling and sorting of debris for disposal, recycling, or reuse;
- Crushing and onsite placement of approximately 3,300 CY of concrete rubble;
- Final washing of remaining impervious surfaces;
- Collection and onsite treatment of impacted wash water and other contact water;
- Demolition of select areas of corroded concrete floor and placement of impermeable cover; and,
- Installation of chain link fence as needed to secure the perimeter.

As part of the demolition work all utility supplies to the Site have been previously cut-off abandoned.

- Potable water – October 22, 2012;
- Electrical Service – September 25, 2012;
- Natural Gas – October 16, 2012; and,
- Sanitary Sewer – January 16, 2013.

2.3.4 Post-Decommissioning

Following the completion of decommissioning activities in early 2013 the Site has been largely inactive. In March 2014, twenty seven (27) soil borings were performed across the Site to obtain information on lead in shallow fill soils around the Site. Typically the borings were advanced to a depth of four feet below ground surface (bgs); however, at eight (8) location the borings were advanced to a depth of 8 feet bgs.

A 10,000 gallon underground storage tank (UST) used for the storage of diesel fuel and a 10,000 gallon UST used for the storage of #2 heating/fuel oil were removed in April 2014. Both tanks were closed through the IDEM LUST program and were granted NFA status on December 23, 2014. However, during the closure low level detections of chlorinated solvents and naphtha compounds were observed in surrounding soils and groundwater at concentrations exceeding IDEM RISC MTG and/or IDEM RISC residential tap water and vapor exposure screening levels. To date, no source for the chlorinated solvents has been identified onsite.

The Site currently is idle. During a meeting at the Site on September 12, 2017 representatives of Advanced GeoServices, Exide, USEPA, and IDEM observed that installation of a new gas main had been performed along Kelley Street. Markings indicating the presence of a 4" steel gas main on the west side of Kelley Street just several feet beyond the eastern Site property line/fence were observed. In addition, markings for water lines and storm drains were observed on the west side of Kelley Street near the northeast corner of the Site.

2.4 ENVIRONMENTAL SETTING

The Site is located as shown on Figure 1 (based on USGS Topographic maps for Michigantown Quadrangle and Frankfort Quadrangle, Indiana-Clinton County 7.5 minute series). The Site is located in central Indiana within Clinton County, approximately 50 miles northwest of Indianapolis. Central Indiana has a humid continental climate with cold winters and hot, wet summers. Measureable snowfall usually begins in late November and ends in late March. Spring is typically the wettest time of the year and is the peak time for tornados. May is

typically the wettest month with average rainfall between four and five inches across the State. Average annual precipitation in Clinton County is approximately 39 inches.

The Site and surrounding region is immediately underlain by Fincastle-Crosby soils. This is a silty loam with slow infiltration rates (Class C) and is somewhat poorly drained. The area is characterized by swell and swale topography. Fincastle soils are typically observed on rises and have a brown silt loam surface layer, and yellowish brown, mottled silty clay loam to clay loam subsoil. Crosby soils are found on high rises and have a brown silt loam surface layer, and yellowish brown, mottled silty clay loam, clay loam, and loam subsoil.

The Site appears to fall near the divide between Devonian and Silurian bedrock units. Devonian formations typically have a carbonaceous shale on the upper portion and are underlain by limestone, dolostone and shale. Silurian bedrock contains the latter rock types. Indiana bedrock geology features a broad anticline with a slight plunge to the northwest. Due to the broadness of the anticline it is often referred to as an arch. Bedrock beneath the Site is located at approximately 550 feet MSL (approximately 300 feet bgs).

The Tipton Complex Aquifer System is characterized by unconsolidated deposits that are quite variable in materials and thickness. Aquifers within the system range from thin to thick and include single or multiple intratill sands and gravels. The aquifers are highly variable in depth and lateral extent and are typically confined by thick clay layers. The total unconsolidated thickness of the Tipton Complex Aquifer System generally ranges from about 200 feet to over 400 feet in Clinton County. The potentiometric surface of the regional unconsolidated aquifer is approximately 800 feet MSL (approximately 50 feet bgs).

This system is capable of meeting the needs of domestic and most high-capacity users in the county. Aquifer layers utilized in the Tipton Complex Aquifer System are generally 5 to 10 feet thick sands and/or gravels. These sands and gravels are overlain by a till cap which is commonly 65 to 190 feet thick with thin intratill sand and gravel layers. Wells in this system are typically completed at depths ranging from 68 to 195 feet. Domestic well yields are commonly 15 to 65 gpm and static water levels are generally 15 to 35 feet below the surface. There are 8 registered

significant ground-water withdrawal facilities (29 wells) in this system in Clinton County. High-capacity well yields of up to 1,200 gpm are reported.

The Tipton Complex Aquifer System is generally not susceptible to contamination because it is typically overlain by thick clay deposits. However, in places surficial clay thickness is thin or not present. These are at moderate to high risk to contamination by surface sources.

3.0 RFI CHRONOLOGY

3.1 CONSENT ORDER

The Administrative Order on Consent (Consent Order) entered into by Exide and the United States Environmental Protection Agency (USEPA Region 5) was executed/effective on May 9, 2017. Section VI, Item 11 of the Consent Order requires several procedural steps to be completed by Exide as the Respondent. This includes the Current Conditions Report, RCRA Facility Investigation (RFI) and RFI Report.

The Current Conditions Report is summarized in Section 3.2 of this document. The RFI is the subject of this Work Plan document, and the RFI Report is discussed in Section 7.0 of this document.

3.2 CURRENT CONDITIONS REPORT

The July 6, 2017 CCR (Advanced GeoServices) reviewed the Site conditions and setting, regulatory and operating history, and existing data for the Site. The CCR listed nine (9) solid waste management units (SWMUs) at the Site, as well as, three (3) additional areas of concern (AOCs):

- SWMU-1 Former Waste Pile #1 (Sludge Storage Area)
- SWMU-2 Sludge Storage Tank
- SWMU-3 Baghouses
- SWMU-4 Hazardous Waste Accumulation Area
- SMWU-5 Wastewater Treatment Building and Sump
- SWMU-6 Filter Building
- SWMU-7 Roll-off Container
- SWMU-8 Former Waste Pile #2
- SWMU-9 Parts Cleaners

- AOC-1 Loading Dock Area
- AOC-2 Castings/Grid Building Area
- AOC-3 Underground Petroleum Storage Tanks

The CCR identified the following contaminants of concern (COCs) for the Site or within specific SWMUs/AOCs:

- Site-wide: Lead, Arsenic, Cadmium
- USTs/AOC-3: Naphthalene/1-methylnaphthalene/2-methylnaphthalene
- USTs/AOC-3: Chlorinated Solvents – tetrachloroethylene (PCE), trichloroethylene (TCE), 1,1-dichloroethane (1,1-DCA), 1,1-dichloroethane (1,1-DCE), 1,2-dichloroethane (1,2-DCE), vinyl chloride (VC)

For purposes of the conceptual site model presented in the CCR the Site was divided into four areas of interest (AOIs):

- Former manufacturing areas – this includes the majority of the SWMUs and AOCs;
- Underground Storage Tanks;
- Overall Site Soils – this includes soils below the currently paved areas on the east side of the property, the parcels on the east side of the overall property that formerly contained houses, and the grassy portions on the north side of the Site that are not know to have been used for facility operations; and,
- Groundwater – the primary aquifer is believed to be very deep beneath the Site. However, a shallow/perched groundwater interval was encountered during previous UST removal and closure investigation activities at depths less than 12 feet bgs.

The CCR and review comments provided by USEPA identified the following data gaps/needs:

- Site-wide sampling to characterize shallow fill soils for lead, arsenic, and cadmium;
- Focused sampling around select former manufacturing area AOCs;
    - SWMU-2 Sludge Storage Tank
    - SWMU-3 Baghouses
    - SMWU-5 Wastewater Treatment Building and Sump
    - SWMU-9 Parts Cleaners
- UST-related soils (AOC-3); and,
- Groundwater potentiometric surface and quality.

The information that was compiled in the CCR and used to develop this RFIWP will be referred to as needed herein and during the performance of the RFI work. Information from the CCR will also be incorporated into the RFI Report where appropriate to enhance the understanding of the Site, subsurface conditions, and potential exposure pathways.

4.0 RFI OBJECTIVES

The overall purpose of the RFI is to fill the existing data gaps as necessary to draw a conclusion regarding the need for corrective measures onsite. The areas to be investigated include the shallow overburden/fill soils on a site wide basis, focused soil investigation around specific SWMUs/AOCs, and groundwater on the Site (focused on the vicinity of the USTs).

Additional background information will be gathered using available historic mapping and information presented in the CCR, site surveying, utility mapping, and local water use evaluation.

Proposed locations of monitoring wells and soil borings are shown on Figure 3. Field procedures are discussed in the following sections of this document. Task specific data quality objectives are presented in the Sampling and Analysis Plan (SAP) in Appendix A and the Quality Assurance Project Plan (QAPP) in Appendix B.

4.1 SITE RECONNAISANCE

As part of the RFI a detailed site reconnaissance effort will be initiated. This will include evaluation/confirmation of utilities on and around the Site, topographic surveying, and a review of features on and adjacent to the Site.

4.1.1 Utility Corridors

To date only limited information has been obtained regarding the location of subsurface utilities onsite. Although there are not believed to be any active energized utilities onsite; a storm sewer system and associated inlets is present. Several utilities have been observed within several feet of the property line along Kelly Street and Hoke Avenue. The locations of utilities on and immediately offsite is important information for avoiding conflict or damage during the RFI field work.

Storm drains may have collected surficial contaminants that were washed into the onsite inlets during storm events. As shown on Figure 5, the onsite inlets and drain piping appears to have a stormwater system on the eastern side of the property that drains to the municipal storm sewer system near the intersection of Hoke Avenue and Washington Avenue (northwest corner of Site). A second stormwater system runs from the central portion of the Site to the northeast corner before connecting to the municipal storm sewer system on Kelly Avenue. Presumably the stormwater ultimately drains northward into the unnamed tributary north of the Site.

The stone bedding around utilities can also function as a preferential path for shallow/perched groundwater flow during storm events. During the UST removal, it was observed that the majority of the stormwater entering the excavations adjacent to Kelly Street was coming from the direction of Kelley Street.

During a site walk on September 12, 2017 a utility mark out for a 4" steel gas main was observed within several feet of the property line on the west side of Kelley Avenue. New gas service connections were reportedly being made to homes on the east side of Kelley Street. Water main and storm sewer markings were observed on the west side of Kelley Street to the north of the Site.

Exide will review its records to confirm the location(s) of stormwater inlets and piping onsite. Exide will also contact the City of Frankfort to obtain utility plans for the streets around the facility.

4.1.2 Storm Sewer Investigation

As part of the field reconnaissance work, Exide will perform a video investigation of the onsite storm sewers that are identified. If significant accumulations of sediment are identified in accessible locations, Exide will attempt to sample the sediment (RCRA 8 metals analysis). If sediment accumulations are not accessible, they will be documented for potential future sampling. If sediment/debris accumulations impede the video investigation the piping may be hydro jetted to remove the obstructions. Waste/sediments that are removed by hydro jetting will

be containerized onsite and sampled for RCRA metals and/or as required to characterize those materials for disposal.

4.2 SITE SURVEY

Detailed topographic information is not known to exist for the Site. As part of the RFI, Exide will perform a survey of the Site. The survey will be performed by a licensed Indiana Professional Surveyor.

In general, a topographic survey will be performed to establish topographic conditions onsite on a 50 foot grid spacing and to identify obvious high and low points onsite. Building slab perimeters and elevations of raised slabs above existing grades will also be included. The alignment of the security fence and any utility poles, stormwater inlets, or other relevant site features will also be shown on the survey. The topographic survey will also include the streets adjacent to the Site to a distance of approximately 100 feet in any direction from the property lines.

The surveyor will also perform a stake out of proposed well and boring locations (including measurement of existing ground surface at those locations). The survey will also show the locations of any right-of-ways (railroad, street, etc.) and easements (utilities, etc.) that may exist on the Exide properties.

Current facility drawings are not oriented on a state plane coordinate system. As a result the proposed locations of soil borings and monitoring wells in this RFIWP cannot be assigned coordinates at this time. Pending completion of the survey, the proper coordinate identifications can be provided.

4.3 SWMU/AOC STATUS

This section summarizes the conclusions of the CCR as it pertains to investigation needs in the various SWMUs/AOCs on the Site. The majority of the SWMUs were designated during a

Preliminary Report/Visual Site Inspection (PR/VSI) issued by IDEM in 1988. More detailed information is provided in the CCR.

4.3.1 SWMU-1 Former Waste Pile #1 (Sludge Storage Area)

The waste pile underwent closure in accordance with an Indiana State Board of Health (ISBH)-approved closure plan in 1986. No history of any releases from this waste pile has been identified because the waste pile was located indoors in an enclosed area on a concrete floor. Leachate generated during the temporary accumulation period was collected and transferred to the wastewater treatment system for processing.

According to the approved closure plan, sludge remaining in the waste pile at the time of closure was placed in a roll-off container and transported to the Adams Center Landfill in Fort Wayne, Indiana. The concrete walls and floors of the sludge storage area and adjacent areas were washed to remove contaminants. The building was subsequently modified to enable sludge to drop directly from dewatering equipment into a large roll-off container. Exide submitted closure certifications to ISBH in June and July 1986. ISBH issued a completion of closure letter to the facility in November 1986.

No additional investigation is necessary for SWMU-1.

4.3.2 SWMU-2 Sludge Storage Tank

The sludge storage tank was located inside the WWTP building on concrete slab. The tank measured approximately 12 feet in diameter and 12 feet tall (maximum capacity 10,000 gallons) and was constructed of fiberglass. Spilled material would have been contained by the building. No history of any releases has been identified with the sludge storage tank.

The WWTP facility including the sludge storage tank was decontaminated and demolished/removed as part of the 2012 decommissioning activities. No sludge remained in the sludge storage tank and no evidence of a release was observed at that time.

In 2014, soil boring B-13 was performed in the vicinity of the former WWTP. Refusal was encountered at a depth of 2-feet bgs. However, the 1.5 to 2 feet deep sample interval produced a lead detection of 13,200 mg/kg. Additional borings are performed to further investigate inorganic contaminants in this area as well as around the WWTP (SWMU-5).

4.3.3 SWMU-3 Baghouses

The baghouses were located outside behind the plant building (north end). When the facility was used to manufacture batteries, lead fumes from melted lead and lead oxide dusts were vented to the baghouses. Dust that accumulated in the baghouses was classified as D008 hazardous waste due to its lead content. The PR/VSI described the ground beneath the baghouses as appearing dark, indicating possible contamination from lead dust. The VSI recommended that soil samples be collected from several depths beneath all baghouses and analyzed for lead. It is reported that use of the baghouses ended in 1997, when the facility ceased manufacturing batteries.

A CEI conducted by IDEM in June 2001 indicated that cleanup of the baghouses had been completed, and that Heritage Environmental Services had removed and disposed of the waste generated from the cleanup activities. It is not known whether any post-cleanup samples were collected from the area beneath or around the baghouses. The remaining baghouse structures were removed from the Facility as part of the 2012 decommissioning.

In 2014, soil boring B-18 was performed in the vicinity of the former baghouse and filter building. B-18 was advanced to a depth of 8-feet bgs and nine samples were collected from various intervals. The maximum lead detection of 510 mg/kg occurred in the 0.5 – 1 foot depth interval. No other intervals had lead detections which exceeded an IDEM RISC residential screening level.

As part of the RFI, additional soil investigation will be performed to evaluate inorganic contaminants around the location of the former baghouses.

4.3.4 SWMU-4 Hazardous Waste Accumulation Area

Hazardous waste drums were accumulated on a concrete floor inside the plant building. There are no documented releases from this unit, but spilled material would likely have been contained by the building. No violations associated with this unit were noted during the June 2001 and June 2010 Compliance Evaluation Inspection (CEIs) performed by IDEM. No hazardous waste was being accumulated in the hazardous waste accumulation area at the time of the June 2010 CEI.

No additional investigation is needed for this SWMU.

4.3.5 SWMU-5 Wastewater Treatment Unit and Sump

The wastewater treatment facility was installed in 1970 and was located in the northwestern corner of the property. Battery manufacturing operations generated approximately 35,000 gallons of wastewater containing dilute sulfuric acid and lead (D002 and/or D008 waste) per day. Process wastewater was collected in a series of floor drains in the plant buildings and piped to the on-site treatment facility. At the treatment facility, wastewater was initially collected in a sump and pumped to one of two aboveground holding tanks for equalization. The holding tanks were located outside the treatment facility, and each tank held approximately 6,350 gallons of wastewater. From the holding tanks, wastewater was pumped into a three stage reaction tank where lime was added for pH neutralization and precipitation of lead. The reaction tank had a design capacity of 48,000 gallons.

In April 1984, an 865,700-gallon clarification tank located outside the wastewater treatment building was placed into operation and was used in the treatment scheme for solids and liquids separation. Solids consisted primarily of calcium sulfate from the lime neutralization process that had settled to the bottom of the clarification tank. Treated and clarified liquid from the clarification tank was discharged under a permit to the city sewer system. Semi-solid sludge at the bottom of the clarifier was withdrawn and pumped to a sludge tank (SWMU 2) for temporary holding prior to dewatering in a filter press. Filtrate from the sludge dewatering operations was

collected and pumped back into the reaction tank for subsequent treatment. Dewatered sludge from the filter press was accumulated in a roll-off container (SWMU 6) prior to off-site transport to Adams Center Landfill for disposal. Prior to June 1986, the sludge was dewatered in a vacuum pan filter and dewatered sludge was accumulated in a waste pile (SWMU 1) in an enclosed building before being transported off site for disposal.

The aboveground wastewater treatment holding tanks were located outside on a concrete pad. The system's sump and reactor tanks were located inside the facility. There are no documented releases from either the sump or the wastewater treatment system itself. It is expected that a significant release of hazardous sludge or wastewater from this unit would have been noted in the historical file material, as it likely would have interrupted process operations at the facility.

The WWTP and sump were decontaminated and removed as part of the 2012 decommissioning activities. The areas below the sumps and clarifier were backfilled using crushed concrete rubble from the facility demolition. No evidence of a release was observed. In 2014, soil boring B-13 was performed in the vicinity of the former WWTP. Refusal was encountered at a depth of 2-feet bgs. However, the 1.5 to 2 feet deep sample interval produced a lead detection of 13,200 mg/kg.

Additional investigation of the former WWTP area (and the sludge storage tank SWMU-2) for inorganic contaminants is included in this RFIWP.

4.3.6 SWMU-6 Filter Building

This unit functioned like a baghouse during the plant's manufacturing operations. Air from inside the plant building was drawn into the filter building where dust was caught in a system of filters. The cleaner air was then recycled back into the plant. The filters were cleaned or replaced as necessary. Old filters were treated as hazardous waste (D008), accumulated in the hazardous waste accumulation area and sent to a secondary lead smelter in Pennsylvania for recycling. Lead-contaminated dust (D008 waste) from the filters was collected in 55-gallon

drums that were also accumulated in the hazardous waste accumulation area for less than 90 days and transported to a secondary lead smelter in Pennsylvania for recycling.

The filter system was totally enclosed and there are no documented releases from the unit. Old filters were shrink-wrapped in plastic before being placed on pallets to prevent residual dust from falling off the used filters. It is assumed that this unit was regulated by the IDEM operating permit (#16313) which governed air emissions and was closed when facility operations ceased. The Filter Building was decontaminated and demolished during the 2012 decommissioning project. Masonry block from the building was crushed and reused onsite as fill material.

One focused sample location (F-6) is proposed for this area.

4.3.7 SWMU-7 Roll-Off Container

This unit was a roll-off container located indoors beneath the filter press. This unit was used for less than 90-day accumulation of dewatered wastewater treatment sludge carrying the D008 hazardous waste code until that waste was transported off site for disposal. It is estimated that use of the SWMU-7 roll-off container for collection of dewatered sludge ended in 1997, when battery manufacturing operations ceased at the Exide facility.

This unit was located inside the plant building on a concrete floor. The PR/VSI indicated that the facility was practicing good housekeeping and no concerns with this unit were identified in compliance inspections conducted at the facility. As a result, environmental releases are unlikely. Accordingly, there are no documented releases from this unit. The roll-off container was removed from the Facility prior to the 2012 decommissioning project.

No specific additional investigation of this SWMU is included in the RFI, although it is located in the same general area as SWMU-2 and SWMU-5, for which additional investigation is proposed.

4.3.8 SWMU-8 Former Waste Pile #2

This unit was a 30-foot by 30-foot waste pile formerly located in the northeast potion of the facility. The waste pile contained lead-contaminated soil that was reportedly excavated during the course of a remodeling project conducted at the facility in 1996. Approximately 123 cubic yards of lead-contaminated soil were placed directly on the ground surface at this unit. All wastes managed in the waste pile were removed from the area and disposed at a permitted off-site TSD facility in February 1996. An additional 6 inches of soil beneath the waste pile was also removed during this operation. Reportedly, there were no indications of spillage or run-off outside the defined 30-foot by 30-foot pile footprint.

The waste pile came to the attention of IDEM during a routine CEI conducted in June 1997. Following the inspection, IDEM issued a NOV to Exide for creating a hazardous waste pile without a permit and for failure to meet the general requirements for a waste pile such as run-on/run-off management, wind dispersal controls, and leachate collection. On July 1998, Exide entered into an AO with IDEM to resolve issues arising from the inspection and resulting NOV.

Exide collected soil samples from four soil borings advanced within the former waste pile footprint and analyzed the soil samples for total lead in August 1998. The soil samples were collected every 6 inches to a depth of 3 feet bgs, and then again at 4 feet bgs and at 5 feet bgs, for a total of 24 samples. Total lead concentrations ranged from 11 to 3,800 mg/kg. The concentrations decreased with depth before increasing in fill materials near the 5-foot depth. Based on the sampling results, IDEM directed Exide to submit a closure plan for the hazardous waste pile. The Closure Plan and Addendas No. 1, 2, and 3 were approved by IDEM in March 2000.

On March 22, 2000, Exide conducted soil sampling to establish background lead concentrations for shallow fills in the vicinity of the former waste pile in accordance with Addendum No. 3 to the hazardous waste pile closure plan. Six soil samples were collected of fill materials to a maximum depth of 18 inches bgs and analyzed for total lead. Total lead concentrations ranged

from 165 to 2,970 mg/kg. In November 2000, Exide removed a uniform layer of 18 inches of soil in an area with dimensions of 40 feet by 40 feet in accordance with the approved closure plan. The excavation area was expanded by 5 feet on each side of the original footprint of the waste pile. No confirmatory samples were collected at the bottom of the excavation because previous sampling events in the proposed excavation area indicated that lead levels below 18 inches bgs were below the RISC Tier 1 Residential values (400 mg/kg). However, lead levels at approximately 5 feet bgs increased to levels above RISC Tier 1 Residential values.

On November 8, 2000, the excavation was backfilled with clean soil imported from Paddock Brothers, Inc. of Frankfort, Indiana. The excavated soils were characterized as hazardous for lead prior to disposal. Approximately 246 tons of excavated materials were treated such that leachable levels of lead were below RCRA TCLP concentrations and disposed at Max Environmental Inc.'s Mill Services TSD facility in Yukon, Pennsylvania. Exide submitted the Hazardous Waste Pile Closure Report to IDEM on January 9, 2001. IDEM approved the closure report on February 14, 2001.

It is not clear whether increasing lead concentrations detected in the fill at 5 feet bgs are isolated to this location or if fill is present at greater depths in other portions of the Site. Soil samples collected from the 2014 investigation boring B-4 (to a depth of 8 feet) in this portion of the Site had low concentrations of lead (< 23.7 mg/kg).

Soil boring R-10 will be performed in this SWMU.

4.3.9 SWMU-9 Parts Cleaners

This unit consisted of two parts cleaners located in the maintenance area in the basement of the plant building (Assembly Basement). The parts cleaners generated waste naphtha, which was disposed off-site by Safety-Kleen. Inspections conducted in 2001 and 2010 did not mention the presence of the parts cleaners on site.

The parts cleaners were located inside the plant building on concrete slab. Spilled material would have been contained by the building. Moreover, because the waste naphtha was a valuable recyclable commodity, it would have made economic sense for both Exide and Safety-Kleen to ensure unit integrity and promptly clean up and containerize any waste naphtha that was spilled. There are no documented releases from this unit.

Given the presence of naptha compounds observed during the 2014 UST closure this location is being further investigated to determine if any link can be made between the two locations. The use of chlorinated solvents for degreasing and parts cleaning is not a practice that is known to have occurred onsite and is not typical for these types of parts cleaners. However, given that the source of the chlorinated solvents observed during the UST closure remains unknown the investigation of SWMU-9 will also look for these compounds.

4.3.10 AOC-1 Loading Dock Area

This AOC was identified by IDEM during a RCRA Compliance Inspection conducted on April 3, 1986. On July 18, 1986, IDEM issued General Battery an NOV (V-137) for depositing waste from spent batteries on the ground in the battery loading area. The loading dock was used for loading spent batteries. The area of concern was a 35-foot by 45-foot area located east of the loading dock.

On January 19, 1988, Exide collected 32 soil samples from 23 locations laid out in a 10-foot grid pattern throughout the 35-foot by 45-foot area east of the battery loading dock. A copy of the results are provided as Attachment A. Soil samples were collected from the first foot from all locations. Lead results from the first sample interval ranged from 17 to 9,300 mg/kg, and cadmium concentrations ranged from 0.09 to 13 mg/kg. Deeper samples (>1.0 ft. bgs) were collected from four locations. Those results ranged from 8.8 to 530 mg/kg for lead, and from <0.05 to 0.71 mg/kg for cadmium. Six samples were also analyzed for leachable levels of lead and cadmium, as measured using the Extraction Procedure Toxicity Test (EP-Tox 1310). EP lead concentrations ranged from 2.4 to 15 mg/L. EP-Tox 1310 concentrations greater than 5.0 mg/L of lead are considered characteristic hazardous waste (D008). The EP-Tox 1310 cadmium

concentrations ranged from non-detect (ND) to 0.02 mg/L, which were below the EP-Tox 1310 regulatory level of 1.0 mg/L for cadmium.

Based on the sampling results, Exide proposed to excavate the 35-foot by 45-foot area to a depth of 6 inches bgs and collect confirmation samples to ensure that the cleanup level of 2,000 mg/kg for lead was successfully achieved. However, IDEM disagreed with the proposed 2,000 mg/kg cleanup level and required that a site-specific cleanup standard be established based on the mean range of a minimum of three background samples collected at least 100 feet from any roadway or process area. In November 1988, IDEM met with Exide to identify appropriate background sample locations. On July 17, 1989, IDEM informed Exide that the site-specific cleanup level would be 78 mg/kg for lead based on the results of the background samples collected by Pollution Control Systems, Inc. In a meeting at the site on July 18, 1989, IDEM and Exide agreed to excavate 1 foot of soil from the surface of the 35-foot by 45-foot spill area and apply a lime buffer to the bottom of the excavation before backfilling to control pH. In August 1992, IDEM issued a notice of compliance for NOV (V-327), which included the area now designated as AOC 1.

No additional specific investigation of AOC-1 is proposed.

4.3.11 AOC-2 Castings/Grid Building Area (Oil Spillage on RR Track)

This AOC was identified by IDEM during a RCRA Compliance Inspection conducted on April 3, 1986. On July 18, 1986, IDEM issued General Battery a NOV (V-137) for depositing oil-contaminated boiler blow-down waste on the ground next to the castings/grid building. During the inspection, oil spillage was noted on the railroad tracks outside of the casting department. According to a facility employee, the oil was from air compressor blow-out. The outfall pipe for "chiller water" was in the same area. Water from the outfall pipe flowed approximately 500 feet through the area and into a loading dock drain.

The oil-contaminated boiler blow-down waste was released to the ground. Consequently, there were no release controls for this AOC. In response to the July 1986 NOV, the facility collected a

sample of waste from the stained area, and then excavated soil in the stained area to a depth of approximately 2 feet bgs, where the soil was no longer visibly stained. The facility collected a soil sample at the bottom of this excavation. Both samples were analyzed for lead, cadmium, oil, and grease. Information regarding the excavation was submitted to IDEM in January 1987.

In a Notice of Inadequacy dated March 27, 1987, IDEM requested further explanation for the 2-foot depth of excavation and asked that the samples of excavated materials also be analyzed for PCBs and total halides. In response to the Notice of Inadequacy, the facility collected another sample of the excavated material and a confirmatory soil sample at the 2-foot depth of the excavation. The samples were analyzed for PCBs and total halides, but no data were found in the available file material during preparation of this CCR.

The facility provided a response letter dated May 29, 1987 which expressed that based on phone conversations with IDEM an excavation to a depth where soil was not visibly stained was sufficient.

Exide also indicated that, based on the analytical results, they planned to dispose of the excavated material as non-hazardous, special waste in a RCRA landfill approved by IDEM.

In August 1992, IDEM issued a notice of compliance for violations associated with AOC 2, including the release of oil-contaminated boiler blow-down waste in the Castings/Grid Building Area.

In 2014 soil boring B-11 was performed in the former grid casting area to a depth of 3 feet. Lead detections in five samples collected ranged from 203 mg/kg in the 0-0.5 feet interval to 9.9 mg/kg at the 2.5-3 feet depth interval. Soil boring B-12 was performed in the former parts casting area to a depth of four feet in five samples collected, the maximum detection was 13.3 mg/kg.

Additional specific investigation is proposed for AOC-2.

4.3.12 AOC-3 Underground Petroleum Storage Tanks (UST-1, 2, and 3)

Three underground storage tanks are known to have existed at the facility. Two of the USTs (UST-2 and UST-3) were located beneath the former fuel shed area located near the northeast gate near the site property line. UST-2 reportedly contained diesel fuel and UST-3 reportedly contained another type (unknown) of fuel oil. A third UST (UST-1) was located centrally onsite (south of Old Formation).

According to a letter from Exide's legal counsel dated October 6, 1997, the 20,000-gallon fuel oil UST (UST-3) was closed in place in April 1987, in accordance with Indiana Fire Marshall regulations in effect at that time. Prior to the closure, a neighboring property owner alleged that a leak from UST-3 had occurred in March 1987. During closure, the UST was emptied of residual fuel oil, cleaned, inspected by the Fire Marshall, and filled with clean fill. According to the maintenance supervisor, no evidence of release was observed during the tank closure.

The 10,000-gallon diesel tank (UST-2) was permanently taken out of service in December 1991 and removed in April 2014. During and following the removal low level detections of vinyl chloride and naptha compounds were observed in surrounding soils (exceeding 2014 IDEM RISC migration to groundwater (MTG) screening levels). Groundwater sampling identified TCE, cis-1,2-DCE, trans-1,2-DCE, 1,1-DCE, 1,1-DCA, vinyl chloride, naphthalene, and 1-methylnaphthalene at concentrations exceeding 2014 IDEM RISC residential tap water and vapor exposure screening levels.

The 10,000 gallon #2 fuel oil tank (UST-1) was also removed in April 2014. Based on sampling performed after removal of the tank and surrounding soil, impacts from petroleum in the vicinity of the former UST-1 location do not exceed the IDEM RISC screening criteria for direct contact or migration to groundwater scenarios. The groundwater sampled onsite around the UST-1 location did not have petroleum or solvent impacts.

Both UST-1 and UST-2 were closed through the IDEM LUST program and granted NFA-status on December 23, 2014. However, the detections of chlorinated solvents in the soils and groundwater (maximum detections of TCE 8,520 ug/L; vinyl chloride 3,590 ug/L) around UST-2 were noted as requiring additional investigation in the future. The EPA reportedly detected concentrations of TCE in indoor air samples collected at an adjacent home at a concentration of 0.44 ppbv (exceeding the vapor intrusion screening limit). As a result, a vapor mitigation system was reportedly installed and is currently operating at the home.

Although the detections of the chlorinated solvents were observed during the removal of the UST located on the Exide property, interviews with a former plant manager conducted during the development of the CCR have not provided any indication that any of those compounds were used, stored, or disposed of onsite. The source of these compounds in the vicinity of the former UST-2 location has not been identified.

Additional investigation of soil and groundwater in the vicinity of UST-2 is needed to determine the extent of contamination as well to identify/confirm an onsite or offsite source. Investigation to confirm the location of UST-3 and any potential impacts is also proposed.

4.4 ONSITE SOIL CHARACTERIZATION

The objective of the soil borings proposed for the RFI is to supplement the existing subsurface information on a site-wide basis as well as in the vicinity of specific SWMUs and AOCs by obtaining samples of the fill/soil materials encountered for chemical analysis.

4.4.1 Random Sampling

A grid-based sampling approach (random sampling) is proposed to gather subsurface information for the soils in former manufacturing areas as well as general site soils. Sample locations will be laid out on a 100 foot grid pattern as shown on Figure 3. The typical boring depth will be 8 feet below the ground surface using a Geoprobe™ direct push sampling device.

Soil samples will be collected at the following intervals: 0-1 foot bgs, 1-2 foot bgs, 3-4 foot bgs, 5-6 foot bgs, and 7-8 foot bgs.

Random sampling soil samples will be field screened for VOCs using a PID. Samples from 0-1 foot bgs, 1-2 foot bgs, 3-4 ft bgs, and 5-6 ft bgs will be analyzed for RCRA 8 metals. The 7-8 foot bgs sample will be archived for possible future analysis. Samples exhibiting odor/staining or elevated PID levels (greater than 10 ppmv over background readings) will be selected by the field sampler for SVOC/VOC analysis. Samples from random sample locations in the former Formation Area (R-20, R-21, R-28, R-29, R-30, and R-37) will be tested in the laboratory for pH.

Samples that are submitted for SVOC/VOC analysis will be analyzed on a 2-day turnaround time in order to allow for follow up sampling. Step out borings could then be performed at locations on an approximate 10-foot offset from SVOC/VOC detections that exceed IDEM non-residential direct contact standards. Step out sampling for random sample locations would be performed based on availability of field personnel and equipment.

4.4.2 Focused Sampling

Focused sampling will be performed to address potential contaminants in the vicinity of the SWMUs and AOCs. The boring depth will be 8 feet below the ground surface using a Geoprobe™ direct push type sampling device.

4.4.2.1 **Focused Sampling at SWMUs**

SWMU-1 (Former Waste Pile #1)

Focused sampling at SWMU-1 will consist of one boring (F-10) advanced in the middle of SWMU-1. Soil samples will be screened and analyzed following the protocol for random sampling described above.

SWMU-2 (Sludge Storage Tank), SWMU-5 (WWTP and Sump), SWMU-7 (Roll Off Container)

Focused sampling at SWMU-2, SWMU-5, and SWMU-7 will include three (3) borings (F-1 through F-3) advanced in the vicinity of the former WWTP structures as shown on Figure 3. Soil samples will be screened and analyzed following the protocol for random sampling described above.

SWMU-3 (Baghouses)

Focused sampling at SWMU-3 will consist of two (2) soil borings (F-4 and F-5) completed at the locations in the vicinity of the former baghouse as shown on Figure 3. Soil samples will be screened and analyzed following the protocol for random sampling described above.

SWMU-4 (Hazardous Waste Accumulation Area)

No focused sampling is proposed for SWMU-4.

SWMU-6 (Filter Building)

Focused sampling at SWMU-6 will consist of one soil boring (F-6) advanced in the middle of former building footprint. Soil samples will be screened and analyzed following the protocol for random sampling described above.

SWMU-8 (Former Waste Pile #2)

No focused sampling is proposed for SWMU-8.

SWMU-9 (Parts Cleaner in Basement)

The exact location of the parts cleaners in the Assembly Basement is unknown. Up to 6 feet of crushed concrete rubble was placed in the basement following demolition of the structures in

2012. The grid locations inside of the Assembly Basement footprint (R-38, R-48, R-49) will also serve as focused sample locations.

Sampling will be attempted at the grid node locations using direct push techniques. If the borings encounter refusal, either on the rubble or the remnants of the basement floor, the grid locations will be offset to the location(s) immediately outside the former basement area. The first soil sample collected from below the original basement floor (or estimated depth if offset locations are utilized) will be analyzed for SVOCs/VOCs.

4.4.2.2 **Focused Sampling at AOCs**

AOC-1 (Loading Dock)

No focused sampling is proposed for AOC-1.

AOC-2 (Casting/Grid Building Area/RR Track)

Focused sampling of AOC-2 will consist of three (3) direct push borings to a depth of 4 feet bgs (F-7, F-8, and F-9). Samples will be field screened using a PID and evaluated for staining and odor. If field screening and/or evaluation suggest a release of oil contaminated water at the boring locations(s), then samples will be collected for SVOC/VOC analysis from one six inch interval within the 0-2 foot increment and from one six inch interval within the 2-4 foot increment representing the most significant odor/staining or greatest PID result as determined by the sampler. No RCRA metals analysis is proposed for the AOC-2 focused sample locations.

(AOC-3) UST Investigation

UST-1 and UST-2 have received a No Further Action (NFA) determination from IDEM; although UST-2 requires further evaluation of VOCs that were identified in soils and groundwater during removal. Focused soil sampling to investigate contamination in the vicinity of the former fuel shed and UST-2 location will be performed using a combination of Geoprobe™ soil and soil-gas sampling.

Geoprobe soil borings will be performed at 6 locations (U-1 through U-6) along Kelley Avenue in the vicinity of the former fuel shed area as shown on Figure 3. Geoprobe borings will be advanced to a depth of 8 feet bgs. Soil Samples will be field screened for VOCs using a PID and samples will be collected for laboratory analysis from the 6 inch interval with the highest PID readings within the 0-4 foot bgs increment and the 4-8 foot bgs increment.

The sampling performed around AOC-3 will be performed early in the investigation and SVOC/VOC samples from locations U-1 through U-6 will be analyzed on a 2-day turn-around schedule so that additional step out sampling can be performed. Step out borings would then be performed at locations on an approximate 10-foot offset from SVOC/VOC detections that exceed IDEM non-residential direct contact standards. Additional rounds of step out sampling may or may not be incorporated into the RFI based on availability of field personnel and equipment.

4.4.2.3 **Focused Sampling at Stormwater Runoff Areas**

During a Site visit in September 2017, evidence of soil erosion was observed at several locations on the perimeter of the Site; most notably near the former rail spur and on the northern fence line. Soil samples will be collected from the surface (0-0.5 foot bgs) and shallow subsurface (0.5-1 foot bgs) intervals at up to five locations (10 samples) with evidence of soil erosion based on field determinations made by Exide. These samples will be analyzed for RCRA 8 metals. Locations will be documented in the RFI Report.

4.5 ONSITE GROUNDATER EVALUATION

The objective of the groundwater investigation in the RFI is to establish the groundwater quality at the Site perimeter as it relates to potential impacts in the shallow zone (approximately 10 feet bgs) and to confirm the groundwater flow conditions at the Site. The gradient/flow in the shallow groundwater zone is anticipated to be towards the north-northeast.

Three (3) shallow (18 feet depth) groundwater monitoring wells (MW-1 through MW-3) will be installed along the northern property boundary at the locations shown on Figure 4 to represent the assumed downgradient conditions. A well (MW-4) will be installed immediately south of the former fuel shed to function as an upgradient well for AOC-3. A site wide upgradient well (MW-5) will be installed in the southeast corner of the Site. Another site wide upgradient well (MW-6) will be installed in the southwest corner of the Site. A downgradient well (MW-7) will be installed in the northwest corner of the property. An additional well (MW-8) will be installed along the western property boundary along Hoke Avenue.

This combination of wells will allow Exide to assess the groundwater quality leaving the Site towards the north-northeast; and provide localized information regarding groundwater quality flowing beneath the former fuel shed.

4.5.1 Geotechnical/Hydraulic Evaluation

During installation of the groundwater monitoring wells, soil samples will be submitted for the following geotechnical or hydraulic testing:

- ASTM D7928 – Particle Size Distribution of Fine Grained Soils Using Hydrometer Analysis;
- ASTM D4318 – Standard Test Methods For Liquid Limit, Plastic Limit, and Plasticity Index of Soils; and,
- ASTM D4044/D4044M – Standard Test Method for Instaneous Change in Head (Slug) Tests for Determining Hydraulic Properties of Aquifers.

Soil samples collected for geotechnical analysis will be taken from the top 1-2 feet of the screened interval.

4.5.2 Well Soil Sampling

Soil borings used to install the monitoring wells using a hollow-stem augur (or direct push) will be completed to depths of up to 18 feet bgs (new monitoring well depth) at eight (8) locations throughout the site. Samples for laboratory analysis of RCRA 8 Metals will be collected from intervals of 0-4 feet bgs, 4-8 feet bgs, 8-12 feet bgs, 12-16 feet bgs, and 16-18 feet bgs. In addition, a sample for VOC/SVOC analysis will be collected from the depth with the highest PID reading. If no elevated PID readings are observed the VOC/SVOC sample will be collected from the bottom of the well.

5.0 FIELD PROCEDURES

5.1 SOIL BORINGS

The following field activities will take place for the soil borings:

- Utilities will be marked in the areas where drilling will take place using both public (i.e., Indiana one call) and private utility locating services.

- The boring locations will be staked out in the field using spray paint markings on asphalt or concrete surface and modifying the location to avoid utilities.

- If necessary, concrete surfaces will be sawcut or cored prior to drilling/boring.

- Samples collected using Geoprobe™ direct push samplers will be advanced in intervals of 4 feet using acetate sleeves. An Advanced GeoServices field representative will log the borings noting the soil types, PID screening results, and presence of free water. Excess soils will be containerized for characterization and disposal.

- Representative samples of the soils and fill materials encountered will be taken from the geoprobe sleeve and analyzed for RCRA 8 metals. Sampling and analysis for TCL VOC/SVOC parameters will be performed at specified locations or when field observations indicate the potential presence of these compounds (PID measurements, odor, sheen, etc.). Discrete samples for VOC analysis will be collected using EnCore or TerraCore samplers.

- After the soil boring is completed, the hole will be backfilled using bentonite chips or grout and the surface restored to its original condition.

Detailed procedures for these activities are laid out in Section 3.0 of the SAP (Appendix A).

5.2 GROUNDWATER

Details of the proposed field procedures to be followed during the RFI are presented in the SAP in Appendix A. The laboratory that will perform the analytical testing is Pace Analytical of Indianapolis, Indiana.

The following tasks will be undertaken for the groundwater:

- Eight (8) new monitoring wells constructed of 2-inch diameter PVC will be installed using hollow-stem auger techniques. The wells are intended to intercept water in the shallow groundwater zone. Drill cuttings will be containerized and disposed of as discussed in Section 5.3. The wells will be developed after installation.

- Two quarters of groundwater sampling will be performed in all 5 wells using low flow sampling techniques and the samples analyzed for total and dissolved RCRA 8 metals and TCL VOCs/SVOCs. Specific conductivity and pH will be measured in the field. Water levels will be obtained in all wells prior to sampling.

Detailed procedures for these activities are laid out in Section 2.4 of the SAP (Appendix A).

5.3 INVESTIGATION DERIVED WASTES

The investigation derived wastes (IDW) expected to be generated include drill cuttings from hollow-stem augers, excess soils and acetate sleeves from geoprobe samplers, groundwater from well development, purging and sampling activities, decontamination fluids and used disposable equipment. The drill cuttings and excess soils and/or collected groundwater will be containerized and representative samples taken for waste characterization purposes based on the landfill requirements. Following review of the waste characterization data, the drill cuttings will be treated as necessary and disposed of off-site at an appropriate disposal facility by Exide.

Spent disposable equipment will be collected in plastic trash bags and disposed of along with facility trash by Exide.

6.0 QUALITY CONTROL AND QUALITY ASSURANCE

To evaluate whether field or laboratory conditions may be impacting analytical samples, equipment blanks, matrix spike/matrix spike duplicate and field duplicate samples will be utilized and evaluated as part of the data review. Pace Analytical's Quality Assurance Manual is included on disk as Attachment B-1 in Appendix B.

6.1 FIELD DULICATE SAMPLES

Field duplicate samples allow for determination of sampling precision of the sampler and the analytical laboratory. One field duplicate sample will be taken for every 20 samples of each medium tested. At least one duplicate will be taken for each medium. For the groundwater, the field duplicate will be taken by filling two sample containers from the same well after purging is complete and the field parameters have stabilized. For the soil samples, two samples will be taken side by side from the sample sleeve and placed into separate containers.

6.2 EQUIPMENT BLANKS

An equipment blank will be prepared when a particular piece of reusable sampling equipment was used for sample collection and subsequently decontaminated for use in additional sampling. The equipment blank will be created in the field by collecting, in the appropriate pre-preserved container, a blank water rinse from the equipment (e.g. geoprobe drive head) after execution of the last step of the field decontamination protocol. One equipment blank shall be collected for every twenty samples collected using the specific piece of equipment. The equipment blanks will be tested for total metals and VOCs.

6.3 MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES

A matrix spike/matrix spike duplicate (MS/MSD) sample will collected as a split for every twenty samples of each medium sampled. The sample will be given the same designation as the parent sample with the addition of MS/MSD. MS/MSD samples determine the accuracy by

recovery rates of the compounds added by the laboratory as defined by the analytical method. The MS/MSD samples also monitor any possible matrix effects specific to the samples and sample medium and the extraction/digestion efficiency. In addition, the analyses of MS and MSD samples check precision by comparison of the two spike recoveries.

6.4 QUALITY ASSURANCE REVIEW

Upon receipt of the electronic data packages, the data will undergo a qualitative QA review to verify the reliability of the data as it is presented. This review is defined in more detail in the Quality Assurance Project Plan (QAPP) in Appendix B. Based on the QA review, qualifier codes will be placed next to specific sample results on the sample data tables, if necessary. A completeness of 90% or greater of unrejected analytical data is required for each medium.

7.0 REPORTING

Following approval of this Work Plan, field activities will begin with the site reconnaissance, initiation of the Site survey and obtaining an Indiana licensed well driller to perform geoprobe borings and install the new monitoring wells.

USEPA will be notified ten days in advance of the start of each type of sampling activity. The field work is expected to take approximately three weeks to complete. The first round of groundwater sampling will occur approximately 30 days later with the second round of groundwater sampling occurring about three months after the first round.

Approximately 60 days following validation of the second set of groundwater sample results, a report on the RFI activities will be prepared and submitted to USEPA for approval. The report will include an analysis of the current groundwater flow conditions, the results of all sampling activities, an evaluation of the groundwater quality at the perimeter of the Facility, and a discussion of the subsurface fill and soil conditions. Recommendations for further assessment and/or corrective measures may also be made.

8.0 SCHEDULE

The UAO (Section VI, Item 11.c) requires that the RFI be completed (including reporting) within 180 days from the USEPA approval of the RFI work plan. The approximate schedule for implementation of the RFIWP is as follows:

- 0-15 Days: Site reconnaissance, obtain utility plans, coordinate site survey, retain well driller/geoprobe operator;
- 15-45 Days: Perform first round of field work (monitoring well installation and soil sampling pending driller availability)
- 45-75 Days: Receive and evaluate soils data, perform first round of groundwater sampling;
- 75-105 Days: Receive and evaluate first round of groundwater data, perform second round of groundwater sampling;
- 105-120 Days: Receive and validate second round of groundwater data; and,
- 120 – 180 Days: Prepare RFI Report and submit to USEPA.

If any observations are made during the RFI period which Exide believes warrant a modification (additional investigation) to the RFIWP, the USEPA will be notified. The 180 day schedule does not anticipate/include delays due to weather, permitting, driller availability, or additional scope.

TABLES

TABLE 1
PROPOSED SOIL BORING ANALYSIS SUMMARY
Exide Technologies
Frankfort, Indiana

Boring ID	Northing	Easting	Depth of Boring (Ft.)	Sample Interval	Minimum Analysis*	Comments
R-1	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-2	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-3	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-4	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-5	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-6	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-7	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-8	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-9	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-10	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-11	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-12	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-13	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-14	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-15	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-16	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-17	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-18	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-19	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-20	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals, PH	Archive/Hold 7-8 ft sample analysis
R-21	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals, PH	Archive/Hold 7-8 ft sample analysis
R-22	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-23	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-24	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-25	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-26	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-27	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-28	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals, PH	Archive/Hold 7-8 ft sample analysis
R-29	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals, PH	Archive/Hold 7-8 ft sample analysis
R-30	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals, PH	Archive/Hold 7-8 ft sample analysis
R-31	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-32	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-33	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-34	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-35	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-36	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-37	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals, PH	Archive/Hold 7-8 ft sample analysis
R-38	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals, VOC/SVOC	Begin Sampling Beneath Basement Floor

TABLE 1
PROPOSED SOIL BORING ANALYSIS SUMMARY
Exide Technologies
Frankfort, Indiana

Boring ID	Northing	Easting	Depth of Boring (Ft.)	Sample Interval	Minimum Analysis*	Comments
R-39	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-40	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-41	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-42	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-43	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-44	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-45	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-46	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-47	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-48	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals, VOC/SVOC	Begin Sampling Beneath Basement Floor
R-49	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals, VOC/SVOC	Begin Sampling Beneath Basement Floor
R-50	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-51	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-52	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-53	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-54	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
R-55	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
F-1	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
F-2	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
F-3	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
F-4	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
F-5	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
F-6	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
F-7	NS	NS	4	0-2 ft, 2-4 ft	TCL VOC/SVOC	
F-8	NS	NS	4	0-2 ft, 2-4 ft	TCL VOC/SVOC	
F-9	NS	NS	4	0-2 ft, 2-4 ft	TCL VOC/SVOC	
F-10	NS	NS	8	0-1 ft, 1-2 ft, 3-4 ft, 5-6 ft, 7-8 ft	RCRA 8 Metals	Archive/Hold 7-8 ft sample analysis
U-1	NS	NS	8	0-4 ft, 4-8 ft	RCRA 8 Metals, TCL VOC/SVOC, GRO/DRO	Select sample based on highest PID readings from each interval.**
U-2	NS	NS	8	0-4 ft, 4-8 ft	RCRA 8 Metals, TCL VOC/SVOC, GRO/DRO	Select sample based on highest PID readings from each interval.**
U-3	NS	NS	8	0-4 ft, 4-8 ft	RCRA 8 Metals, TCL VOC/SVOC, GRO/DRO	Select sample based on highest PID readings from each interval.**
U-4	NS	NS	8	0-4 ft, 4-8 ft	RCRA 8 Metals, TCL VOC/SVOC, GRO/DRO	Select sample based on highest PID readings from each interval.**
U-5	NS	NS	8	0-4 ft, 4-8 ft	RCRA 8 Metals, TCL VOC/SVOC, GRO/DRO	Select sample based on highest PID readings from each interval.**

TABLE 1
PROPOSED SOIL BORING ANALYSIS SUMMARY
Exide Technologies
Frankfort, Indiana

Boring ID	Northing	Easting	Depth of Boring (Ft.)	Sample Interval	Minimum Analysis*	Comments
U-6	NS	NS	8	0-4 ft, 4-8 ft	RCRA 8 Metals, TCL VOC/SVOC, GRO/DRO	Select sample based on highest PID readings from each interval.**
MW-1	NS	NS	18	0-4 ft, 4-8 ft, 8-12 ft, 12-16 ft, 16-18 ft	RCRA 8 Metals, TCL VOC/SVOC	Select SVOC/VOC sample based on highest PID readings from well depth.**
MW-2	NS	NS	18	0-4 ft, 4-8 ft, 8-12 ft, 12-16 ft, 16-18 ft	RCRA 8 Metals, TCL VOC/SVOC	Select SVOC/VOC sample based on highest PID readings from well depth.**
MW-3	NS	NS	18	0-4 ft, 4-8 ft, 8-12 ft, 12-16 ft, 16-18 ft	RCRA 8 Metals, TCL VOC/SVOC	Select SVOC/VOC sample based on highest PID readings from well depth.**
MW-4	NS	NS	18	0-4 ft, 4-8 ft, 8-12 ft, 12-16 ft, 16-18 ft	RCRA 8 Metals, TCL VOC/SVOC	Select SVOC/VOC sample based on highest PID readings from well depth.**
MW-5	NS	NS	18	0-4 ft, 4-8 ft, 8-12 ft, 12-16 ft, 16-18 ft	RCRA 8 Metals, TCL VOC/SVOC	Select SVOC/VOC sample based on highest PID readings from well depth.**
MW-6	NS	NS	18	0-4 ft, 4-8 ft, 8-12 ft, 12-16 ft, 16-18 ft	RCRA 8 Metals, TCL VOC/SVOC	Select SVOC/VOC sample based on highest PID readings from well depth.**
MW-7	NS	NS	18	0-4 ft, 4-8 ft, 8-12 ft, 12-16 ft, 16-18 ft	RCRA 8 Metals, TCL VOC/SVOC	Select SVOC/VOC sample based on highest PID readings from well depth.**
MW-8	NS	NS	18	0-4 ft, 4-8 ft, 8-12 ft, 12-16 ft, 16-18 ft	RCRA 8 Metals, TCL VOC/SVOC	Select SVOC/VOC sample based on highest PID readings from well depth.**
Runoff-1	NS	NS	1	0-6", 6-12"	RCRA 8 Metals	Surface soil sample to be collected where visible runoff is observed
Runoff-2	NS	NS	1	0-6", 6-12"	RCRA 8 Metals	Surface soil sample to be collected where visible runoff is observed
Runoff-3	NS	NS	1	0-6", 6-12"	RCRA 8 Metals	Surface soil sample to be collected where visible runoff is observed
Runoff-4	NS	NS	1	0-6", 6-12"	RCRA 8 Metals	Surface soil sample to be collected where visible runoff is observed
Runoff-5	NS	NS	1	0-6", 6-12"	RCRA 8 Metals	Surface soil sample to be collected where visible runoff is observed
Runoff-6	NS	NS	1	0-6", 6-12"	RCRA 8 Metals	Surface soil sample to be collected where visible runoff is observed

TABLE 1
PROPOSED SOIL BORING ANALYSIS SUMMARY
Exide Technologies
Frankfort, Indiana

Boring ID	Northing	Easting	Depth of Boring (Ft.)	Sample Interval	Minimum Analysis*	Comments
Runoff-7	NS	NS	1	0-6", 6-12"	RCRA 8 Metals	Surface soil sample to be collected where visible runoff is observed
Runoff-8	NS	NS	1	0-6", 6-12"	RCRA 8 Metals	Surface soil sample to be collected where visible runoff is observed
Runoff-9	NS	NS	1	0-6", 6-12"	RCRA 8 Metals	Surface soil sample to be collected where visible runoff is observed
Runoff-10	NS	NS	1	0-6", 6-12"	RCRA 8 Metals	Surface soil sample to be collected where visible runoff is observed

NS- Not Surveyed
* All soil samples will be screened for VOCs using a PID. Soil samples exhibiting staining/odor or elevated PID levels (> 10 ppm above background) will be sampled and analyzed for VOCs/SVOCs at the judgement of the field sampler.
** If no elevated PID readings are observed then the VOC/SVOC sample will be collected from the bottom of the well.

Table 2
Proposed Monitoring Well Construction Summary
Exide Technologies
Frankfort, Indiana

Well Identification	*Stick-up or Flush mount*	*Inner Casing Construction*	*Outer Casing Construction*	*Well Pad*	*Screen Interval Depth (Ft. bgs)*	*Screen Interval Elevation (Ft. above MSL)*	*Top of Inner Casing (Ft. above MSL)*
MW-1	SU	2" PVC	6" Steel	Concrete	8 - 18', TBD	TBD	TBD
MW-2	SU	2" PVC	6" Steel	Concrete	8 - 18', TBD	TBD	TBD
MW-3	SU	2" PVC	6" Steel	Concrete	8 - 18', TBD	TBD	TBD
MW-4	SU	2" PVC	6" Steel	Concrete	8 - 18', TBD	TBD	TBD
MW-5	SU	2" PVC	6" Steel	Concrete	8 - 18', TBD	TBD	TBD
MW-6	SU	2" PVC	6" Steel	Concrete	8 - 18', TBD	TBD	TBD
MW-7	SU	2" PVC	6" Steel	Concrete	8 - 18', TBD	TBD	TBD
MW-8	SU	2" PVC	6" Steel	Concrete	8 - 18', TBD	TBD	TBD

SU: Stick Up
* Final determination on using stickup or flush mount well to be made at time of installation.

TABLE-3
MONITORING WELL ANALYSIS SUMMARY
Exide Frankfort
Frankfort, Indiana

Sampling Event	Matrix	Analytical Parameters	Analytical Method	Geotechnical Testing
RFI Groundwater	Groundwater	TCL Volatiles	8260C	NA
		TCL Semivolatiles	8270C/8270C SIM	NA
		GRO	8015D	NA
		DRO	8015D	NA
		Total RCRA 8 Metals	6010B/7470A	NA
		Dissolved RCRA 8 Metals	6010B/7470A	NA
Well Installation	Soil	TCL Volatiles	8260C	Hydrometer/Grain size
		TCL Semivolatiles	8270C	Atterberg Limits
		Total RCRA 8 Metals	6010B/7471A	Slug test

TABLE-3
MONITORING WELL ANALYSIS SUMMARY
Exide Frankfort
Frankfort, Indiana

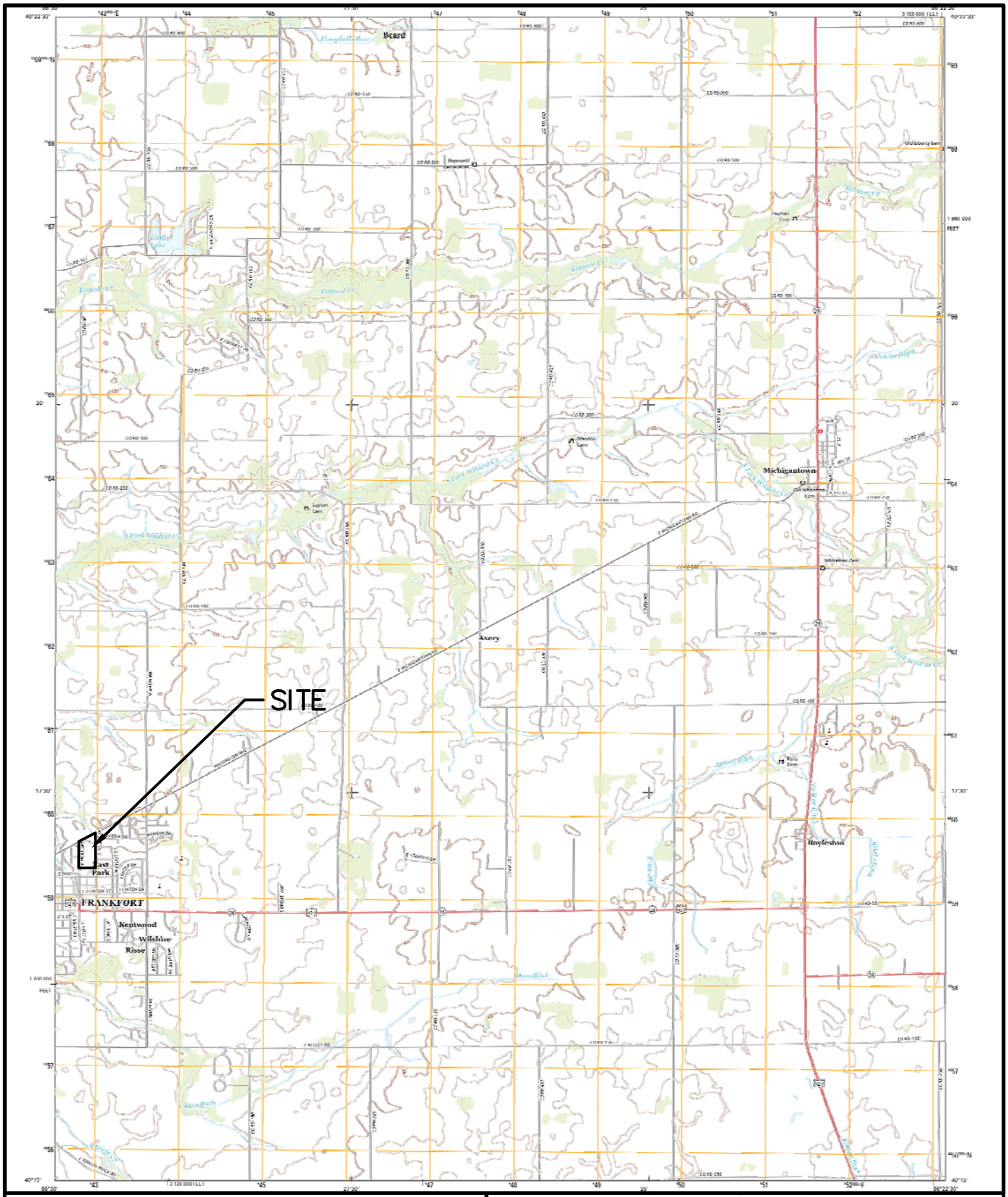
Sampling Event	Matrix	Analytical Parameters	Analytical Method	Geotechnical Testing
RFI Groundwater	Groundwater	TCL Volatiles	8260C	NA
		TCL Semivolatiles	8270C/8270C SIM	NA
		GRO	8015D	NA
		DRO	8015D	NA
		Total RCRA 8 Metals	6010B/7470A	NA
		Dissolved RCRA 8 Metals	6010B/7470A	NA
Well Installation	Soil	TCL Volatiles	8260C	Hydrometer/Grain size
		TCL Semivolatiles	8270C	Atterberg Limits
		Total RCRA 8 Metals	6010B/7471A	Slug test

TABLE 4
SAMPLE QUANTITIES, CONTAINERS AND PRESERVATIVES
Exide Technologies
Frankfort, Indiana

Sampling Event	Total Number of Samples	Matrix	Analysis	Prep Method	Analytical Method	Container	Preservative	Holding Time
RFI Soil	21 (+ 2-MS/MSD/FD)	Soil	TCL Volatiles	5035	8260C	Terracore Sampling Kit	Ice	48 hours to extraction, 14 days to analysis
	21 (+ 2-MS/MSD/FD)		TCL Semivolatiles	3550C	8270C	4 oz WMG	Ice	14 days to extraction, then 40 days to analysis
	12 (+ 1-MS/MSD/FD)		GRO	NA	8015D	Terracore Sampling Kit	Ice	48 hours to extraction, 14 days to analysis
	12 (+ 1-MS/MSD/FD)		DRO	NA	8015D	4 oz WMG	Ice	14 days
	276 (+ 14-MS/MSD/FD)*		RCRA 8 Metals	3050B	6010B/7471A	4 oz WMG	Ice	180 days (Hg-28 days)
	6 (+ 1-MS/MSD/FD)		pH	NA	9045C	4 oz WMG	Ice	ASAP (24 hours)
RFI Groundwater (2 Sampling Events)	8 (+ 1 - MS/MSD/FD)	Groundwater	TCL Volatiles	NA	8260C	3 40mL VOA vials	pH < 2, HCl, Ice	14 days
			TCL Semivolatiles	3510C	8270C/8270C SIM	100 ml Amber	Ice	7 days to extraction, then 40 days to analysis
			1,4-Dioxane	3510C	8270C SIM	1 liter Amber	Ice	7 days to extraction, then 40 days to analysis
			GRO	NA	8015D	3 40mL VOA vials	pH < 2, HCl, Ice	14 days
			DRO	NA	8015D	2 L Amber	Ice	7 days
			Total RCRA 8 Metals	3010A	6010B/7470A	500 ml HDPE	HNO3	180 days (Hg-28 days)
			Dissolved RCRA 8 Metals	3010A	6010B/7470A	500 ml HDPE	Field Filtered, HNO3	180 days (Hg-28 days)
Well Installation	8 (+ 1-MS/MSD/FD)	Soil	TCL Volatiles	5035	8260C	Terracore Sampling Kit	Ice	48 hours to extraction, 14 days to analysis
	8 (+ 1-MS/MSD/FD)		TCL Semivolatiles	3550C	8270C	4 oz WMG	Ice	14 days to extraction, then 40 days to analysis
	40 (+ 2-MS/MSD/FD)		Total RCRA Metals	3050B	6010B/7471A	4 oz WMG	Ice	180 days (Hg-28 days)
Equipment Blanks	3	Aqueous	TCL Volatiles	NA	8260C	3 40mL VOA vials	pH < 2, HCl, Ice	14 days
	3		TCL Semivolatiles	3510C	8270C/8270C SIM	100 ml Amber	Ice	7 days to extraction, then 40 days to analysis
	3		1,4-Dioxane	3510C	8270C SIM	1 liter Amber	Ice	7 days to extraction, then 40 days to analysis
	2		GRO	NA	8015D	3 40mL VOA vials	pH < 2, HCl, Ice	14 days
	2		DRO	NA	8015D	2 L Amber	Ice	7 days
	17		Total RCRA 8 Metals	3010A	6010B/7470A	500 ml HDPE	HNO3	180 days (Hg-28 days)
	1		Dissolved RCRA 8 Metals	3010A	6010B/7470A	500 ml HDPE	Field Filtered, HNO3	180 days (Hg-28 days)

NA - Not applicable. The Extraction method is included in the analytical method.
WMG - Wide mouth glass
Sample containers are based on individual sampling analysis. The laboratory may combine and increase sample container sizes if multiple analysis is requested.
FD - Field Duplicate
EB - Equuipment Blank
* Up to 10 surface soil samples will be collected from the surface in ares where visible runoff is observed. Actual sample locations will be determined in the field. The 10 samples have been included in the total number of RCRA 8 Metals samples. Contingent analysis of 7-8 foot interval samples not included.

FIGURES



F:\Projects\2011\20112678-Exide Frankfort Decon Demo\Cad\UST Removal\Figure 1 - USGS Topo.dwg

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USGS TOPOGRAPHIC MAP

EXIDE TECHNOLOGIES
FRANKFORT, INDIANA

PROJECT ENGINEER:	PGS	SCALE:	NTS
CHECKED BY:	JSD	PROJECT NUMBER:	2011-2678
DRAWN BY:	KEZ	DATE:	FIGURE: 1

F:\Projects\2011\20112678-Exide Frankfort Decon Dem\Cad\2011-2678 Frankfort\Fig 2 Site Plan.dwg

NOTES:

1. THIS DRAWING DEVELOPED FROM EXIDE DRAWING 55-SLI-1D (REV38) DATED 4-29-98.
2. PARCEL BOUNDARIES OBTAINED FROM CLINTON COUNTY BEACON ONLINE TAX MAP. APPROXIMATE LOCATION AS SHOWN.
3. UST LOCATIONS ARE APPROXIMATE.
4. AERIAL PHOTO FROM GOOGLE MAPS (2017).
5. SWMU AND AOC LOCATIONS INFERRED FROM REVIEW OF RCRA DOCUMENTS AND INTERVIEW WITH EXIDE EMPLOYEE.

LEGEND:

APPROXIMATE PARCEL BOUNDARY

100 0 100 200

SCALE IN FEET

EXIDE TECHNOLOGIES
555 HOKE AVENUE
FRANKFORT, INDIANA

CURRENT CONDITIONS 2017 WITH SWMU AND AOC LOCATIONS

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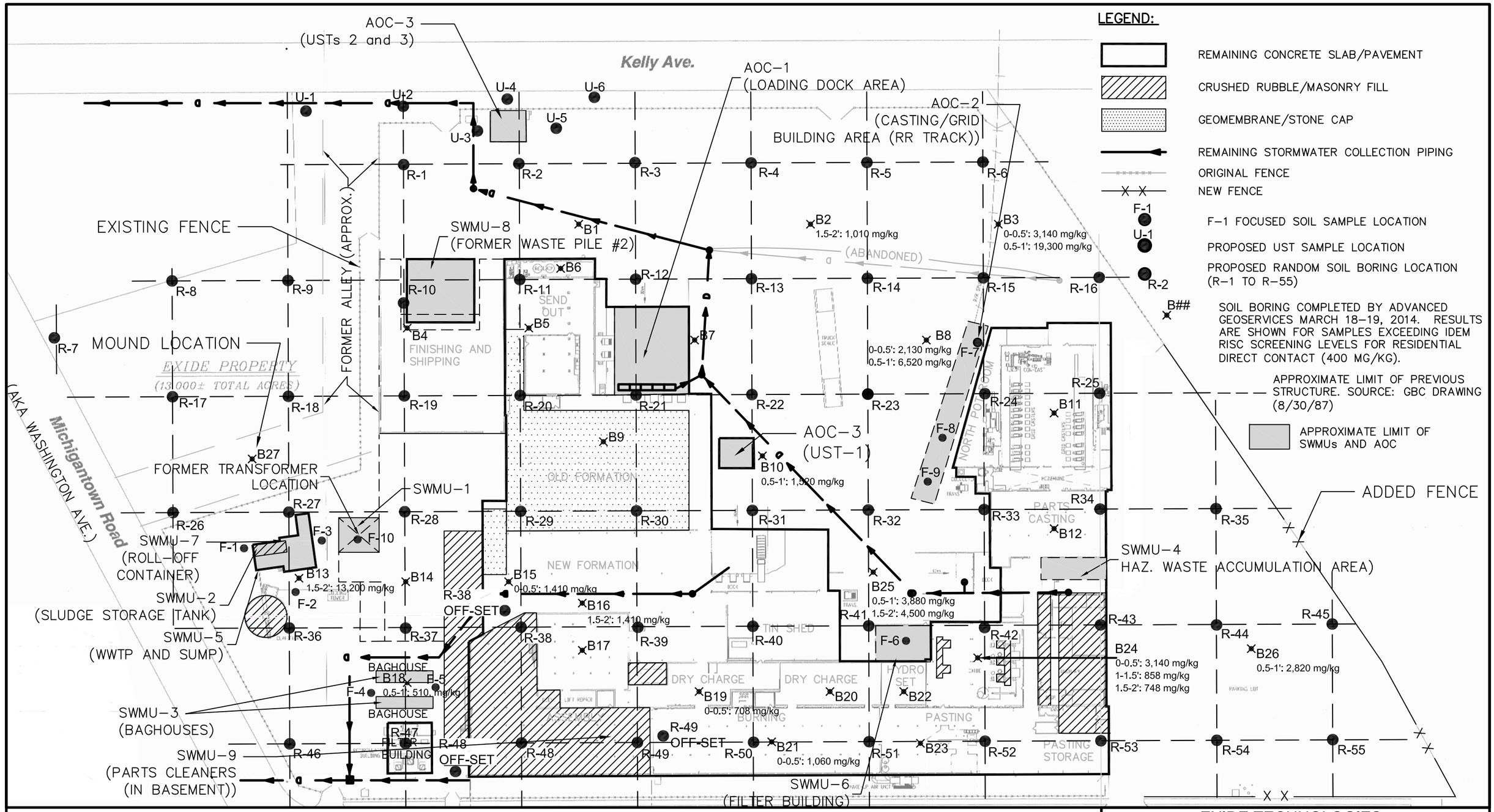
Scale:	1" = 100'
Drawn By:	JSD
Checked By:	PGS
Project Mgr.:	PGS
Originated By:	JSD
Project No.:	20112678
Drawing Date:	5/26/2017
Sheet No.:	OF 1
Revision Number:	

FIGURE 2



NOTES:

1. THIS DRAWING DEVELOPED FROM EXIDE DRAWING 55-SLI-1D (REV38) DATED 4-29-98.
2. AREAS SHOWN FOR REMAINING CONCRETE SLAB, CRUSHED RUBBLE/MASONRY FILL, GEOMEMBRANE/STONE CAP, USTs, AND UTILITY LINES ARE APPROXIMATE, ACTUAL LOCATION MAY VARY.
3. FORMER BUILDING AND WORK AREAS SHOWN FOR REFERENCE ONLY. STRUCTURES NO LONGER EXIST.
4. UP TO 10 ADDITIONAL SOIL SAMPLES WILL BE COLLECTED FROM AREAS WHERE VISIBLE RUNOFF FROM THE SITE HAS BEEN OBSERVED. THE EXACT LOCATIONS WILL BE DETERMINED IN THE FIELD.

80 0 80 160

SCALE IN FEET

EXIDE TECHNOLOGIES

555 HOKE AVENUE
FRANKFORT, INDIANA

EXISTING CONDITIONS WITH SWMU AND AOC LOCATIONS

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Scale:	1" = 80'
Drawn By:	S.D.W.
Checked By:	J.S.D
Project Mgr.:	P.G.S.
Originated By:	J.S.D.
Project No.:	2011-2678
Drawing Date:	1/18/2018
Sheet No.:	OF
Revision Number:	1

FIGURE 3

F:\Projects\2011\2011678-Exide Frankfort Decon Dem0\Cad\2011-2678 Frankfort\Fig 3 Soil Boring Location Plan.dwg

LEGEND:

- REMAINING CONCRETE SLAB PERIMETER
- CRUSHED RUBBLE/MASONRY FILL
- GEOMEMBRANE/STONE CAP
- REMAINING STORMWATER COLLECTION PIPING
- ORIGINAL FENCE
- NEW FENCE
- MW-5 PROPOSED MONITORING WELL LOCATION
- APPROXIMATE LIMIT OF SWMUs AND AOC

MW-1
MW-2
MW-3
MW-4
MW-5
MW-6
MW-7
MW-8

AOC-1 (LOADING DOCK AREA)
AOC-2 (CASTING/GRID BUILDING AREA (RR TRACK))
AOC-3 (USTs 2 and 3)
AOC-3 (UST-1)
SWMU-1 FORMER TRANSFORMER LOCATION
SWMU-2 (SLUDGE STORAGE TANK)
SWMU-3 (BAGHOUSES)
SWMU-4 HAZ. WASTE ACCUMULATION AREA)
SWMU-5 (WWTP AND SUMP)
SWMU-6 (FILTER BUILDING)
SWMU-7 (ROLL-OFF CONTAINER)
SWMU-8 (FORMER WASTE PILE #2)
SWMU-9 (PARTS CLEANERS (IN BASEMENT))

EXIDE PROPERTY
(13.000± TOTAL ACRES)

(AKA WASHINGTON AVE.)
Michigantown Road

Hoke Ave.

(ABANDONED)
SEND OUT
FINISHING AND SHIPPING
OLD FORMATION
NEW FORMATION
NORTH POT ROOM
PARTS CASTING
TIN SHED
DRY CHARGE
DRY CHARGE
HYDRO SET
BURNING
PASTING
PASTING STORAGE
ASSEMBLY
LIFT REPAIR
COOLING TOWER
CLARIFIER
BAGHOUSE
BAGHOUSE
FILTER BUILDING
RECIRCULATION BUILDING
PARKING LOT
TRUCK SCALE
BASEMENT (MAINTENANCE AREA)

NOTES:

1. THIS DRAWING DEVELOPED FROM EXIDE DRAWING 55-SLI-1D (REV38) DATED 4-29-98.
2. AREAS SHOWN FOR REMAINING CONCRETE SLAB, CRUSHED RUBBLE/MASONRY FILL, GEOMEMBRANE/STONE CAP, USTs, AND UTILITY LINES ARE APPROXIMATE, ACTUAL LOCATION MAY VARY.
3. FORMER BUILDING AND WORK AREAS SHOWN FOR REFERENCE ONLY. STRUCTURES NO LONGER EXIST.

80 0 80 160
SCALE IN FEET

EXIDE TECHNOLOGIES
555 HOKE AVENUE
FRANKFORT, INDIANA

MONITORING WELL LOCATION PLAN

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Checked By:	PGS
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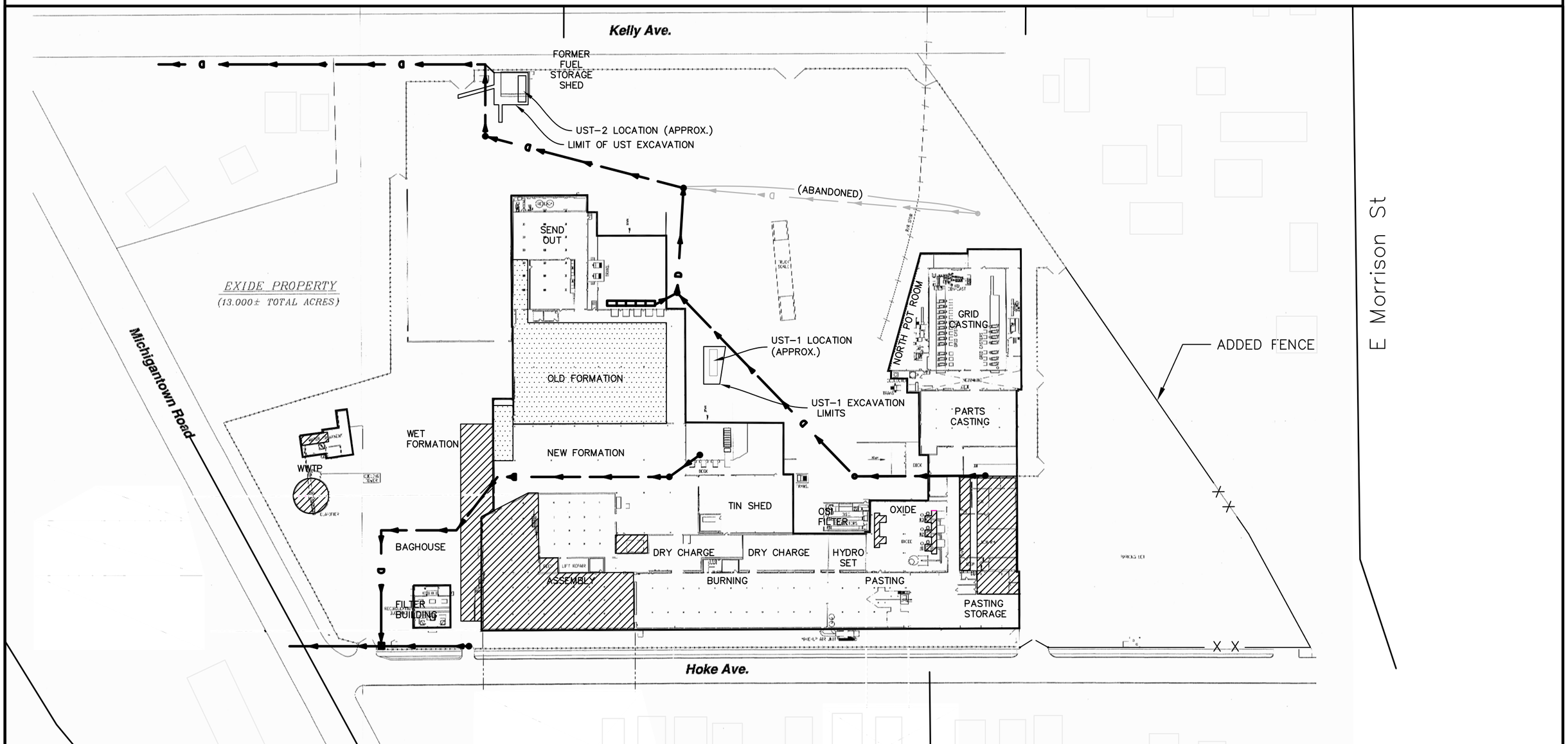
Originated By:	JSD
Project No.:	20112678
Drawing Date:	1/16/2018
Sheet No.:	1 OF 1
Revision Number:	1

FIGURE 4

F:\Projects\2011\20112678-Exide Frankfort Decon Demo\Cad\2011-2678 Frankfort\Fig 4 Monitoring Well Locaton Plan.dwg



NOTES:

1. THIS DRAWING DEVELOPED FROM EXIDE DRAWING 55-SLI-1D (REV38) DATED 4-29-98.
2. AREAS SHOWN FOR REMAINING CONCRETE SLAB AND UTILITY LINES ARE APPROXIMATE.
3. UST LOCATIONS ARE APPROXIMATE.
4. UTILITY LOCATIONS SHOWN ARE APPROXIMATE. CONTRACTOR TO VERIFY, MARK, AND PROTECT.

LEGEND:

- REMAINING CONCRETE SLAB PERIMETER
- CRUSHED RUBBLE/MASONRY FILL
- GEOMEMBRANE/STONE CAP
- REMAINING STORMWATER COLLECTION PIPING
- — X X — NEW SITE FENCE

100 0 100 200

SCALE IN FEET

EXIDE TECHNOLOGIES

555 HOKE AVENUE
FRANKFORT, INDIANA

POST-DEMOLITION CONDITIONS 2014

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Project No.:	20112678
Drawing Date:	5/26/2017
Sheet No.:	3 OF 1
Revision Number:	

FIGURE 5

F:\Projects\2011\20112678-Exide Frankfort Decon Dem\Cad\2011-2678 Frankfort\Historic-Parcel.dwg

ATTACHMENT A

1988 AOC-1 Loading Dock Supplemental Data

POLLUTION CONTROL SYSTEMS INCORPORATED

STATE ROAD THREE AT COUNTY ROAD 550 SOUTH
LAOTTO, INDIANA 46763 TELEPHONE: (219) 637-3137

ANALYTICAL DATA SUMMARY

Sample ID	Sample Depth, feet	Total Lead, mg/k	Total Cadmium, mg/k
1	0.0 - 0.5	940	0.42
2	0.0 - 0.5	2200	1.1
3	0.0 - 0.5	190	13
4	0.0 - 0.5	270	1.3
5	0.0 - 0.5	120	0.36
6	0.0 - 0.5	330	1.1
6	1.5 - 2.0	220	0.16
6	3.0 - 3.5	140	0.20
6	4.5 - 5.0	29	0.11
7	0.5 - 1.0	170	1.0
8	0.5 - 1.0	910	0.78
9	0.0 - 0.5	9300	1.1
9	1.0 - 1.5	530	0.59
10	0.0 - 0.5	2300	0.60
11	0.0 - 0.5	420	1.0
12	0.0 - 0.5	17	0.56
13	0.4 - 0.9	19	0.92
13	1.0 - 1.5	82	0.29
14	0.0 - 0.5	310	0.27
15	0.0 - 0.5	1900	1.4
16	0.0 - 0.5	310	1.2
16	1.0 - 1.5	54	0.71
16	2.0 - 2.5	12	<0.05
16	3.0 - 3.5	15	<4.9
16	4.5 - 5.0	8.8	0.12
17	0.5 - 1.0	100	0.77
18	0.5 - 1.0	740	2.6
19	0.0 - 0.4	220	0.28
20	0.0 - 0.5	8.5	0.09
21	0.0 - 0.5	1100	1.1
22	0.5 - 0.9	200	0.13
23	0.5 - 1.0	1600	1.4

EXIDATA/FEB88/CRO

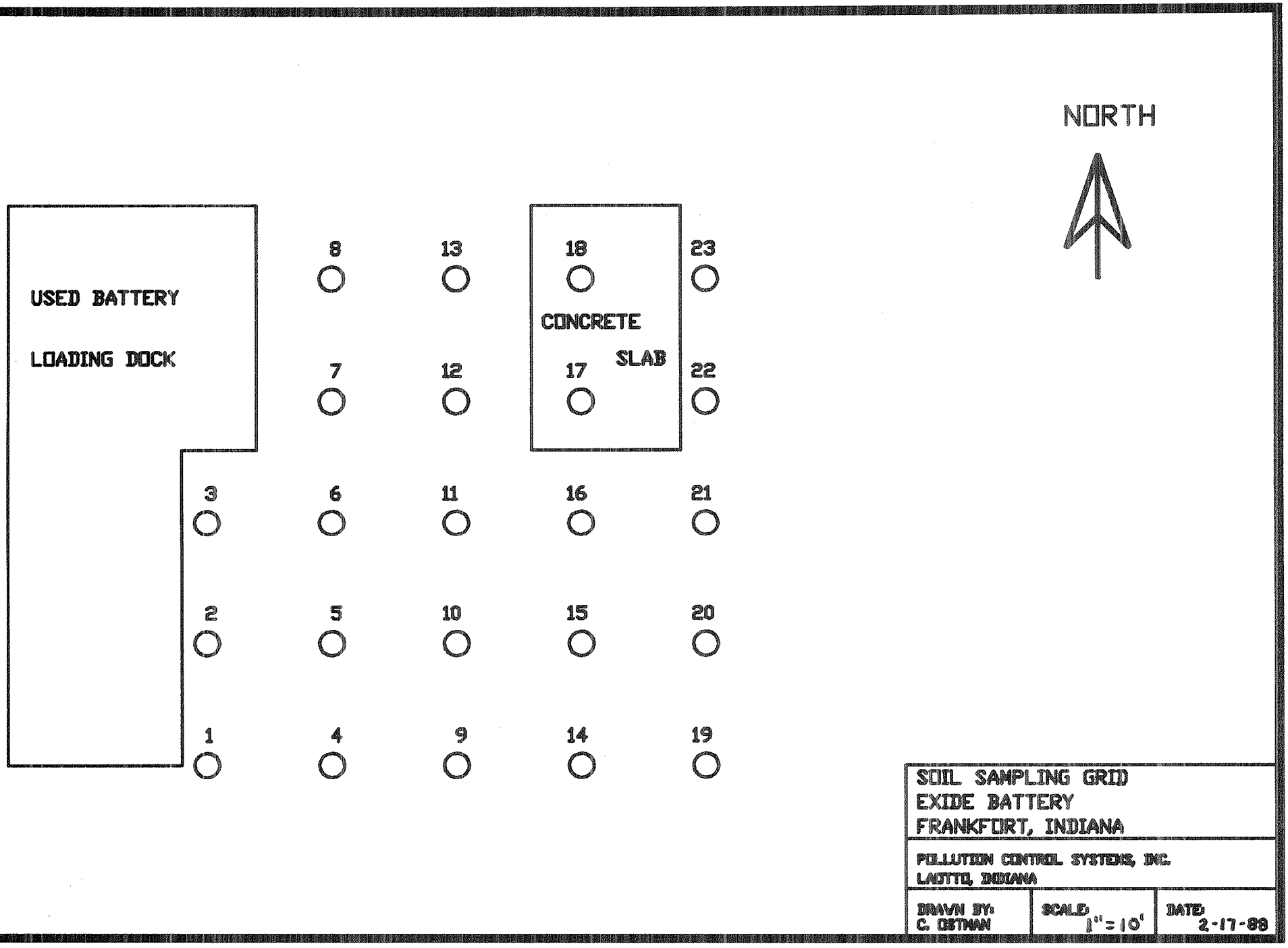
POLLUTION CONTROL SYSTEMS INCORPORATED

STATE ROAD THREE AT COUNTY ROAD 550 SOUTH
LAOTTO, INDIANA 46763 TELEPHONE: (219) 637-3137

ANALYTICAL DATA SUMMARY

Sample ID	Sample Depth, feet	Leachable Lead, mg/l	Leachable Cadmium, mg/l
3	0.0 - 0.5	15	0.02
4	0.0 - 0.5	15	0.02
7	0.5 - 1.0	5.6	0.02
13	0.4 - 0.9	4.3	0.02
15	0.0 - 0.5	4.1	0.02
22	0.5 - 0.9	2.4	<0.01

EXIDATA2/FEB88/CRO


NORTH
USED BATTERY
LOADING DOCK
CONCRETE
SLAB
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
SOIL SAMPLING GRID
EXIDE BATTERY
FRANKFORT, INDIANA
POLLUTION CONTROL SYSTEMS, INC.
LAFAYETTE, INDIANA
DRAWN BY:
C. OSTMAN
SCALE:
1" = 10'
DATE:
2-17-88

APPENDIX A

Sampling and Analysis Plan

SAMPLING AND ANALYSIS PLAN
FOR THE
RCRA FACILTY INVESTIGATION
FORMER EXIDE MANUFACTURING FACILITY
Frankfort, Indiana

Prepared for:

EXIDE TECHNOLOGIES
Milton, Georgia

Prepared by:

ADVANCED GEOSERVICES
West Chester, Pennsylvania

Project No. 2011-2678-11
February 26, 2018

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PAGE NO.

TABLE OF CONTENTS
(Continued)

PAGE NO.

LIST OF FIGURES

FIGURE

1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) has been developed for the RCRA Facility Investigation (RFI) Work Plan (WP) at the former Exide Technologies (Exide) battery manufacturing facility (the Facility) located at 555 North Hoke Avenue in Frankfort, Indiana (the Site). This SAP provides the specific descriptions of field groundwater and soil sampling procedures to be performed during the RFI activities. The Quality Assurance Project Plan (QAPP), provided as Appendix B, provides the quality assurance procedures for the field sampling and laboratory analysis of the samples. Some of the field QA procedures have been included in each section of the SAP, where appropriate.

The SAP consists of sections for each type of sampling that will occur followed by general procedures as follows:

Section 2	Groundwater
Section 3	Soil Borings
Section 4	Sample Handling Procedures

1.1 FACILITY BACKGROUND INFORMATION

Based on available information contained in the Consent Order and a November 28, 2011 Letter Report prepared by USEPA, Prest-O-lite Manufacturing owned the Site during the World War II era. Prest-O-Lite was a car equipment manufacturer (including lead acid batteries). Based on Polk's City Directory in 1959 a telephone exchange registered to P.R. Mallory & Co. Inc. was listed for the address. P.R. Mallory & Co. manufactured electronics including dry cell batteries and eventually became Duracell. General Battery Corporation purchased the Site in 1963, which is consistent with additional information from Polk's City Directory which shows the telephone exchange for the address belonging to General Battery & Ceramic Co. Exide assumed ownership of the Site during the acquisition of General Battery Corporation in 1988. Exide currently owns the parcels that make up the Site. At its peak the facility produced over 12,000 automotive batteries

per day. The Standard Industry Classification (SIC) code for the facility was 3691, Battery manufacturing. Battery manufacturing operations ceased in 1997. The Site was used by Exide for equipment storage until the demolition project in October 2012.

In October 2012, Exide voluntary performed a decontamination of the facility followed by demolition of the above grade structures. The decontamination and demolition project was completed in January 2013. Aside from UST removal and a limited soil investigation performed in 2014 the Site has been inactive since the completion of demolition. The Site buildings have been demolished and it is currently vacant.

1.2 FACILITY OPERATIONS

During the battery manufacturing process, metallic lead was received at the facility, melted, and cast into grids and posts. Lead oxide paste was also manufactured at the facility and subsequently applied to the grids. Lead oxide was created by feeding molten lead into a reactor and mixing it with air to oxidize the lead. Pasted grids were placed in stacks that formed the core of the battery. The cores were placed in battery cases that were produced offsite. Then the remaining components were added. Electrolyte (dilute sulfuric acid) was then added to the battery and a charge was applied (formation). After formation, the battery was cleaned, finished, labelled, and packaged for shipment to retailers and distributors.

Water was used to cool batteries after charging and also to wash batteries prior to shipment. Cooling water was collected in floor drains and emptied to a sump in the wastewater treatment plant (WWTP) located on the northwest corner of the Site. The sump collected wastewater from the entire plant, including acidic water from the cooling and washing process, and lead from washing and dry charge operations. The corrosive wastewater (containing primarily dilute sulfuric acid and lead) was pumped into two above ground holding tanks outside the building and then into reactor tanks located inside the WWTP building (SWMU-5).

Wastewater was treated with lime to neutralize the pH and precipitate dissolved metals. Following neutralization, the wastewater flowed into a large clarifier immediately adjacent to the WWTP building. Precipitated solids settled to the bottom and were pumped to a sludge holding tank (SWMU-2). From June 1986 until operations ceased the sludge was dewatered using a filter press. Sludge cake generated by the filter press was collected in a roll-off container (SWMU-7) for offsite disposal. Extracted water was recycled back to the clarifier. Prior to the use of the filter press, sludge was dewatered using vacuum pan filters and the filter cake was temporarily stored in an enclosure building (SWMU-1) prior to offsite disposal. Clarified water was then discharged to the City of Frankfort sanitary sewer system in accordance with a discharge permit.

Lead emissions were generated from molten lead handled in melting pots and casting machines. Plastic fumes were also generated from the battery case heat sealing process. Air handling equipment was used to vent these emissions to baghouses on the southeast side of the building. Lead oxide dust also was vented through ducting to a baghouse (SWMU-3). In general, air from the Facility was cycled through a filtering system called the OSI in the filter building (SWMU-6).

2.0 GROUNDWATER

The objective of the groundwater investigation during the RFI is to quantify the groundwater quality at the downgradient limits of the Site (expected to be north-northeast). Soil and groundwater sampling completed as part of the UST closure in 2014 identified chlorinated solvents and naphtha compounds in the vicinity of former UST-2 and UST-3. No site-wide groundwater network exists.

During the UST closure in 2014 geoprobe groundwater samples were collected from depths of up to 12 feet below ground surface (bgs). Groundwater was encountered at approximately 10 feet bgs. The results were not definitive as to the hydraulic gradient onsite, although expectations are that shallow groundwater flow is toward the north-northeast.

Eight new monitoring wells will be installed at the locations shown on the Monitoring Well Location Plan, attached as Figure 4 of the RFI Work Plan to serve as upgradient and downgradient monitoring locations. Following installation of the new monitoring wells, a round of groundwater elevations will be collected from all accessible wells to create the piezometric maps for the facility. Two rounds of low flow sampling spaced approximately three months apart and analysis of the samples for total and dissolved RCRA 8 metals, and TCL VOCs/SVOCs. Specific conductivity, pH, oxidation-reduction potential (ORP), turbidity, dissolved oxygen, and temperature will be measured in the field during groundwater sampling.

2.1 DATA QUALITY OBJECTIVES

Groundwater monitoring will be conducted to determine whether site-related constituents are present at the Site perimeter at concentrations above the Indiana Department of Environmental Management (IDEM) Office of Land Quality (OLQ) Screening Levels for Residential Tap Water (RCGs) and to confirm the groundwater flow conditions. To meet this goal, the following data quality objectives (DQOs) have been established. To determine the project DQOs, a series of planning steps are used. The DQO development process is used to optimize the data collection

necessary to meet the applicable decision criteria as defined in USEPA *Guidance for the Data Quality Objective Process* dated August 2000 and USEPA *Data Quality Objectives Process for Hazardous Waste Site Investigations* dated January 2000. The seven steps of the DQO process are presented below.

2.1.1 State the Problem

There is a potential that heavy metals identified in site soils could be released to the groundwater. Based on limited chemical data they can contain lead and/or other heavy metals at elevated concentrations. Infiltration into these fill materials is limited by the buildings and the asphalt and concrete pavements at the ground surface. Furthermore, lead and the other heavy metals associated with facility operations are soluble only in very low or very high pH conditions.

During the UST closure groundwater samples detected low levels of naphtha compounds and elevated levels of chlorinated solvents at concentrations exceeding the RCGs. Chlorinated solvents were not known to be used on the Site and the source of the detections observed in previous groundwater samples has not been determined (on or offsite). Monitoring well locations around the former USTs and in likely upgradient perimeter locations are intended to help identify/delineate the source for the chlorinated solvents.

Low flow sampling techniques will be used that minimize the resuspension of sediments and yield results that are representative of the groundwater conditions.

2.1.2 Identify the Decision

Groundwater monitoring will be conducted to determine if site contaminants, if present, have been released beyond the facility perimeter as well as to assist in determining a location of the source.

2.1.3 Identify Inputs to the Decision

The Performance Standards are the IDEM 2017 OLQ Screening Levels established for groundwater. These are referred to as RCGs. The most stringent groundwater criteria is for residential tap water and those standards will be used for screening. No industrial groundwater standards exist for tap water. Residential and commercial/industrial standards for groundwater vapor exposure exist, but are less stringent than the tap water screening level.

Pace Analytical is expected to be utilized for analysis of groundwater samples. The laboratory that was selected participates regularly in performance evaluation audits as part of their laboratory certification efforts. Pace is a NELAP accredited laboratory. USEPA SW-846 methods will be used in the sample analysis of total and dissolved metals (RCRA 8) and TCL list VOCs and SVOCs; see Table 3 in the QAPP (see Appendix B) for the specific methods.

2.1.4 Define the Study Boundaries

Groundwater sampling will occur at the following wells:

Downgradient (Site):	MW-1, MW-2, MW-3, MW-7
Upgradient (former USTs):	MW-4
Upgradient (Site):	MW-5, MW-6, MW-8

2.1.5 Develop a Decision Rule

Total and dissolved RCRA 8 metals concentrations will be compared to the RCGs. If the results exceed the RCGs at the downgradient perimeter, then additional evaluation and investigation may be conducted to identify the source of the elevated concentrations. If groundwater results exceed the RCGs, a fate and transport study will be conducted along with a risk assessment in order to determine the need for corrective action, if any.

VOC/SVOC concentrations (notably chlorinated solvents and fuel related compounds) will be compared to the RCGs. Upgradient wells (MW-4, MW-5, MW-6, and MW-8) will provide important information on the direction of groundwater flow and potential source of the contaminants (i.e., onsite or offsite).

2.1.6 Specify Limits of Decision Errors

The sample data are subject to random and systematic errors during field collection and sample analysis. The combination of errors is referred to as "total study error." The two contributors to the total study error are the statistical sampling error and the measurement error. The statistical sampling error occurs when the data collection plan fails to provide a limited variability within the decision unit necessary for accurate decision. Measurement errors are introduced during the processes to which the sample is subjected such as physical collection, sample handling, sample preparation, sample analysis, and data reduction. Since the total study error directly affects the possibility of making a decision error, the total decision error must be managed by minimizing the sample design and measurement errors.

The following are some ways to minimize the statistical sampling error:

- Use low-flow purging techniques when collecting the groundwater samples to minimize the turbidity of the samples.

The following are some ways to minimize measurement errors:

- Have the samplers collect the samples in a standardized manner;
- Ensure that each sample is properly preserved, labels, and transported to the laboratory under chain-of-custody;
- Specify that the laboratory use accepted USEPA Methods and report the data using the proper units;

- Specify that the laboratory participate in regular performance testing and also has NELAP certification;
- Receive the analytical data from the laboratory in an electronic format to minimize transcription errors; and,
- Perform a qualitative review (data validation) of the analytical data, as specified in the QAPP (see Appendix B), to verify the reliability of the data.

2.1.7 Optimize the Design for Obtaining Data

Upon review of the analytical data, future groundwater monitoring may be proposed to optimize the long-term program design. The design options will be evaluated based on cost and the ability to meet the DQOs and presented to the USEPA for approval. Design options may include additional monitoring well locations and/or additional groundwater parameters to aid in fate and transport modelling.

2.2 SAMPLE LOCATION AND FREQUENCY

Two groundwater monitoring events will be conducted after the installation of eight proposed wells onsite. The groundwater monitoring events will be completed approximately 3 months apart with the first event being at least 2 to 4 weeks after well development. The well locations for sampling are shown on Figure 4 of the RFI Work Plan.

2.3 SAMPLE DESIGNATION

Groundwater sampling will be conducted at the wells identified in Section 2.1.4 of this SAP. Samples will be labeled by the well identification (e.g., MW-1, MW-2, etc.).

2.4 WELL INSTALLATION

Three shallow zone wells (MW-1, MW-2, and MW-3) will be installed along the northern property boundary. An upgradient (inferred) shallow zone well (MW-5) will be installed in the southwest corner of the Site inset from Hoke Avenue. Another upgradient (inferred) shallow zone well (MW-4) will be installed on the eastern side of the Site south of the UST area. An upgradient well (MW-6) will be installed in the southeast corner of the Site. A downgradient well (MW-7) will be installed in the northwest corner of the property. An additional well (MW-8) will be installed along the western property boundary near Hoke Avenue.

The wells will be installed using hollow-stem auger techniques (direct push methods may also be accepted). During drilling, soil samples will be collected from the 0-4 foot bgs, 4-8 foot bgs, 8-12 foot bgs, 12-16 foot bgs and 16-18 foot bgs intervals. Soil samples collected during well installation will be analyzed for RCRA 8 metals. In addition, a sample will be collected from the 6 inch interval with the highest PID reading for SVOC/VOC analysis. If no elevated PID reading is observed, the SVOC/VOC sample will be collected from the bottom of the well. Soil conditions will be recorded and provided on boring logs.

The proposed monitoring well construction detail is shown on Figure A-1. Hollow stem auger drilling techniques using 6-inch (minimum) inner diameter augers will be used to drill and install the monitoring wells. The monitoring wells will be finished as stick-up wells (flush mount may be considered depending on the conditions where they are installed).

Eight monitoring wells will be constructed. All wells will be constructed of two-inch diameter Schedule 40 PVC screen and riser. A 5 to 10-foot long 0.010" slot screen will be installed in the first water bearing zone encountered to intercept the uppermost part of the aquifer. The bottom of the screen set is expected to be set approximately 18 feet below the ground surface.

Specific monitoring well installation procedures include:

- Design of monitoring well construction criteria;
- Approval of construction detail by Advanced GeoServices Indiana Professional Geologist;
- Installation of PVC screen with riser;
- Installation of shallow well sand pack in preferred shallow zone strata;
- Installation of seal layer; and,
- Installation of outer casing stick-up.

The Advanced GeoServices field technician will design the monitoring well based on the soil/sediment conditions encountered. The field technician will contact the Advanced GeoServices Indiana Professional Geologist prior to installation of the monitoring well for confirmation of design parameters.

Based on the measurements of the total depth and depth to water, the borehole bottom will be plugged with a 2 feet of Portland Type II and bentonite (5%) if necessary to reach two feet below the design elevation for the bottom of the well. A two-foot bentonite layer using 3/4-inch, coated bentonite pellets will be installed over the top of the grout. The bentonite will be hydrated using approximately three gallons of potable water after installation. The bentonite will be hydrated for a minimum period of fifteen minutes prior to any other installation procedures.

The screen will be installed above the bottom bentonite layer. A sand pack (#1 sand) will be installed in the borehole by tremie pipe to approximately two feet above the screen. A two-foot thick seal of bentonite pellets will be installed above the sand pack and hydrated (using approximately three gallons of potable water) for 15 minutes. An outer steel casing will be installed and grout will be installed using a tremie pipe above the bentonite seal using a 5% bentonite/Portland Type II mixture to approximately 0.5-feet above surface grade for stickup wells or 0.5 feet below surface grade for flush-mount wells, within the annular space of the inner and outer casings.

Locking caps and locks will be installed on the top of each PVC inner casings. Aluminum locking covers will be properly bolted to the outer casing upon completion (cast iron lids will be used for any flush mount wells). All monitoring well construction procedures will be documented in the field notebook. Monitoring well designations (i.e., MW-1, MW-2, etc.) will be clearly labeled using a permanent marker on the lid (inside and outside). The location of the well and the elevation of the top of the inner well casing will be determined by an Indiana licensed surveyor.

Following installation and development of the new monitoring wells, a round of groundwater elevations will be collected from all accessible on-site wells to generate peizometric maps for the Facility. The new monitoring wells will be sampled in the two groundwater sample events planned as part of the RFIWP as noted above in Section 2.2 and will follow sampling procedures as described below in Section 2.5

2.5 FIELD PROCEDURES

2.5.1 Well Development

Following installation of the wells and at least 2 weeks prior to groundwater sampling, development will be performed in the new wells. Development will consist of agitation of the well bottom to suspend silt and removal of silt laden water via air-lifting followed by purging with submersible pump until purge water generally has low levels of turbidity (less than 20 NTUs) and a true bottom has been established. It is anticipated that approximately 3 well volumes may need to be purged to meet the development turbidity goals but in no case will more than 5 well volumes be purged. Purge water will be collected and stored onsite until it can be characterized and disposed of by Exide.

2.5.2 Sampling Equipment

The following equipment will be used for the groundwater well sampling:

- Low-flow bladder pump and control box capable of sampling groundwater at depths expected at the Site;(peristaltic pumps are an acceptable alternative)
- Nitrogen tank;
- Electronic depth to water meter;
- Flow-through-cell consisting of pH/temperature meter, conductivity meter, dissolved oxygen meter and an oxidation-reduction potential meter;
- Turbidity Meter;
- Laboratory supplied containers for the collection of samples; and,
- Teflon® tubing.

The groundwater sampling is comprised of synoptic water level measurements, field analysis, well purge techniques, low-flow sample collection, and decontamination procedures.

2.5.3 Synoptic Water Levels

Prior to all groundwater sampling events, depth-to-water will be measured in each well using an electronic water level indicator. The synoptic measurements will include the measurement of water levels and well depths in the monitoring wells in as short a time frame as possible to determine the potentiometric surface across the Site. The field personnel will measure the water levels in the wells to the nearest 0.01 foot using the surveyed point at the top of the inner well casing for reference.

2.5.4 Field Measurements

Field measurements that will be performed during well purging will include pH, specific conductivity, temperature, oxidation/reduction potential (ORP), dissolved oxygen (DO), and turbidity. Measurements will be collected by inserting the appropriate probe in a closed non-dedicated plastic container (flow-through-cell) that is rinsed with deionized water prior to purging the well. Turbidity samples will be collected from the flow-through-cell outflow.

Calibration of the instruments will be completed at the beginning of each sampling day, checked in the middle of the day, and as otherwise necessary based on the functioning of the meters and equipment. The following items outline the calibration of each meter. Each meter will be field calibrated in accordance with the manufacturer's specifications and appropriate calibration solutions. All calibrations will be recorded in the field logbook. Field calibration procedures at a minimum will include the following:

- Calibration of the field instruments will be performed by trained technicians prior to the mobilization of equipment to the Site. All the instruments will be calibrated as specified by the manufacturer. Standard solutions will also be checked to determine stability and operating conditions. All results of field calibrations and measurements will be maintained in bound field logbooks at least daily when the instrument is in use. The recorded calibration information will include date and time of calibration results.

- pH meters will be calibrated according to the manufacturer's instructions prior to each use and will, at a minimum, consist of three standard buffer solutions (pH 4, 7 and 10) obtained from chemical supply houses. The pH values of the buffers will be compensated for the temperature at which the pH sample is measured. Verification checks will be completed at least once per day using a standard solution. The verification check results must agree within ±0.05 pH standard units or re-calibrations will be performed.

- All temperature measurements will be measured using a field thermometer and recorded to ±0.2ºC.

- Dissolved oxygen meters will be calibrated to ambient air conditions.

- Specific conductance meters will be calibrated prior to each use using a potassium chloride solution (1,000 µmhos or 1,423 µmhos) as prepared by a qualified laboratory or chemical supplier.

- Turbidity meters will be calibrated daily prior to use by a minimum of two standards of known turbidity as prepared by the manufacturer of the instrument.

- Oxidation – Reduction Potential probes will be checked against standard solutions (at least one) prepared by a qualified laboratory or chemical supplier.

All calibration procedures performed will be documented in the field logbook and will include the date and time of calibration, name of the person performing the calibration, reference standards used and instrument readings.

If equipment fails calibration or equipment malfunction is noted during calibration or use, the equipment will be tagged and removed from service.

2.5.5 Purging Procedures

Sampling procedures will include water level measurements, calculation of well volumes, purging, and sampling activities. The following step-by-step procedures are in adherence to the Pennsylvania Department of Environmental Protection (PADEP) *Groundwater Monitoring Guidance Manual* (December 1999) and USEPA Region III groundwater sampling protocols for low flow pump purging and sampling, which are based upon the method of Puls and Barcelona (EPA/540/S-9S/504).

Step 1 Measure depth-to-water of every well at the Site.

Step 2 Calculate one well volume of the screened or open interval.

Step 3 Lower the low-flow pump in the well to collect groundwater samples. The pump intake will be placed at the approximate mid-point of the screened or open borehole section of the well. Likely groundwater sample interval depth/elevations are presented in the Well Construction Details (see Figure A-1).

Step 4 Calibrate meters.

Step 5 Begin to purge well. USEPA recommends a purge rate of 100 to 300 milliliters/minute (ml/min). The purge rate should not exceed the recharge rate (i.e., less than 0.3 feet of draw down from the static water level).

Step 6 Measure purging parameters at a minimum of 1 measurement every 3 to 5 minutes. Measurements will be collected via a flow-through-cell for pH, temperature, specific conductivity, ORP, and DO. Turbidity will also be measured at the outflow of the flow through cell every 3 to 5 minutes. All measurements will be recorded in the field logbook.

Step 7 After conductivity and temperature have stabilized to within 3% over three readings, pH readings differ <0.1 standard pH units, ORP readings differ within 10 mV, and turbidity measurements differ within ±10%, sampling can begin after the flow-through-cell is disconnected.

Step 8 The sample will be collected out of the discharge line. The date and time of the sample collection will be recorded in the field logbook.

The pump and sampling equipment will be decontaminated before and between each well. Decontamination procedures are presented in Section 4.2.5.

2.5.6 Sample Collection Procedures

Groundwater samples will be collected for total and dissolved RCRA 8 metals and TCL VOCs/SVOCs using the low-flow pump and tubing at a rate of 100 to 300 ml/min with the flow-through-cell disconnected. For total metal, VOC, and SVOC analyses, groundwater will be collected directly into a laboratory prepared, pre-preserved container. For dissolved metal analyses, groundwater will be transferred through a new 0.45 micron filter and then collected into a laboratory supplied, pre-preserved container. As per QAPP Table B-3 (see Appendix B), metal analyses samples will be collected in 500 ml HDPE bottles that are preserved with nitric acid to a pH value of less than 2 standard units. SVOC analysis samples will be collected in 100 mL amber jars. VOC analysis samples will be collected in three (3) 40 mL VOA vials that are preserved with hydrochloric acid to a pH value of less than 2 standard units. VOC and SVOC samples will also be refrigerated or preserved with ice to maintain a temperature of less than 6 °C. Pump and tubing will be decontaminated while filters will be discarded after each use.

The selected analytical laboratory will perform all analyses in accordance with accepted USEPA publication SW-846 methods so that the detection limits are lower than the applicable RCG.

Sample handling will be in accordance with the procedures outlined in Section 5.0. The appropriate methods for extraction and analysis and required holding times to be met are given in the QAPP in Appendix B.

2.5.7 Decontamination and Waste Handling

The pump will be disassembled and pump components and tubing will be decontaminated in the following manner.

- Alconox and water wash,
- Distilled water rinse,
- Nitric acid rinse (10% solution),
- Distilled water rinse, and,
- Air dry and store in plastic.

After decontamination of the pump components and tubing, two volumes of distilled water will be flushed through the pump assembly and tubing to ensure all decontamination fluids have been removed. Decontamination water will be collected for disposal with collected purge water.

2.5.8 Purge/Waste Water Sampling

If groundwater sample data is not considered acceptable for disposal characterization of purge water; samples of containerized purged water will be collected using a bailer or a dip sampler and analyzed in accordance with disposal facility requirements.

2.6 QUALITY ASSURANCE/QUALITY CONTROL

The field quality control/quality assurance sample results will be used to assess the variability of the groundwater sample results at the end of the sampling period when considering whether corrective action is needed.

2.6.1 Temperature Blank

Temperature blanks (indicator) are designed to address sample preservation. The temperature blank is a vial (usually plastic) filled with water. Each cooler submitted to the laboratory will contain a temperature blank. Upon receipt by the laboratory, the sample custodian opens the cooler and places a NIST-traceable thermometer into the temperature blank, closes the lid and after a few minutes measures the cooler temperature. The temperature blank is used for analytical analysis. Samples for all analytical parameters, except for metals, are required to be cooled to 4ºC ± 2ºC after the sample has been collected into the appropriate container(s). For samples that are received above 6ºC, the laboratory will contact the Advanced GeoServices QA Manager to determine the appropriate corrective action.

An alternate method of measuring the cooler temperature is through the use of an infrared digital thermometer. The sample custodian in this case, opens the cooler, aims the thermometer at the inside of the cooler, pulls the trigger and reads the temperature from the digital display. These kinds of thermometers are not NIST-traceable, but are calibrated by the manufacturer.

2.6.2 Equipment (Rinsate) Blank

Equipment blanks will be collected to ensure that the sampling equipment has been properly decontaminated and that the potential for cross contamination has been minimized by the decontamination procedures. These blanks will be collected by decontaminating the sampling device and pouring ultrapure de-ionized water (provided by the laboratory) over the device and collected into the appropriate sample containers. One equipment blank per day or per 20 samples, whichever is more frequent, will be collected whenever non-dedicated or non-disposable equipment is used. The equipment blanks will be analyzed for identical parameters as the samples.

2.6.3 Trip Blank

Trip blanks are designed to address sample contamination from ambient/environmental sources outside of the Site and laboratory. The trip blank typically three (3) 40-mL VOA vials filled with deionized water. Each grouping of samples submitted to the laboratory for VOC analysis will contain a trip blank.

2.6.4 Field Duplicates

These samples will be collected to allow for the determination of analytical and sampling precision. One field duplicate will be collected for every 20 samples per matrixand submitted for the identical parameters as the true sample. In order to prevent bias, these samples will be submitted blind to the laboratory and will not be identified as field duplicates on the Chain of Custody (CHOC).

2.6.5 Matrix Spike/Matrix Spike Duplicates

These samples will also be submitted as further QC checks. One MSD/MSD sample will be collected for every 20 samples per matrix. These will allow for the accuracy to be determined by the percent recovery of the compounds spiked. The laboratory will perform the spiking of the MS/MSDs. Precision will also be assessed by comparison of the MS/MSD relative percent duplicate results. The purpose of these laboratory spikes is to monitor any possible matrix effects specific to the samples collected from the Site. MS/MSD samples will be identified on the CHOC.

2.7 REPORTING

The groundwater monitoring program will generate data concerning the groundwater quality and hydrogeologic conditions at the Site. The data will be tabulated and transmitted to the USEPA in the Phase III RFI Report after the data have been validated. The form of the evaluation and reporting to be included in the report is detailed in the following outline:

- **Introduction** - including date and wells monitored.

- **Groundwater Sampling and Analysis Procedures** - This section will summarize well purging, sample collection procedures, field parameters, sample preparation and preservation, and decontamination procedures.

- **Groundwater Flow Direction** - Depth-to-water information will be converted to groundwater elevation data using the top-of-casing as the reference point and presented in tabular form. Elevations will be plotted on a Site map to illustrate the potentiometric surface of the groundwater. .

- **Groundwater Quality** - Groundwater quality data will be tabulated, and validated. A brief validation quality report consisting of general information and qualifying statements that describe the usability of the data, difficulties encountered, and qualifier codes defining the reliability of the samples will be presented in data summary tables. Data deliverables from the laboratory will be paginated, and include at a minimum:

  - a page including the facility name and address, laboratory certification number, date of analytical report preparation, and signature of laboratory director or group leader;
  - a listing of the field sample identification number and the corresponding laboratory sample identification numbers;

- a listing of analytical methods used;
- detection limits of each analyte;
- tabulated sample results, including date of analysis;
- method blank results;
- instrument raw data; and,
- chain of custody documents.

Any issues noted in the data validation will be reviewed to determine its impact on the data interpretation. Groundwater analytical data will be compared to historic groundwater data for the Site and the appropriate Pennsylvania water quality criteria.

- **Conclusion and Recommendations** - Will include a statement about the general Site groundwater conditions, comparison of data to the performance standards and recommendations for any follow-up activities.

3.0 SOIL/SEDIMENT SAMPLES

The objective of the soil sampling proposed for the RFI is to supplement the existing limited subsurface information by obtaining samples of the fill/soil materials encountered for chemical analysis. Soil borings will be performed across the Site (random sampling) with respect to inorganic contaminants (RCRA 8 metals) with an emphasis on certain SWMUs (SWMU-2, 3, 5, and 9) and AOCs (focused sampling) where impacts from past operations is possible. Soil sampling for VOCs/SVOCs will be performed for soil samples collected from the proposed groundwater monitoring wells as well as other locations based on PID measurements.

Soil samples will also be collected from surface locations with signs of soil erosion or stormwater runoff. Sediment samples will be collected from accessible stormwater structures.

The following field activities will take place for the soil samples/borings:

- Utilities will be marked in the areas where digging/drilling will take place.
- The boring locations will be staked out in the field using spray paint markings on asphalt or concrete surfaces and modifying the location to avoid utilities.
- If necessary, concrete surfaces will be saw cut or cored prior to drilling.
- Plastic will be used on the ground surface around the borehole to contain the drill cuttings.
- The soil borings will be advanced using direct-push (Geoprobe™) techniques. Soil sampling for monitoring wells will be collected using hollow stem augers. Samples will be obtained using acetate sleeves (direct push) or a split spoon sampler (hollow stem auger) on a continuous basis until the end of the boring or refusal. An experienced field technician working under the supervision of and Indiana Professional Geologist will log the borings, for split spoon sampling the number of blows required to advance the sampler 6 inches will be recorded, and the soil/fill types encountered and the presence of any free water in the subsurface will be

documented. Drill cuttings and excess sample volume will be containerized for proper disposal.

- Representative samples of soils and fill materials encountered will be taken from the acetate sleeve or split spoon sampler and analyzed. Sample homogenization will take place in the laboratory (inorganics only).
- After the soil boring is completed, the hole will be grouted and the surface restored to its original condition.
- Surface soil samples will be collected by hand excavation using shovels or augers.
- Sediment samples will be collected by hand excavation using shovels or trowels.
- Reusable sampling equipment that is contact with soils will be decontaminated between borings using DI water and a scrub brush. The split spoon sampler will be dry brushed to remove visible soil between samples and decontaminated using a water-Alconox solution between boring locations.
- The boring location and ground surface elevation will be surveyed.

3.1 DATA QUALITY OBJECTIVES

Soil borings will be conducted to obtain samples of the soils/fill material encountered for chemical analysis. To meet this goal, the following data quality objectives (DQOs) have been established. To determine the project DQOs, a series of planning steps are used. The DQO development process is used to optimize the data collection necessary to meet the applicable decision criteria as defined in USEPA *Guidance for the Data Quality Objective Process* dated August 2000 and USEPA *Data Quality Objectives Process for Hazardous Waste Site Investigations* dated January 2000. The seven steps of the DQO process are presented below.

3.1.1 State the Problem

There is a potential that soils/fill materials onsite associated with the former manufacturing area (SWMUs and AOCs) and on the overall site outside of the manufacturing areas may contain lead and other heavy metals at elevated concentrations. Previous investigations have provided some indication that these contaminants are only present in shallow fill areas.

Impacts from VOCs/SVOCs requiring further investigation have been observed in the vicinity of the former fuel shed during removal of UST-2. Soils with impacts above the residential screening level were removed during the UST removal, although groundwater impacts from VOC/SVOCs may remain. Soil sampling will be performed to confirm that these contaminants are not present in soil at concentrations above the RCGs.

3.1.2 Identify the Decision

Soil sampling/borings will be conducted to characterize site subsurface conditions. Results of soil sampling will be utilized in conjunction with results from previous environmental investigations to characterize conditions and delineate limits of historic impacts. Depending on the magnitude of those impacts, further evaluation, corrective measures and/or risk evaluation may be necessary.

3.1.3 Identify Inputs to the Decision

Site specific performance standards have not been established for soils or fill materials at the site. Results of soil sampling will be compared to IDEM 2017 OLQ Screening Levels established for soil to determine whether or not further evaluation under the RCRA Corrective Action Process is necessary. The Site is a commercial/industrial property and the screening levels for this use are the most appropriate. These are referred to as the RCGs. VOC results will also be compared to the migration to groundwater screening level.

Pace Analytical is proposed for analysis of soil boring samples. The proposed laboratory participates regularly in performance evaluation audits as part of their laboratory certification requirements. Pace Analytical is a NELAP certified laboratory. USEPA SW-846 methods will be used in the sample analysis of priority pollutant metals; see Table B-3 in the QAPP (Appendix B) for the specific methods.

3.1.4 Define the Study Boundaries

Soil borings using a hollow-stem augur will be completed to depths of up to 18 feet bgs (new monitoring well depth) at eight (8) locations throughout the site. Soil samples will be obtained continuously in the borings by means of the Standard Penetration Test (ASTM D 1586) using split spoon samplers. Samples for laboratory analysis of RCRA 8 Metalswill be collected from intervals of 0-4 feet bgs, 4-8 feet bgs, 8-12 feet bgs, 12-16 feet bgs, and 16-18 feet bgs. In addition, a sample for VOC/SVOC analysis will be collected from the depth with the highest PID reading. If no elevated PID readings are observed the VOC/SVOC sample will be collected from the bottom of the well.

At sixty five (65) locations (R-1 through R-55 and F-1 through F-10) within the Site boundary (see Figure 3 of RFI Work Plan) geoprobe soil borings will be performed to a depth of up to eight feet below the ground surface (top of pavement) to typically collect soil samples from the following intervals: 0-1 feet bgs. 1-2 feet bgs, 3-4 feet bgs, 5-6 feet bgs, and 7-8 feet bgs.

In the vicinity of the former UST-2, six (6) geoprobe soil borings (U-1 through U-6) will be performed to a depth of eight feet bgs to collect one sample each from the 0-4 feet bgs and 4-8 feet bgs intervals. The specific location of the sample will target the highest observed PID measurement and/or areas with staining or odors observed. At any given boring location, the boring depth may be increased if evidence of deeper contamination is observed (discoloration, odors, debris, PID measurements). Soil samples from the UST-2 borings will be analyzed for VOCs/SVOCs and diesel (DRO) and gasoline range (GRO) total petroleum hydrocarbons (TPH).

Soil sampling during the RFI will follow the sampling protocols presented below. See Figure 3 in the RFI Work Plan for the proposed soil boring locations.

3.1.5 Develop a Decision Rule

All surface soil samples, or split spoon and geoprobe samples with sufficient recovery will be logged for soil characteristics. Results of the laboratory analysis will be tabulated and presented in the RFI Report.

3.1.6 Specify Limits of Decision Errors

The sample data are subject to random and systematic errors during field collection and sample analysis. The combination of errors is referred to as "total study error." The two contributors to the total study error are the statistical sampling error and the measurement error. The statistical sampling error occurs when the data collection plan fails to provide a limited variability within the decision unit necessary for accurate decision. Measurement errors are introduced during the processes to which the sample is subjected such as physical collection, sample handling, sample preparation, sample analysis, and data reduction. Since the total study error directly affects the possibility of making a decision error, the total decision error must be managed by minimizing the sample design and measurement errors.

The following are some ways to minimize the statistical sampling error:

- use standardized sampling techniques when performing the soil borings to minimize variations in recovery over the sampling intervals.

The following are some ways to minimize measurement errors:

- have the samplers collect the samples in a standardized manner;

- ensure that each sample is properly preserved, labels, and transported to the laboratory under chain-of-custody;
- specify that the laboratory has to use accepted USEPA Methods and reports the data using the proper units;
- specify that the laboratory participates in regular performance testing and also has NELAP certification;
- receive the analytical data from the laboratory in an electronic format to minimize transcription errors; and,
- perform a qualitative review (data validation) of the analytical data, as specified in the QAPP (see Appendix B), to verify the reliability of the data.

3.1.7 Optimize the Design for Obtaining Data

Upon review of the analytical data, the sampling frequency may be changed to optimize the design. The design options will be evaluated based on cost and the ability to meet the DQOs and presented to the USEPA for approval.

3.2 SAMPLE LOCATION AND FREQUENCY

The proposed soil sample/borings are shown on Figure 3 of the RFI Work Plan. Samples will be collected as described in Section 3.1.4. Physical characteristics of soil samples will be logged on standard geotechnical boring logs. Samples representative of the various soil and fill materials encountered in each sample interval will be sent for laboratory analysis.

3.3 SAMPLE DESIGNATION

Soil boring samples will be identified using the boring identification and depth interval (e.g., R17-3-4, etc.).

Surface samples collected in areas of soil erosion will be identified using a unique descriptor (e.g., surface-0-0.5, etc.)

Any sediment samples collected will be identified using "SED" followed by a unique numeric ID.

3.4 FIELD PROCEDURES

3.4.1 Utility Location and Mark-out

Prior to drilling, Indiana One-Call system will be notified to mark out the underground public utilities in the areas of the study. Coordination of the field work with Exide's caretaker will also be conducted during one site visit with the driller prior to the start of work to locate the borings in the field and to adjust boring locations as needed based upon site restrictions or the presence of utilities. The assistance of the Site caretaker during the site visit will also be requested to identify any private underground utilities/structures in the areas of study. A private utility locating service will also be contracted to identify utilities where test borings are being performed.

3.4.2 Sampling Equipment

The following equipment will be used for the soil boring sampling:

- Concrete coring equipment;
- Plastic sheeting;
- Standard drilling rig with Hollow Stem Augur capable of drilling to depths at least 40 feet;
- Split spoon sample equipment;
- Geoprobe ™ direct push boring equipment (with acetate sleeves) capable of boring to depths of at least 20 feet;
- Photo-ionization Detector (PID);
- En-Core or Terra-Core samplers;

- Electronic depth to water meter; and,
- Laboratory supplied containers for the collection of samples.

The soil boring sampling is comprised of drilling or boring, split spoon or acetate sleeve sample collection, field characterization of physical features, homogenization for metals analysis, and decontamination procedures.

Surface soil and/or sediment samples will be collected using the following equipment:

- Stainless steel hand auger, shovels, and/or trowels;
- Disposable plastic trowels;
- Aluminum pans, plastic bags, or stainless steel mixing bowls for homogenization

3.4.3 Field Measurements

In cases where groundwater is encountered during boring/drilling activities, depth-to-water will be measured in the boring using an electronic water level indicator. The field personnel will measure the water levels in the borings to the nearest 0.01 foot from the ground surface for reference.

3.4.4 Sample Collection Procedures

Soil samples from borings R-1 through R-55, F-1 through F-6, and F-10; as well as surface erosion samples, and any sediment samples will be collected for total RCRA 8 metals. Soil samples from borings R-20, R-21, R-28, R-29, R-30, and R-37 will also be analyzed for pH.

In borings F-7 through F-9, U-1 through U-6, and MW-1 through MW-8 VOC and SVOC analysis will be performed. Samples from borings U-1 through U-6 will also be analyzed for DRO/GRO TPH.

As per QAPP Table B-3 found in Appendix B, the samples will be collected in 4-oz glass jars (or larger) or resealable bags (inorganic analysis) that are unpreserved. VOC samples will be collected using En-Core or Terra Core techniques. The selected analytical laboratory will perform all analyses in accordance with USEPA publication SW-846 methods.

Sample handling will be in accordance with the procedures outlined in Section 4.0. The appropriate methods for extraction and analysis and required holding times to be met are given in the QAPP in Appendix B.

3.4.5 Decontamination and Waste Handling

Sample equipment will have soil particles removed via dry brushing between various sample depths at the same boring location. Split spoons and geoprobe drive shoes will be disassembled and decontaminated between soil boring locations in the following manner. Shovels, hand augers, or other reusable sample equipment will also be decontaminated between sample locations.

- Alconox and water wash;
- Distilled water rinse;
- Distilled water rinse, and,
- Air dry.

Investigation derived waste (IDW) includes decontamination water, soil spoils, trash , and PPE generated during the investigation. Decontamination water will be collected and stored onsite until it can be disposed of offsite. Soil spoils will be collected in a drum and stored onsite until it can be disposed of offsite. Trash and PPE will be placed in plastic trash bags. Disposal of IDW will be performed by Exide.

3.5 QUALITY ASSURANCE/QUALITY CONTROL

The field quality control/quality assurance sample results will be used to assess the variability of the soil sample results at the end of the sampling period when considering whether corrective action is needed.

3.5.1 Temperature Blank

Temperature blanks will be utilized for samples (groups of samples) that are designated for VOC/SVOC analysis, but are not required for soil samples designated for metals analysis.

3.5.2 Equipment (Rinsate) Blank

Equipment blanks will be collected to ensure that the sampling equipment has been properly decontaminated and that the potential for cross contamination has been minimized by the decontamination procedures. These blanks will be collected by decontaminating the sampling device and pouring ultrapure de-ionized water (provided by the laboratory) over the device and collected into the appropriate sample containers. One equipment blank per day or per 20 samples, whichever is more frequent, will be collected whenever non-dedicated or non-disposable equipment is used. The equipment blanks will be analyzed for identical parameters as the samples collected on that day.

3.5.3 Trip Blank

Trip blanks are designed to address sample contamination by VOCs from ambient/environmental sources outside of the Site and laboratory. The trip blank typically three (3) 40-mL VOA vials filled with deionized water. Each grouping of samples submitted to the laboratory for VOC analysis will contain a trip blank.

3.5.4 Field Duplicates

These samples will be collected to allow for the determination of analytical and sampling precision. One field duplicate sample per twenty samples will be split from the parent sample after homogenization and submitted for the identical parameters as the parent sample. In order to prevent bias, these samples will be submitted blind to the laboratory and will not be identified as field duplicates on the Chain of Custody (CHOC).

3.5.5 Matrix Spike/Matrix Spike Duplicates

These samples will also be submitted as further QC checks. These will be collected at the frequency of one MS/MSD pair per twenty samples collected and will be split from the parent sample after homogenization. The MS/MSD samples will allow for the accuracy to be determined by the percent recovery of the compounds spiked. The laboratory will perform the spiking of the MS/MSDs. Precision will also be assessed by comparison of the MS/MSD relative percent duplicate results. The purpose of these laboratory spikes is to monitor any possible matrix effects specific to the samples collected from the Site. MS/MSD samples will be identified on the CHOC.

3.6 REPORTING

The soil boring sampling will generate data concerning the subsurface conditions and extent of fill material at the Site. The data will be tabulated and transmitted to the USEPA in the RFI Report after the data has been validated. The form of the evaluation and reporting to be included in the report is detailed in the following outline:

- **Introduction** - including date, number and depth of borings completed.

- **Soil Boring Sampling and Analysis Procedures** - This section will summarize drilling, sample collection procedures, geotechnical characterization, and decontamination procedures.

- **Subsurface Profile** – Auger/Geoprobe refusal depth information and nature of materials encountered will be converted to elevation data using the ground surface as the reference point. The soil boring information generated in the RFI will be combined with subsurface information from previous investigations conducted at the Facility to gain an understanding of the extent and character of the soil and fill at the site.

- **Soil Analytical** - Soil data will be tabulated, and validated. A brief validation quality report consisting of general information and qualifying statements that describe the usability of the data, difficulties encountered, and qualifier codes defining the qualitative and quantitative reliability of the samples will be presented in data summary tables. Data deliverables from the laboratory will be paginated, and include at a minimum:

  - a page including the facility name and address, laboratory certification number, date of analytical report preparation, and signature of laboratory director or group leader;
  - a listing of the field sample identification number and the corresponding laboratory sample identification numbers;
  - a listing of analytical methods used;
  - detection limits of each analyte;
  - tabulated sample results, including date of analysis;
  - method blank results;
  - instrument raw data; and,
  - chain of custody documents.

  Any issues noted in the data validation will be reviewed to determine its impact on the data interpretation.

- **Conclusion and Recommendations** – This section will include a description of the general Site subsurface soil conditions, and recommendations for follow-up activities, if any.

4.0 SAMPLE HANDLING PROCEDURES

4.1 SAMPLE HANDLING

All sample containers will be identified by the use of sample labels with the sample identification. Each sample label will be filled out by the sampler to avoid any possibility of sample misidentification and attached to the sample container. Indelible ink will be used to complete the sample labels. Each sample label will be labeled at the time of collection with, at a minimum, the following information:

- Sample identification;
- Initials of the sample collector;
- Time and date of the sample collection;
- Site name and location number (if any);
- Requested analyses;
- Any preservative added or field preparation performed; and,
- Sample designation if this sample is a quality assurance sample.

Each member of the sampling team will use a new pair of gloves at each sample location; however, the same pair of gloves can be used when collecting samples for compositing.

The field sampler will maintain custody of the samples following the procedures outlined in the following sections until the samples are properly relinquished to the laboratory or a common carrier for delivery to the laboratory or are archived on-site. Once at the laboratory, each sample will be assigned a unique laboratory identification number that will be used for analysis assignment, sample tracking, and data reporting while the samples are at the laboratory.

4.2 SAMPLE CUSTODY/SAMPLE CONTROL

A sample is physical evidence collected from the Site. Due to the evidentiary nature of the data generated from sampling, sample custody must be traceable from the time the empty sample containers are prepared by the laboratory through the reporting of the results of the analyses. Therefore, sample control procedures have been established to ensure sample integrity. All sample containers and samples will be maintained under strict custody procedures throughout the investigation. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files.

A sample, sample container, or evidence file will be considered under custody if:

- An item is in the actual possession of the person; or
- The item is in the view of the person, after being in actual possession of the person; or
- The item was in the person's actual physical possession but is now locked up or sealed in a tamper-proof manner; or
- The item is placed in a designated secured restricted area.

4.2.1 Field Custody Procedures

The field personnel in charge of collecting the samples will maintain custody of the samples collected. The field personnel will be responsible for documenting each sample transfer and maintaining custody of all samples until they are shipped to the laboratory or archived. The appropriate sample containers, preservatives, methods for extraction and analysis and required holding times to be met are given in the QAPP in Appendix B.

4.2.2 Field Data Documentation/Field Logs

A system of logging all pertinent data collected during sampling operations will be maintained using a dedicated field logbook(s). Each page will be numbered, dated and initialed by the person making the entry. All entries will be made in indelible ink. Incorrect entries will be crossed out with a single line and verified with the recorder's initials. At the completion of the day, if a page is not complete, a diagonal line will be drawn through the remainder of the page with the recorder's signature at the bottom.

All sample locations will be recorded in the field logbook and referenced to the site map so that each location is permanently established. Samples will be tagged with all pertinent site information at the time of sampling. Pertinent site information to be supplied in the field logbook for each task is listed below:

- Signature of recorder;
- Name and location of sample;
- Date and time of arrival and departure;
- Names of all personnel on-site and their affiliation;
- Purpose of the visit/description of the field activity;
- All field instruments used, date and time of calibration and calibration checks, method of calibration, and standards used;
- All field measurement results;
- Date, time, and location of all sampling;
- Method of sample collection;
- Any factors which could affect sample integrity;
- Name of sampler(s);
- Sample identification, sample description, and sample preservation, if any;
- Documentation of all conversations with the client, agency personnel, field decisions and approval; and,
- Weather conditions.

Field logbooks should contain only factual information entered as real-time notes, which will enable the user to recreate events on-site. In addition, chain-of-custody records will be prepared and kept as part of the field records.

4.2.3 Chain-of-Custody Procedures

The following chain-of-custody procedures will be used for this project:

- New, certified clean sample containers will be prepared and relinquished by the laboratory on a chain-of-custody record. The chain-of-custody record will be kept as part of the permanent record.
- Any transfer of custody of containers of samples will be noted on a chain-of-custody record.
- Each sample collected for the event will be entered on the chain-of-custody record.
- The chain-of-custody will be completed as soon as possible after sample collection. The following information must be supplied to complete the chain-of-custody record.

  - Site specific project name and number;
  - Signature of sampler(s);
  - For each sample, sampling station number, date and time (military is preferred) of collection, grab or composite sample designation, and a brief description of the type of sample and sampling location;
  - Number of sample containers per each sample location;
  - Analysis required;
  - Type of preservative;
  - Signatures of individuals involved in sample transfer (i.e., relinquishing and accepting samples). Individuals receiving the samples shall sign, date, and note the time they received the samples/cooler on the record; and,
  - Type of carrier service.

The original chain-of-custody record will accompany the sample containers during transport to document their custody.

If custody is relinquished through a common carrier for delivery to the laboratory, the following protocol will be followed:

- In the space for the sample receiver, the name of the common carrier and the date relinquished will be written. In addition, if known, the tracking number will be included on the chain-of-custody record;
- The original completed chain-of-custody record will be placed inside the shipping package; and,
- The shipping package will be sealed with tape and custody seals affixed. The seals will be placed on the package in such a manner that the package cannot be opened without breaking the seals. The seals will serve to document that the shipping container was not opened during the shipment through the common parcel carrier.

4.2.4 Sample Shipment Procedures

Prior to expiration of holding times for any parameters, all samples scheduled for analysis will be packaged in shipping containers for shipment to the analytical laboratory using the following steps:

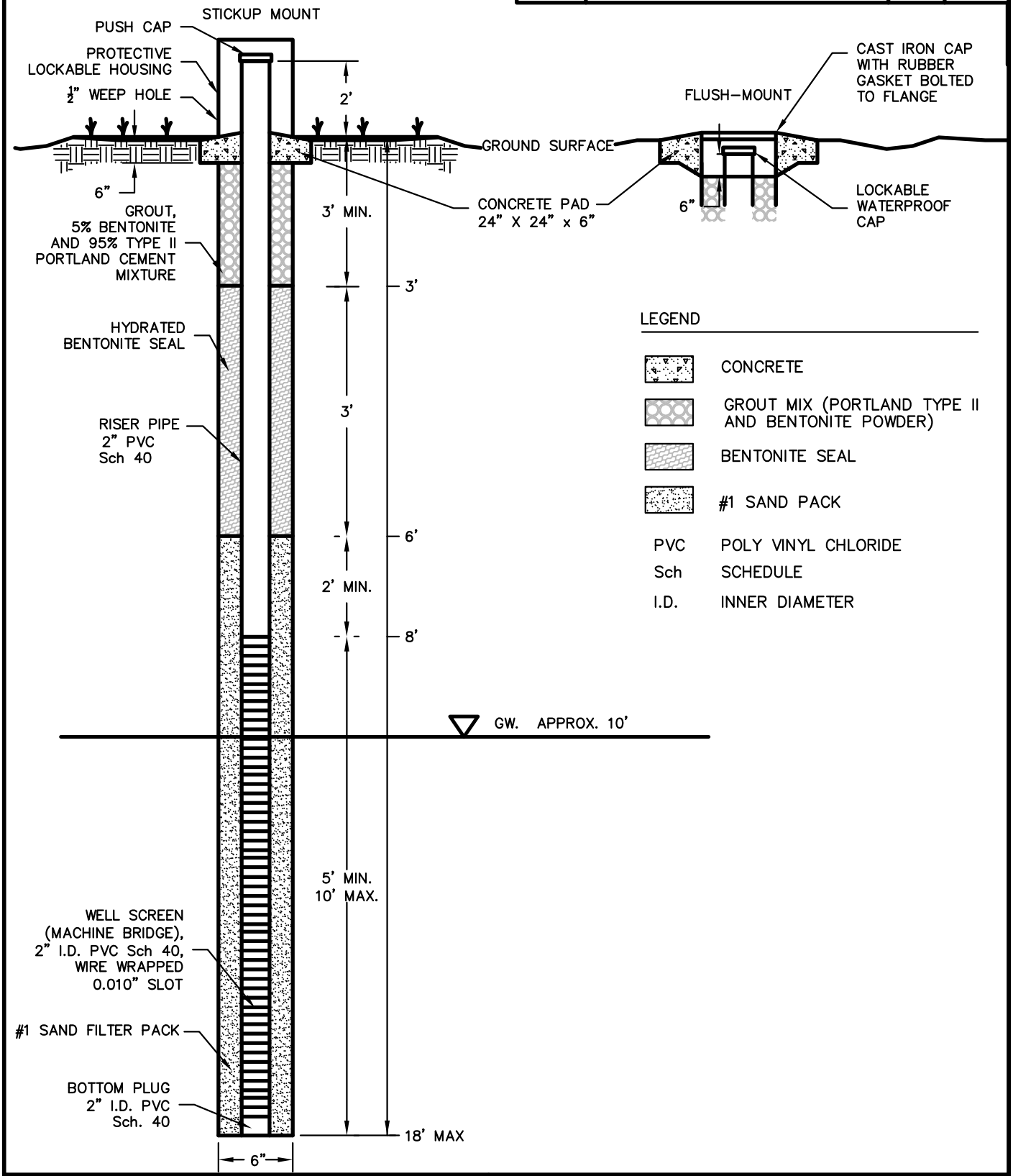
1. Check each sample container for a properly completed sample identification label.
2. Ship the samples in a large capacity cooler, or specific laboratory prepared sample-shipping container. Place packing material on the bottom of the cooler to prevent sample bottle breakage.
3. Place the sample bottles in the shipping container in a manner such that they do not touch and will not touch during shipment. Secure with packing material as needed to fill void space.
4. A cooler with samples to be analyzed for metals only do not need to be cooled (iced) during shipping.

5. Place the original chain-of-custody record in a plastic bag, seal, and tape it to the inside of the shipping container lid.
6. Retain the pink copy of the chain-of-custody for the Advanced GeoServices QA Manager.
7. Tape the cooler drain shut. Tape the cooler or shipping container closed at a minimum of two locations.
8. Place two signed and dated custody seals across each edge of the shipping container.
9. Attach the completed shipping label to the top of the shipping container.
10. Relinquish the cooler to the courier with the required signed and dated handbill/waybill.
11. Retain receipt of the handbill/waybill as part of the permanent documentation.

If the sample coolers are not shipped but instead picked-up by the laboratory courier, step number 6 and 12 will be omitted and the chain-of-custody will be handed to and signed by the laboratory courier. The pink copy of the chain-of-custody will be maintained by the sampler and submitted to the Advanced GeoServices QA Manager.

FIGURE

Revision	Description	Date	By



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MONITORING WELL CONSTRUCTION DETAILS
RFI SAP
EXIDE TECHNOLOGIES
FRANKFORT, INDIANA

PROJECT ENGINEER:	P.G.S.	SCALE:	N.T.S.
CHECKED BY:	P.G.S.	PROJECT NUMBER:	2011-2678-11
DRAWN BY:	J.S.D.	DATE:	FIGURE: A-1

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APPENDIX B

Quality Assurance Project Plan

QUALITY ASSURANCE PROJECT PLAN
FOR THE
RCRA FACILTY INVESTIGATION
EXIDE TECHNOLOGIES FORMER MANUFACTURING FACILITY
Frankfort, Indiana

Prepared for:

EXIDE TECHNOLOGIES
Milton, Georgia

Prepared by:

ADVANCED GEOSERVICES
West Chester, Pennsylvania

Project No. 2011-2678-11
February 21, 2018

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West Chester, Pennsylvania

Project No. 2011-2678-11
February 21, 2018

USEPA Peter Ramanauskas, Project Manager	**Date**
USEPA Zachary Sasnow, QA Contact	**Date**
Advanced GeoServices, Paul G. Stratman, P.E., Project Manager	2/20/18 **Date**
Advanced GeoServices, Amy Graham, QA Scientist	2/21/2018 **Date**

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ATTACHMENT

Exide Technologies
Frankfort, Indiana
February 21, 2018

1.0 PROJECT MANAGEMENT

1.1 PROJECT/TASK ORGANIZATION

This Quality Assurance Project Plan (QAPP) has been developed to present the quality assurance measures that will be used during the RFI field activities at the Exide Technologies' former manufacturing facility property located at 555 North Hoke Avenue in Frankfort, Indiana (the Site). The QAPP has been prepared based on guidance presented in the "United States Environmental Protection Agency (USEPA) Requirements for Quality Assurance Project Plans," (QA/R-5, EPA/240/B-01/003, March 2001) and the "Guidance on Systematic Planning Using the Data Quality Objective Process" (QA/G-4, EPA/240/B-06/001, February 2006).

While all personnel involved in an investigation and in the generation of data are implicitly a part of the overall project and quality assurance program, certain individuals have specifically delegated responsibilities. The RFI supervising professional firm is Advanced GeoServices Corp (Advanced GeoServices). The Advanced GeoServices personnel with quality assurance/quality control (QA/QC) responsibilities are the Project Coordinator, QA Manager, QA Scientist, and the field technicians. The Advanced GeoServices organizational chart is included in Attachment B-2. For samples collected by Advanced GeoServices personnel, the analyses of the samples will be performed by Pace Analytical located in Indianapolis, Indiana. The laboratory retains the responsibility for analytical data quality assurance, however; specific laboratory personnel with QA/QC responsibilities include the Laboratory QA Officer and Laboratory Sample Custodian.

1.1.1 Project Coordinator

The Project Coordinator is an experienced manager and technical professional who assists in the coordination of the RFI, participates in major meetings and regulatory negotiations and provides upper level contact for the client. The designated Project Coordinator is Paul Stratman, P.E, P.G.

1.1.2 QA Manager

The QA Manager will work on all projects requiring the collection of data, and as such is not directly involved in the routine performance of the technical aspects of the investigations including sample collection and utilizing the data outside of the data processing. The QA Manager's responsibilities include the development, evaluation, and implementation of the QAPP and procedures appropriate to the investigation. Additional responsibilities include reviewing project plans and revising the plans to ensure proper QA is maintained. The QA Manager is also responsible for all data processing activities, data processing QC, and final analytical data quality review.

It is a primary responsibility of the QA Manager to ensure that all personnel have a good understanding of the QAPP, and understanding of their respective roles relative to one another, and an appreciation of the importance of the roles to the overall success of the program.

1.1.3 QA Scientist

The QA Scientist has primary responsibility for analytical data validation and review. In this capacity, the QA Scientist will prepare data validation reports describing data usability and analytical QC problems encountered.

1.1.4 Field Technicians

Experienced Advanced GeoServices Field Technicians will conduct all sampling tasks to be conducted by Advanced GeoServices. One of the Field Technicians will be designated as the Field Team Leader. Their responsibilities will include the documentation of the proper sample collection protocols, sample collection, field measurements, calibration of field equipment, equipment decontamination, and logbook and CHOC documentation.

1.1.5 Analytical Laboratory QA Officer

The QA Officer has the responsibility for maintenance of all laboratory QA activities and documentation. The role and responsibilities of the laboratory's QA Manager has been included as part of the laboratory quality assurance manual (QAM). The QAM and the Standard Operation Procedures (SOP) from Pace Analytical, Indianapolis have been included in an electronic format as Attachment B-1

1.1.6 Analytical Laboratory Sample Custodian

The Sample Custodian's responsibilities include ensuring proper sample entry and sample handling procedures by laboratory personnel.

1.2 QUALITY OBJECTIVES AND CRITERIA

Data quality objectives (DQOs) have been established as described in the sampling and analysis plan. DQOs are qualitative and quantitative statements specifying the quality of the environmental data required to support the decision making process. Separate DQOs are designed for field sampling and laboratory analysis so that clear distinctions can be isolated with respect to cause between any problems found in the system. Conversely, the DQOs are also designed to provide an indication in the form of data quality indicators (DQIs) of the variability of the overall system. The overall QA objective is to keep the total uncertainty within an acceptable range that will not hinder the intended use of the data. To achieve this, specific data requirements are required such as precision, accuracy, sample representativeness, data comparability, data completeness, and sensitivity.

Precision

Precision measures the reproducibility of data or measurements under specific conditions. Precision is a quantitative measure of the variability of a group of data compared to their average value. Precision also characterizes the natural variation of the matrix and how the contamination exists or varies within that matrix. Precision is measured by comparison of the field duplicate with the original parent sample and by comparison of the specific compound recoveries for the matrix spike/matrix spike duplicate samples. For this project, the DQO for precision is to meet the performance criteria listed with the method as shown in Table B-1.

Accuracy

Accuracy is defined as the degree of agreement of a measurement or an average of measurements with an accepted reference value. Accuracy measures the bias in a measurement system which may result from sampling or analytical error. Sources of error that may contribute to poor accuracy are laboratory error, sampling inconsistency, field and/or laboratory contamination, sample handling, matrix interference, and sample preservation. The field component of accuracy will be reduced if the sampling, preservation and handling techniques described in the SAP and this QAPP are followed. Accuracy in the laboratory methods and procedures will be evaluated through the use of matrix spike and matrix spike duplicate samples. For this project the DQI for accuracy is to meet the performance criteria listed within the method.

Representativeness

Representativeness expresses the degree to which sample data represents the characteristics of the environment from which they are collected. Samples that are considered representative are properly collected to accurately characterize the contamination at a sample location. Therefore,

the samples will be collected in a standardized method. Representativeness will be measured by a review of the precision obtained from the field and laboratory duplicate samples.

Comparability

Comparability expresses the confidence with which one data set can be compared with another data set from a different phase or from a different program. Data comparability will be ensured by the control of sample collection methodology, analytical methodology, and data reporting.

Completeness

Completeness is defined as the percentage of data that is judged to be valid to achieve the objectives of the investigation compared to the total amount of data. Deficiencies in the data may be due to sampling techniques, or poor accuracy, precision and laboratory error. While deficiencies may affect certain aspects of the data, usable data may still be extracted from applicable samples. The level of completeness, with respect to usable data, will be measured during the data assessment process by comparing the total number of data points to the number of data points determined to be usable. A completeness of 90% or greater of unrejected analytical data is the objective for the project.

Sensitivity

Sensitivity is used broadly to describe the instrument detection/method detection/reporting limits established to meet project-specific DQIs. Limits have been established to describe project sensitivity requirements, such as method detection limits (MDLs) and reporting limits (RLs). All off-site laboratory analysis will be performed using USEPA methods. Table B-2A and Table B-2B contains the laboratory reporting limits and method detection limits for the analytical

parameters. Specific reporting limits are highly matrix-dependent and may not always be achievable.

1.2.1 Project Data Quality Objectives

To determine the project DQOs, a series of planning steps are used. The DQO development process is used to optimize the data collection necessary to meet the applicable decision criteria as defined in USEPA *Guidance on Systematic Planning Using the Data Quality Objective Process* dated February 2006 and USEPA *Data Quality Objectives Process for Hazardous Waste Site Investigations* dated January 2000. The seven steps of the DQO process for each sampling activity have been integrated into the individual sampling plans.

To achieve the DQOs, this QAPP is designed to ensure that a sufficient number of samples will be collected using technically valid, scientific procedures. The DQOs for this project require qualitative validation. Substantiating the results of the data provides the level of confidence in the data necessary so decisions can be made regarding the monitoring activities and long-term concerns.

1.2.2 Field Investigation Quality Objectives

The main field investigation DQO is to collect high quality data using the proper collection techniques in a repeatable and consistent manner. The SAP discusses the boundaries and the decision rule for each sampling activity. To reduce the random and systematic errors that are introduced in the measurement process during physical sample collection, sample handling, and sample analysis, field duplicates, equipment blanks, and matrix spike/matrix spike duplicate (MS/MSD) samples will be collected.

- Field duplicates are independent samples collected in such a manner that they are equally representative of the sampling point and parameters of interest at a given point in space and time. Field duplicate samples provide precision information of homogeneity, handling, shipping, storage, preparation, and analysis. One field duplicate will be collected for every twenty (20) samples per matrix.

- Trip blanks serve to detect possible cross-contamination of aqueous samples by volatile organic compounds (VOCs) resulting from handling, storage and shipment procedures. Trip Blanks will be submitted for VOC analysis whenever VOC samples are collected at a rate of one per shipment.

- Equipment blanks are designed to address cross-contamination between sample sources in the field due to deficient equipment decontamination procedures. This blank also addresses field preservation procedures, environmental Site interferences, and the integrity of the source water for field decontamination/cleaning. The equipment blanks will be analyzed for identical parameters as the samples. One equipment blank will be collected per day per matrix or per 20 samples per matrix per day (whichever is more frequent) when sampling equipment is decontaminated.

- The MS/MSD samples monitor any possible matrix effects specific to samples collected from the Site. In addition, the analysis of MS/MSD pairs checks precision by comparison of the two spike recoveries Precision will also be assessed by comparison of the MS/MSD relative percent duplicate results. One MS/MSD pair will be collected for every twenty (20) samples collected per matrix.

The acceptance criteria for the above QA/QC samples are presented in Table B-1.

1.2.3 Laboratory Data Quality Objectives

The "ideal objective" for the laboratory is to analyze the samples by the most appropriate analytical method to meet the cleanup criteria, which in turn will provide the highest quality data possible. However, it is not probable that the analyses will be performed flawlessly and without any need to re-extract or re-analyze samples due to necessary dilutions, sample matrix, poor surrogate recoveries, analyst error, etc. To reduce the random and systematic errors that are introduced in the measurement process during sample handling, sample preparation, sample analysis, and data reduction; method blanks, laboratory control samples (LCSs), MS/MSD, and laboratory duplicate samples will be included during preparation and analysis of the Site samples.

- Method blanks are generated within the laboratory during the processing of the actual samples. These blanks will be prepared using the same reagents, procedures, and at the same time as the project samples are being analyzed. If contamination is found in the method blank, it indicates that similar contamination found in associated samples may have been introduced in the laboratory and may not have been present in the samples themselves. Guidelines for accepting or rejecting data based on the level of contamination found in the method blank are presented in the specified analytical method. A minimum of one method blank per twenty (20) samples will be analyzed or, in the event that a quality control batch consists of less than twenty (20) samples, one method blank sample will be analyzed per batch.

- MS/MSD samples determine the accuracy by the recovery rates of the compounds added by the laboratory (the spiking compounds are defined in the analytical methods). The MS/MSD samples also monitor any possible matrix effects specific to samples collected from the Site and the extraction/digestion efficiency. In addition, the analysis of MS/MSD samples check precision by comparison of the two spike recoveries.

- The laboratory control sample (LCS) is prepared by the laboratory by adding analytes of known concentrations to solution (de-ionized water for metals) for the selected analyses. The LCS is prepared, analyzed, and reported once per quality control batch. The LCS must be prepared and analyzed concurrently with the samples in the batch using the same instrumentation as the samples. The LCS is designed to access the capability of the laboratory to perform the analytical methods. If the analytes present in the LCS are not recovered within the criteria defined in the specified analytical methods, the samples will be re-analyzed or the laboratory will qualify the data.

The acceptance criteria for the laboratory QC checks are presented in Table B-1.

1.2.4 Criteria Objectives

Criteria objectives are presented in Table B2. The table also contains the laboratory reporting limits (RLs) for organics and method detection limits (MDLs) for inorganics to show that the analytical methods selected are below or meet the criteria objectives. The laboratory will be expected to report the RLs or MDLs for all samples in the appropriate statistical reporting units for all analytes.

1.2.5 Data Management Objectives

It is a data management objective that all aspects of the investigation from the sample design, collections, shipment, analysis use/decisions, etc. be performed in conjunction with rigorous QA/QC documentation. The specific details of this documentation can be found throughout this document.

It is expected that by the design of separate data quality requirements for field sampling and laboratory analysis, clear distinctions can be made such that any problems found in the system can be isolated with respect to the cause. Conversely, the data quality requirements are also designed to provide an indication of the variability inherent to the overall system. The overall data management objective is to provide a complete database with a high degree of confidence through the use of a phased approach of sampling, analysis, data assessment (data review), data qualification, and feedback.

1.3 SPECIAL TRAINING/CERTIFICATION

One or more technicians, with at least one of the technicians having at least two (2) years of field sampling experience, will perform the field sampling. A Senior Technician will be matched to the project based on the field sampling being performed and the sampling-specific experience level of the technician(s) performing the field sampling.

The training and/or certification for the laboratory personnel are presented in the laboratory QAMs (Attachment B-1).

Data Validation will be performed by a trained QA Scientist. The QA Scientist will have experience validating inorganic and organic laboratory data packages according to Federal guidelines outlined in Section 4.2

1.4 DOCUMENTS AND RECORDS

The documentation of sample collection will include the use of bound field logbooks in which all information on sample collection and field instrument calibration will be entered in indelible ink. Appropriate information will be entered to reconstruct the sampling event, including Site name (top of each page), sample identification, brief description of the sample, date and time of

collection, sampling methodology, field measurements and observations, and sampler's initials (bottom of page with date).

The following documents will be collected and filed: logbooks, field data records, correspondence, chain-of-custody records, analytical reports, data packages, photographs, computer disks, and reports.

2.0 DATA GENERATION AND ACQUISITION

2.1 ANALYTICAL METHODS

The samples should be analyzed using the most current USEPA SW-846 Methods. The appropriate methods for extraction and analysis and required holding times to be met are given in Table B-3. These methods are the most appropriate to achieve all DQOs. Laboratory SOPs for the methods to be used are included in Attachment B-1.

2.2 QUALITY CONTROL

Quality control checks for the analytical laboratory and the field are presented below.

2.2.1 Laboratory Internal QC Checks

Laboratory QC Checks are presented in the laboratory QAM (Attachment B-1). These will be a continuation of the field QC checks presented below. Some of the laboratory internal QC checks include the use of method blanks, surrogates, internal standard compounds, laboratory control samples, laboratory duplicates, interference check samples, and serial dilutions.

2.2.2 Field QC Checks

The specific field QC Checks that will be utilized during this investigation have been included in each individual sampling and analysis plan section. The frequency of collection for the field QC samples has also been included in each individual sampling and analysis plan section.

2.3 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

2.3.1 Laboratory Maintenance

Laboratory preventative maintenance programs and requirements are described in the laboratory QAM (Attachment B-1).

2.3.2 Field Maintenance

The routine daily maintenance procedures conducted in the field have been included for each planning sampling activity and can be found in the SAP.

2.4 INSTRUMENT EQUIPMENT CALIBRATION AND FREQUENCY

2.4.1 Laboratory Calibration

Laboratory calibration and frequency is specified in the USEPA SW-846 Methods for both inorganic and organic parameters and is summarized in the laboratory QAM (Attachment B-1).

2.4.2 Field Calibration

In addition to the laboratory analyses being conducted, field measurements will also be taken. Field personnel will be responsible for making sure the field equipment is properly calibrated prior to use. Any calibrations performed in the field will be recorded directly into the field logbook. If equipment fails calibration or equipment malfunction is noted during calibration or use, the equipment will be tagged and removed from service by the field technician.

The specific field calibration procedures conducted in the field have been included for each sampling activity and can be found in the SAP if calibration of a field meter/instrument is required.

2.5 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Supplies and consumables are inspected upon receipt by the field technician to check for damage during shipment to confirm that no potential cross contamination occurred, and to confirm the items ordered were shipped. Spare and replacement parts stored at the Advanced GeoServices office equipment room; on-Site; and/or in the Advanced GeoServices company vehicle(s) to minimize downtime include the following:

- Appropriately sized batteries;
- Locks;
- Extra sample containers and appropriate preservatives;
- Extra sample coolers, packing material, and sample location flags/stakes;
- Additional supply of health and safety equipment, i.e., gloves, hardhats, safety glasses, etc.; and,
- Additional equipment as necessary for the field tasks.

2.6 NON-DIRECT MEASUREMENTS

There will be no non-direct measurements.

2.7 DATA MANAGEMENT

All field data is documented in bound, pre-numbered logbooks. Once a logbook has been filled, the logbook is returned to the office and filed in the project files. Samples collected are documented in the logbook and on the CHOC record for submittal to the laboratory on a standard laboratory turn around time (2-day turn-around-time may be requested on some VOC/SVOC samples). Pace Analytical, Inc standard turn around time consists of ten working days.

All laboratory data is submitted to Advanced GeoServices in both an electronic report and as an electronic data deliverable (EDD).

A copy of the CHOC is used to hand enter the sample identification into the database. All hand entries are 100% checked and validated by a designated individual that did not enter the CHOC originally. The EDD is used to enter the analytical results into the database. No modifications of the data are made to the EDD. Once the field data and laboratory data have been entered into the database, tables are made for use in the validation of the data. Through the process of data validation, the tables are checked to the laboratory report to confirm that the EDD was accurately reported by the laboratory.

Any qualifiers assigned during the validation are entered by the QA Scientist and 100% checked by a designated individual who did not enter the qualifiers originally.

3.0 ASSESSMENT AND OVERSIGHT

3.1 LABORATORY ASSESSMENTS AND RESPONSE ACTIONS

The purpose of a QA assessment or audit is to provide an objective, independent assessment of a measurement effort. The QA audit determines whether the laboratory's data generating, data gathering, and measurement activities produce reliable and valid results. There are two forms of QA audits: performance evaluation audits and system audits.

3.1.1 Performance Evaluation Audits

The purpose of performance evaluation audits is to quantitatively measure the quality of the data. These audits provide a direct evaluation of the various measurement systems' capabilities to generate quality data.

The laboratory regularly participate in performance evaluation audits as part of their laboratory certification efforts. Performance audits are conducted by introducing control samples in addition to those routinely used. The results for the performance audits are summarized and maintained by the Laboratory QA Supervisor and distributed to the Section Supervisors who must investigate and respond to any out of control results.

Pace Analytical is a NELAP accredited laboratory.

3.1.2 Technical System Audits

A technical system audit is an on-site, qualitative review of various aspects of a total sampling and/or analytical system. The purpose of the technical audit is to assess the overall effectiveness, through an objective evaluation, of a set of interactive systems with respect to strength, deficiencies, and potential areas of concern. Typically, the audit consists of observations and documentation of all aspects of sample analyses. External and internal audits are conducted of the

laboratory throughout each year. Corrective actions for the laboratory are presented in their QAM. The Section Supervisors will provide documentation as to what, if any, corrective actions were initiated concerning external and/or internal audits and are reported to the Laboratory QA Supervisor.

4.0 DATA VALIDATION AND USABILITY

4.1 DATA REVIEW, VERIFICATION, AND VALIDATION

Data validation practices will be followed to ensure that raw data are not altered and that an audit trail is developed for those data which required reduction. All field data, such as those generated during field measurements, observations, and field instrument calibrations, will be entered directly into a bound field logbook.

Upon receipt of the final laboratory report, the laboratory data will be qualitatively validated by the QA Scientist.

Analytical data for soil/solid matrices will be reported as micrograms per kilogram (µg/kg) or milligrams per kilogram (mg/kg); for aqueous matrices will be reported as microgram per liter (µg/L) or milligram per liter (mg/L). Sample concentrations detected between the reporting limit and the method detection limit will be reported by the laboratory and flagged as estimated with a "J" qualifier. Data packages associated with the analyses of samples collected during the investigation will be prepared utilizing USEPA Contract Laboratory Program (CLP) similar deliverable formats.

All raw field data will be summarized, reduced, or tabulated for use in the investigation reports. All laboratory analytical data will be summarized and tabulated upon receipt, validated, and qualified (see Section 4.2) and the final data submitted to the project team for use in the investigation reports.

The following documents will be collected and filed: logbooks, field data records, correspondence, CHOC records, analytical reports, data packages, photographs, computer disks/CDs, and reports.

To maintain control in the transfer of data, all copies of raw data from the field notebooks and the data as received from the laboratory, will be entered into a data file and assigned an appropriate document control identification number. The data file will serve as the ultimate archive for all information and data generated during this investigation. All data files are stored offsite electronically until the completion of the project. At the completion of the project, the data files are updated to a satellite office to be archived indefinitely.

The documentation of sample collection will include the use of bound field logbooks in which all information on sample collection and field instrument calibration will be entered in indelible ink. Appropriate information will be entered to reconstruct the sampling event, including the Site name (top of each page), sample identification, brief description of the sample, date and time of collection, sampling methodology, field measurements and observations, and sampler's initials (bottom of each page with date).

4.2 VERIFICATION AND VALIDATION METHODS

All analytical data generated during the investigation will undergo data review. This review will be performed in accordance with the USEPA *National Functional Guidelines for Organic Superfund Methods Data Review, January 2017* and USEPA *National Functional Guidelines for Inorganic Superfund Methods Data Review, January 2017.*

A preliminary review will be performed on all analysis to verify the necessary paperwork (chain-of-custodies, traffic reports, analytical reports, laboratory personnel signatures) and deliverables are present.

A detailed QA review will be performed on the soil borings data and groundwater analyses by the QA Scientist to verify the reliability of the data as it is presented. This review will include a detailed review and interpretation of all data generated by the laboratory. The primary tools which will be used by the experienced QA Scientist will be guidance documents, established (contractual) criteria, and professional judgment. The items that will be examined during the detailed QA

review of organic and/or inorganic data will consist of: holding times; sample receipt condition, preservation, and cooler temperature; initial calibration; initial calibration verification; continuing calibration; CRDL standards; blanks (method, preparation, trip, initial, continuing, and equipment); GC/MS tune summaries; ICP interference check samples; surrogate recoveries; laboratory duplicates; field duplicates; internal standards; MS/MSD pairs; laboratory control samples; serial dilutions; GC/ECD instrument performance checks; and GC/ECD and GC/MS target compound identification; and overall system performance.

Based upon the detailed review of the analytical data a data validation report will be prepared which will state in a technical, yet "user-friendly" fashion the qualitative and quantitative reliability of the analytical data. The report will consist of a general introduction section, followed by qualifying statements that should be taken into consideration for the analytical results to best be utilized. Based upon the QA review, qualifier codes will be placed next to specific sample results on the sample data tables, if necessary. These qualifier codes will serve as an indication of the qualitative and quantitative reliability of the data. Common qualifier codes include:

- "U" – the analyte was not detected at or above the reporting limit;
- "J" – the analyte was positively identified and detected; however, the concentration is an estimated value because the result is less than the reporting limit or quality control criteria were not met;
- "UJ" – the analyte was not detected, the associated reporting limit is an estimated value;
- "R" – data are rejected due to significant exceedance of quality control criteria. The analyte may or may not be present. Additional sampling and analysis are required to determine;

The data tables and data validation reports will be signed and dated by the QA Scientist.

4.3 RECONCILIATION WITH USER REQUIREMENTS

Completeness will be calculated to reconcile the useable validated data to the entire data set. A completeness of 90% or greater of unrejected analytical data is the objective for the project. Specific reconciliation with the user requirements has been included in their respective sampling sections of the SAP.

TABLES

TABLE B-1
FIELD AND LABORATORY ACCEPTANCE CRITERIA
Exide Technologies
Frankfort, Indiana

ANALYSIS	Field Criteria				Laboratory Criteria					
	Trip Blank	Equipment Blank	Field Duplicate RPD (Aqueous)	Field Duplicate RPD (Soil)	Laboratory Blank	MS/MSD %R	MS/MSD RPD *	Laboratory Duplicate	Surrogate %R	LCS %R
Gasoline Range Organics (8015D)	NA	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	46-134% aqueous 17-153% soil	20%	NA	68-136% aqueous 29-151% soil	65-133% aqueous 68-122% soil
Diesel Range Organics (8015D)	NA	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	37-101% aqueous 10-175% soil	20%	NA	35-99% aqueous 10-162% soil	35-99% aqueous 68-122% soil
Volatile Organic Compounds (8260B)	< MDL	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	Vary	20%	NA	Vary	Vary
Semivolatile Organic Compounds (8270C/8270C SIM)	NA	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	Vary	20%	NA	Vary	Vary
Inorganics (6010B)	NA	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	75-125%	20%	NA	NA	80-120%
Mercury (7471/7470A)	NA	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	75-125%	20%	NA	NA	80-120%
pH (9045C)	NA	NA	NA	<40%	NA	NA	2%	2%	NA	NA

RL - Reporting Limit
NA - Not applicable
%R - Percent recovery
RPD - Relative percent difference
LCS - Laboratory control sample

The MS/MSD, LCS, and Surrogate percent recoveries are statistically calculated and are subject to change.

The semivolatile and volatile laboratory criteria vary for the indivdual parameters and surrogates recoveries based on in house control limits and method criteria.

TABLE B-2A
Groundwater reporting limits and minimum detection limits versus 2017 Remediation Closure Guide Criteria
Exide Technologies
Frankfort, Indiana

Analysis	Units	CAS Number	2017 RCG Ground Water Tap Limit	Method	RL	MDL
Volatile Organic Compounds						
Acetone	ug/L	67-64-1	14000	8260C	100	50
Benzene	ug/L	71-43-2	5	8260C	5.0	1.0
Bromodichloromethane	ug/L	75-27-4	80	8260C	5.0	2.5
Bromoform	ug/L	75-25-2	80	8260C	5.0	2.5
Bromomethane	ug/L	74-83-9	7.5	8260C	5.0	3.9
Bromochloromethane	ug/L	74-97-5	83	8260C	5.0	2.5
2-Butanone (MEK)	ug/L	78-93-3	5600	8260C	25	12
Carbon disulfide	ug/L	75-15-0	810	8260C	10	5.0
Carbon tetrachloride	ug/L	56-23-5	5	8260C	5.0	2.5
Chlorobenzene	ug/L	108-90-7	100	8260C	5.0	2.5
Chloroethane (Ethyl Chloride)	ug/L	75-00-3	21000	8260C	5.0	2.5
Chloroform	ug/L	67-66-3	80	8260C	5.0	2.5
Chloromethane	ug/L	74-87-3	190	8260C	5.0	2.5
Cyclohexane	ug/L	110-82-7	13000	8260C	100	50
Dibromochloromethane	ug/L	124-48-1	80	8260C	5.0	2.5
1,2-Dibromoethane (EDB)	ug/L	106-93-4	0.05	8260C	5.0	2.5
1,2-Dibromo-3-chloropropane (DBCP)	ug/L	96-12-8	0.2	8260C	10	5.0
1,2-Dichlorobenzene	ug/L	95-50-1	600	8260C	5.0	2.5
1,3-Dichlorobenzene	ug/L	541-73-1	NA	8260C	5.0	2.5
1,4-Dichlorobenzene	ug/L	106-46-7	75	8260C	5.0	2.5
Dichlorodifluoromethane	ug/L	75-71-8	200	8260C	5.0	5.0
1,1-Dichloroethane	ug/L	75-34-3	28	8260C	5.0	2.5
1,2-Dichloroethane (EDC)	ug/L	107-06-2	5	8260C	5.0	2.5
1,1-Dichloroethene	ug/L	75-35-4	7	8260C	5.0	2.5
cis -1,2-Dichloroethene	ug/L	156-59-2	70	8260C	5.0	2.5
trans -1,2-Dichloroethene	ug/L	156-60-5	100	8260C	5.0	2.5
1,2-Dichloropropane	ug/L	78-87-5	5	8260C	5.0	2.5
cis -1,3-Dichloropropene	ug/L	10061-01-5	4.7	8260C	5.0	2.5
trans -1,3-Dichloropropene	ug/L	10061-02-6	4.7	8260C	5.0	2.5
Ethylbenzene	ug/L	100-41-4	700	8260C	5.0	2.5
2-Hexanone	ug/L	591-78-6	38	8260C	25	12
lsopropylbenzene (Cumene)	ug/L	98-82-8	450	8260C	5.0	2.5
Methylacetate	ug/L	79-20-9	20000	8260C	50	25
Methylcyclohexane	ug/L	108-87-2	NA	8260C	50	25
Methylene Chloride	ug/L	75-09-2	5	8260C	5.0	3.2
4-Methyl-2-pentanone (MIBK)	ug/L	108-10-1	6300	8260C	25	12
Methyl-tert-butyl-Ether (MTBE)	ug/L	1634-04-4	140	8260C	4.0	2.1
Styrene	ug/L	100-42-5	100	8260C	5.0	2.5
1,1,2,2-Tetrachloroethane	ug/L	79-34-5	0.76	8260C	5.0	2.5
Tetrachloroethene (PCE)	ug/L	127-18-4	5	8260C	5.0	1.2
Toluene	ug/L	108-88-3	1000	8260C	5.0	2.5
1,2,3-Trichlorobenzene	ug/L	87-61-6	7	8260C	5.0	2.5
1,2,4-Trichlorobenzene	ug/L	120-82-1	70	8260C	5.0	2.5
1,1,1-Trichloroethane (TCA)	ug/L	71-55-6	200	8260C	5.0	2.5
1,1,2-Trichloroethane	ug/L	79-00-5	5	8260C	5.0	2.5
1,1,2-Trichloro-1,2,2-trifluoroethane	ug/L	76-13-1	55000	8260C	5.0	2.5
Trichloroethene (TCE)	ug/L	79-01-6	5	8260C	5.0	1.9
Trichlorofluoromethane	ug/L	75-69-4	5200	8260C	5.0	2.5
Vinyl Chloride	ug/L	75-01-4	2	8260C	2.0	2.0
Xylenes, Total	ug/L	1330-20-7	190	8260C	10	5.0
Semivolatiles Organic Compounds						
Acenaphthene	ug/L	83-32-9	530	8270C SIM	1.0	0.012
Acenaphthylene	ug/L	208-96-8	NA	8270C SIM	1.0	0.012

TABLE B-2A
Groundwater reporting limits and minimum detection limits versus 2017 Remediation Closure Guide Criteria
Exide Technologies
Frankfort, Indiana

Analysis	Units	CAS Number	2017 RCG Ground Water Tap Limit	Method	RL	MDL
Acetophenone	ug/L	98-86-2	1900	8270C	10.0	5
Anthracene	ug/L	120-12-7	1800	8270C SIM	0.10	0.021
Atrazine	ug/L	1912-24-9	3	8270C	10.0	5
Benzaldehyde	ug/L	100-52-7	190	8270C	50	25
Benzo[a]anthracene	ug/L	56-55-3	0.12	8270C SIM	0.10	0.023
Benzo[a]pyrene	ug/L	50-32-8	0.2	8270C SIM	0.10	0.013
Benzo[b]fluoranthene	ug/L	205-99-2	0.34	8270C SIM	0.10	0.026
Benzo[g,h,i]perylene	ug/L	191-24-2	NA	8270C SIM	0.10	0.014
Benzo[k]fluoranthene	ug/L	207-08-9	3.4	8270C SIM	0.10	0.018
1,1-Biphenyl	ug/L	92-52-4	0.83	8270C	10.00	5
Bis(2-chloroethoxy) methane	ug/L	111-91-1	59	8270C	10	5.0
Bis(2-chloroethyl) ether	ug/L	111-44-4	0.14	8270C	10	5.0
Bis(2-ethylhexyl) phthalate	ug/L	117-81-7	6	8270C	10	5.0
4-Bromophenyl phenyl ether	ug/L	101-55-03	NA	8270C	10	5.0
Butyl benzyl phthalate	ug/L	85-68-7	160	8270C	10	5.0
Caprolactam	ug/L	105-60-2	9900	8270C	10	5.0
Carbazole	ug/L	86-74-8	NA	8270C	10	5.0
4-Chloroaniline	ug/L	106-47-8	3.7	8270C	10	10
4-Chloro-3-methylphenol	ug/L	59-50-7	1400	8270C	10	7.0
2-Chloronaphthalene	ug/L	91-58-7	750	8270C	10	5.0
2-Chlorophenol	ug/L	95-57-8	91	8270C	10	5.0
4-Chlorophenyl phenyl ether	ug/L	7005-72-3	NA	8270C	10	5.0
Chrysene	ug/L	218-01-9	34	8270C SIM	0.50	0.025
1,4 - Dioxane (p-Dioxane)	ug/L	123-91-1	4.6	8270C SIM	3	0.3
Dibenz[a,h]anthracene	ug/L	53-70-3	0.034	8270C SIM	0.10	0.020
Dibenzofuran	ug/L	132-64-9	7.9	8270C	10	5.0
Di-*n* -butyl phthalate	ug/L	84-74-2	900	8270C	10	5.0
3,3'-Dichlorobenzidine	ug/L	91-94-1	1.3	8270C	20	10
2,4-Dichlorophenol	ug/L	120-83-2	46	8270C	10	5.0
Diethyl phthalate	ug/L	84-66-2	15000	8270C	10	5.0
2,4-Dimethylphenol	ug/L	105-67-9	360	8270C	10	5.0
Dimethylphthalate	ug/L	131-11-3	NA	8270C	10	5.0
4,6-Dinitro-2-methylphenol (4,6-Dinitro-o-cresol)	ug/L	534-52-1	1.5	8270C	50	25
2,4-Dinitrophenol	ug/L	51-28-5	39b	8270C	50	25
2,4-Dinitrotoluene	ug/L	121-14-2	2.4	8270C	10	5.0
2,6-Dinitrotoluene	ug/L	606-20-2	0.49	8270C	10	5.0
Di-*n* -octyl phthalate	ug/L	117-84-0	200	8270C	10	5.0
Fluoranthene	ug/L	206-44-0	800	8270C SIM	1.0	0.022
Fluorene	ug/L	86-73-7	290	8270C SIM	1.0	0.022
Hexachlorobenzene	ug/L	118-74-1	1	8270C	10	5.0
Hexachloro-1,3-butadiene	ug/L	87-68-3	1.4	8270C	10	5.0
Hexachlorocyclopentadiene	ug/L	77-47-4	50	8270C	10	5.0
Hexachloroethane	ug/L	67-72-1	3.3	8270C	10	5.0
lndeno[1,2,3-cd]pyrene	ug/L	193-39-5	0.34	8270C SIM	0.10	0.015
Isophorone	ug/L	78-59-1	780	8270C	10	5.0
2-Methylphenol (o-Cresol)	ug/L	95-48-7	930	8270C	10	5.0
3 & 4-Methylphenol (m & p Cresols)	ug/L	108-39-4, 106-44-5	930	8270C	20	10
2-Methylnaphthalene	ug/L	91-57-6	36	8270C SIM	1.0	0.062
N -Nitroso-di-*n* -propylamine	ug/L	621-64-7	0.11	8270C	10	5.0
N -Nitrosodiphenylamine	ug/L	86-30-6	120	8270C	10	5.0
Naphthalene	ug/L	91-20-3	1.7	8270C SIM	1.0	0.098
2-Nitroaniline	ug/L	88-74-4	190	8270C	50	25
3-Nitroaniline	ug/L	99-09-2	NA	8270C	50	25
4-Nitroaniline	ug/L	100-01-6	38	8270C	50	25

TABLE B-2A

Groundwater reporting limits and minimum detection limits versus 2017 Remediation Closure Guide Criteria

Exide Technologies

Frankfort, Indiana

Analysis	Units	CAS Number	2017 RCG Ground Water Tap Limit	Method	RL	MDL
Nitrobenzene	ug/L	98-95-3	1.4	8270C	10	5.0
2-Nitrophenol	ug/L	88-75-5	NA	8270C	10	5.0
4-Nitrophenol	ug/L	100-02-7	NA	8270C	50	50
2,2'-Oxybis(1-Chloropropane)	ug/L	108-60-1	710	8270C	10	5
Pentachlorophenol	ug/L	87-86-5	1	8270C	50	25
Phenanthrene	ug/L	85-01-8	NA	8270C SIM	1.0	0.03
Phenol	ug/L	108-95-2	5800	8270C	10	5.0
Pyrene	ug/L	129-00-0	120	8270C SIM	1.0	0.015
1,2,4,5-Tetrachlorobenzene	ug/L	95-94-3	1.7	8270C	10.0	5
2,3,4,6-Tetrachlorophenol	ug/L	58-90-2	240	8270C	10.0	5
2,4,5-Trichlorophenol	ug/L	95-95-4	1200	8270C	10	5.0
2,4,6-Trichlorophenol	ug/L	88-06-2	12	8270C	10	5.0
Inorganics						
Arsenic	ug/L	7440-38-2	10	6010B	10	3.18
Barium	ug/L	7440-39-3	2000	6010B	10	0.83
Cadmium	ug/L	7440-43-9	5	6010B	2.0	0.24
Chromium	ug/L	7440-47-3	100	6010B	10	4.439
Lead	ug/L	7439-92-1	15	6010B	10	4.47
Mercury	ug/L	7439-97-6	2	7471A	2.0	0.10
Selenium	ug/L	7782-49-2	50	6010B	10	3.76
Silver	ug/L	7440-22-4	94	6010B	10	8.64
Analysis	**Units**	**CAS Number**	**2009 RISC Closure Level[1]**	**Method**	**RL**	**MDL**
Total Petroleum Hydrocarbons						
Gasoline Range Organics C5-C12	mg/L	NA	14	8015D	0.20	0.10
Diesel Range Organics C8-C28	mg/L	NA	2.5	8015D	0.10	0.038

Notes:

[1]TPH Closure Level - RISC Announcements July 06, 2009

TABLE B-2B
Soil reporting limits and minimum detection limits versus 2017 Remediation Closure Guide Criteria
Exide Technologies
Frankfort, Indiana

Analysis	Units	CAS Number	2017 RCG Soil Direct Contact Residential Limit	Method	RL	MDL
Volatile Organic Compounds						
Acetone	mg/Kg	67-64-1	85000	8260C	0.1	0.05
Benzene	mg/Kg	71-43-2	17	8260C	0.005	0.001
Bromodichloromethane	mg/Kg	75-27-4	4.1	8260C	0.005	0.0025
Bromoform	mg/Kg	75-25-2	270	8260C	0.005	0.0032
Bromomethane	mg/Kg	74-83-9	9.5	8260C	0.005	0.004
Bromochloromethane	mg/Kg	74-97-5	210	8260C	0.005	0.0025
2-Butanone (MEK)	mg/Kg	78-93-3	28000	8260C	0.025	0.012
Carbon disulfide	mg/Kg	75-15-0	740	8260C	0.010	0.005
Carbon tetrachloride	mg/Kg	56-23-5	9.1	8260C	0.005	0.0025
Chlorobenzene	mg/Kg	108-90-7	390	8260C	0.005	0.0025
Chloroethane (Ethyl Chloride)	mg/Kg	75-00-3	2100	8260C	0.005	0.0025
Chloroform	mg/Kg	67-66-3	4.5	8260C	0.005	0.0025
Chloromethane	mg/Kg	74-87-3	150	8260C	0.005	0.0025
Cyclohexane	mg/Kg	110-82-7	120	8260C	0.050	0.1
Dibromochloromethane	mg/Kg	124-48-1	120	8260C	0.005	0.0025
1,2-Dibromoethane (EDB)	mg/Kg	106-93-4	0.5	8260C	0.005	0.0025
1,2-Dibromo-3-chloropropane (DBCP)	mg/Kg	96-12-8	0.076	8260C	0.005	0.0025
1,2-Dichlorobenzene	mg/Kg	95-50-1	380	8260C	0.005	0.0025
1,3-Dichlorobenzene	mg/Kg	541-73-1	NA	8260C	0.005	0.0025
1,4-Dichlorobenzene	mg/Kg	106-46-7	36	8260C	0.005	0.0025
Dichlorodifluoromethane	mg/Kg	75-71-8	120	8260C	0.005	0.005
1,1-Dichloroethane	mg/Kg	75-34-3	50	8260C	0.005	0.0025
1,2-Dichloroethane (EDC)	mg/Kg	107-06-2	6.4	8260C	0.005	0.0025
1,1-Dichloroethene	mg/Kg	75-35-4	320	8260C	0.005	0.0025
cis -1,2-Dichloroethene	mg/Kg	156-59-2	220	8260C	0.005	0.0025
trans -1,2-Dichloroethene	mg/Kg	156-60-5	1900	8260C	0.005	0.0025
1,2-Dichloropropane	mg/Kg	78-87-5	14	8260C	0.005	0.0025
cis -1,3-Dichloropropene	mg/Kg	10061-01-5	25	8260C	0.005	0.0025
trans -1,3-Dichloropropene	mg/Kg	10061-02-6	25	8260C	0.005	0.0025
1,4 - Dioxane (p-Dioxane)	mg/Kg	123-91-1	74	8260C	0.5	0.25
Ethylbenzene	mg/Kg	100-41-4	81	8260C	0.005	0.0025
2-Hexanone	mg/Kg	591-78-6	280	8260C	0.1	0.05
lsopropylbenzene (Cumene)	mg/Kg	98-82-8	270	8260C	0.005	0.0025
Methylacetate	mg/Kg	79-20-9	29000	8260C	0.003	0.005
Methylcyclohexane	mg/Kg	108-87-2	NA	8260C	0.003	0.005
Methylene Chloride	mg/Kg	75-09-2	490	8260C	0.02	0.01
4-Methyl-2-pentanone (MIBK)	mg/Kg	108-10-1	3400	8260C	0.03	0.012
Methyl-tert-butyl-Ether (MTBE)	mg/Kg	1634-04-4	660	8260C	0.005	0.0025
Styrene	mg/Kg	100-42-5	870	8260C	0.005	0.0025
1,1,2,2-Tetrachloroethane	mg/Kg	79-34-5	8.4	8260C	0.005	0.0025
Tetrachloroethene (PCE)	mg/Kg	127-18-4	110	8260C	0.005	0.0014
Toluene	mg/Kg	108-88-3	820	8260C	0.005	0.0025
1,2,3-Trichlorobenzene	mg/Kg	87-61-6	88	8260C	0.005	0.0025
1,2,4-Trichlorobenzene	mg/Kg	120-82-1	81	8260C	0.005	0.0025
1,1,1-Trichloroethane (TCA)	mg/Kg	71-55-6	640	8260C	0.005	0.0025
1,1,2-Trichloroethane	mg/Kg	79-00-5	2.1	8260C	0.005	0.0025
1,1,2-Trichloro-1,2,2-trifluoroethane	mg/Kg	76-13-1	910	8260C	0.003	0.005
Trichloroethene (TCE)	mg/Kg	79-01-6	5.7	8260C	0.005	0.001
Trichlorofluoromethane	mg/Kg	75-69-4	1200	8260C	0.005	0.0025
Vinyl Chloride	mg/Kg	75-01-4	0.83	8260C	0.005	0.0025
Xylenes, Total	mg/Kg	1330-20-7	260	8260C	0.010	0.005
Semivolatiles Organic Compounds						
Acenaphthene	mg/Kg	83-32-9	5000	8270C	0.33	0.16

TABLE B-2B
Soil reporting limits and minimum detection limits versus 2017 Remediation Closure Guide Criteria
Exide Technologies
Frankfort, Indiana

Analysis	Units	CAS Number	2017 RCG Soil Direct Contact Residential Limit	Method	RL	MDL
Acenaphthylene	mg/Kg	208-96-8	NA	8270C	0.33	0.16
Acetophenone	mg/kg	98-86-2	2500	8270C	0.33	0.165
Anthracene	mg/Kg	120-12-7	25000	8270C	0.33	0.16
Atrazine	mg/kg	1912-24-9	34	8270C	0.33	0.165
Benzaldehyde	mg/kg	100-52-7	1200	8270C	0.33	0.165
Benzo[a]anthracene	mg/Kg	56-55-3	2.2	8270C	0.33	0.16
Benzo[a]pyrene	mg/Kg	50-32-8	0.22	8270C	0.33	0.16
Benzo[b]fluoranthene	mg/Kg	205-99-2	2.2	8270C	0.33	0.16
Benzo[g,h,i]perylene	mg/Kg	191-24-2	NA	8270C	0.33	0.16
Benzo[k]fluoranthene	mg/Kg	207-08-9	22	8270C	0.33	0.16
1,1-Biphenyl	mg/kg	92-52-4	66	8270C	0.33	0.165
Bis(2-chloroethoxy) methane	mg/Kg	111-91-1	270	8270C	0.33	0.16
Bis(2-chloroethyl) ether	mg/Kg	111-44-4	3.2	8270C	0.33	0.16
Bis(2-ethylhexyl) phthalate	mg/Kg	117-81-7	550	8270C	0.33	0.16
4-Bromophenyl phenyl ether	mg/Kg	101-55-03	NA	8270C	0.33	0.16
Butyl benzyl phthalate	mg/Kg	85-68-7	4100	8270C	0.33	0.16
Caprolactam	mg/kg	105-60-2	43000	8270C	0.33	0.33
Carbazole	mg/kg	86-74-8	NA	8270C	0.33	0.165
4-Chloroaniline	mg/Kg	106-47-8	38	8270C	0.33	0.16
4-Chloro-3-methylphenol	mg/Kg	59-50-7	8800	8270C	0.33	0.16
2-Chloronaphthalene	mg/Kg	91-58-7	6700	8270C	0.33	0.16
2-Chlorophenol	mg/Kg	95-57-8	550	8270C	0.33	0.16
4-Chlorophenyl phenyl ether	mg/Kg	7005-72-3	NA	8270C	0.33	0.16
Chrysene	mg/Kg	218-01-9	220	8270C	0.33	0.16
Dibenz[a,h]anthracene	mg/Kg	53-70-3	0.22	8270C	0.33	0.16
Dibenzofuran	mg/Kg	132-64-9	100	8270C	0.33	0.16
Di-*n* -butyl phthalate	mg/Kg	84-74-2	8800	8270C	0.33	0.16
3,3'-Dichlorobenzidine	mg/Kg	91-94-1	17	8270C	0.66	0.33
2,4-Dichlorophenol	mg/Kg	120-83-2	270	8270C	0.33	0.16
Diethyl phthalate	mg/Kg	84-66-2	71000	8270C	0.33	0.16
2,4-Dimethylphenol	mg/Kg	105-67-9	1800	8270C	0.33	0.16
Dimethylphthalate	mg/Kg	131-11-3	NA	8270C	0.33	0.16
4,6-Dinitro-2-methylphenol (4,6-Dinitro-o-cresol)	mg/Kg	534-52-1	7.1	8270C	1.6	0.8
2,4-Dinitrophenol	mg/Kg	51-28-5	180	8270C	1.6	0.8
2,4-Dinitrotoluene	mg/Kg	121-14-2	24	8270C	0.33	0.16
2,6-Dinitrotoluene	mg/Kg	606-20-2	5	8270C	0.33	0.16
Di-*n* -octyl phthalate	mg/Kg	117-84-0	880	8270C	0.33	0.16
Fluoranthene	mg/Kg	206-44-0	3400	8270C	0.33	0.16
Fluorene	mg/Kg	86-73-7	3400	8270C	0.33	0.16
Hexachlorobenzene	mg/Kg	118-74-1	2.9	8270C	0.33	0.16
Hexachloro-1,3-butadiene	mg/Kg	87-68-3	17	8270C	0.33	0.16
Hexachlorocyclopentadiene	mg/Kg	77-47-4	2.5	8270C	0.33	0.33
Hexachloroethane	mg/Kg	67-72-1	25	8270C	0.33	0.16
lndeno[1,2,3-cd]pyrene	mg/Kg	193-39-5	2.2	8270C	0.33	0.16
Isophorone	mg/Kg	78-59-1	8000	8270C	0.33	0.16
2-Methylphenol (o-Cresol)	mg/Kg	95-48-7	4500	8270C	0.33	0.16
3 & 4-Methylphenol (m & p Cresols)	mg/Kg	108-39-4, 106-44-5	4500	8270C	0.33	0.16
2-Methylnaphthalene	mg/Kg	91-57-6	340	8270C	0.33	0.16
N -Nitroso-di-*n* -propylamine	mg/Kg	621-64-7	1.1	8270C	0.33	0.16
N -Nitrosodiphenylamine	mg/Kg	86-30-6	1500	8270C	0.33	0.16
Naphthalene	mg/Kg	91-20-3	53	8270C	0.33	0.16
2-Nitroaniline	mg/Kg	88-74-4	880	8270C	1.6	0.8
3-Nitroaniline	mg/Kg	99-09-2	NA	8270C	1.6	0.8
4-Nitroaniline	mg/Kg	100-01-6	350	8270C	1.6	0.8

TABLE B-2B
Soil reporting limits and minimum detection limits versus 2017 Remediation Closure Guide Criteria
Exide Technologies
Frankfort, Indiana

Analysis	Units	CAS Number	2017 RCG Soil Direct Contact Residential Limit	Method	RL	MDL
Nitrobenzene	mg/Kg	98-95-3	71	8270C	0.33	0.16
2-Nitrophenol	mg/Kg	88-75-5	NA	8270C	0.33	0.16
4-Nitrophenol	mg/Kg	100-02-7	NA	8270C	1.6	0.8
2,2'-Oxybis(1-Chloropropane)	mg/kg	108-60-1	1000	8270C	0.33	0.165
Pentachlorophenol	mg/Kg	87-86-5	14	8270C	1.6	1.6
Phenanthrene	mg/Kg	85-01-8	NA	8270C	0.33	0.16
Phenol	mg/Kg	108-95-2	27000	8270C	0.33	0.16
Pyrene	mg/Kg	129-00-0	2500	8270C	0.33	0.16
1,2,4,5-Tetrachlorobenzene	mg/kg	95-94-3	32	8270C	0.33	0.165
2,3,4,6-Tetrachlorophenol	mg/kg	58-90-2	2700	8270C	0.33	0.165
2,4,5-Trichlorophenol	mg/Kg	95-95-4	8800	8270C	0.33	0.16
2,4,6-Trichlorophenol	mg/Kg	88-06-2	88	8270C	0.33	0.16
Inorganics						
Arsenic	mg/Kg	7440-38-2	5.5	6010B	1	0.5
Barium	mg/Kg	7440-39-3	21000	6010B	1	0.5
Cadmium	mg/Kg	7440-43-9	99	6010B	0.5	0.25
Chromium	mg/Kg	7440-47-3	NA	6010B	1	0.5
Lead	mg/Kg	7439-92-1	400	6010B	1	0.5
Mercury	mg/Kg	7439-97-6	3.1	7471A	0.2	0.1
Selenium	mg/Kg	7782-49-2	550	6010B	1	0.5
Silver	mg/Kg	7440-22-4	550	6010B	0.5	0.391
Analysis	**Units**	**CAS Number**	**2009 RISC Closure Level[1]**	**Method**	**RL**	**MDL**
Total Petroleum Hydrocarbons						
Gasoline Range Organics C5-C12	mg/Kg	NA	4300	8015D	1	0.5
Diesel Range Organics C8-C28	mg/Kg	NA	5800	8015D	10	5

Notes:
[1]TPH Closure Level - RISC Announcements July 06, 2009

TABLE B-3
SAMPLE CONTAINERS AND PRESERVATIVES
Exide Technologies
Frankfort, Indiana

Sampling Event	Matrix	Analysis	Extraction Method	Analytical Method	Container	Preservative	Holding Time
RFI Soil	Soil	TCL Volatiles	5035	8260C	Terracore Sampling Kit	Ice	48 hours to extraction, 14 days to analysis
		TCL Semivolatiles	3550C	8270C	4 oz WMG	Ice	14 days to extraction, then 40 days to analysis
		GRO	NA	8015D	Terracore Sampling Kit	Ice	48 hours to extraction, 14 days to analysis
		DRO	NA	8015D	4 oz WMG	Ice	14 days
		RCRA 8 Metals	3050B	6010B/7471A	4 oz WMG	Ice	180 days (Hg-28 days)
		pH	NA	9045C	4 oz WMG	Ice	ASAP (24 hours)
RFI Groundwater	Groundwater	TCL Volatiles	NA	8260C	3 40mL VOA vials	pH < 2, HCl, Ice	14 days
		TCL Semivolatiles	3510C	8270C/8270C SIM	100 ml Amber	Ice	7 days to extraction, then 40 days to analysis
		1,4-Dioxane	3510C	8270C SIM	1 liter Amber	Ice	7 days to extraction, then 40 days to analysis
		GRO	NA	8015D	3 40mL VOA vials	pH < 2, HCl, Ice	14 days
		DRO	NA	8015D	2 L Amber	Ice	7 days
		Total RCRA 8 Metals	3010A	6010B/7470A	500 ml HDPE	HNO3	180 days (Hg-28 days)
		Dissolved RCRA 8 Metals	3010A	6010B/7470A	500 ml HDPE	Field Filtered, HNO3	180 days (Hg-28 days)
Well Installation	Soil	TCL Volatiles	5035	8260C	Terracore Sampling Kit	Ice	48 hours to extraction, 14 days to analysis
		TCL Semivolatiles	3550C	8270C	4 oz WMG	Ice	14 days to extraction, then 40 days to analysis

NA - Not applicable. Extraction method is included in the analytical procedures.
WMG - Wide mouth glass
Sample containers are based on individual sampling analysis. The laboratory may combine and increase sample container sizes if multiple analysis is requested.

Attachment B-1

Laboratory Quality Assurance Manual and Standard Operating Procedures

QUALITY ASSURANCE MANUAL

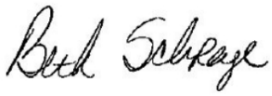
Quality Assurance/Quality Control Policies and Procedures

Pace Analytical Services, LLC – Indianapolis
7726 Moller Road
Indianapolis, IN 46268
(317)228-3100

APPROVAL

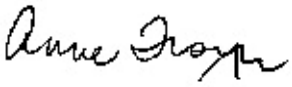
April 18, 2017
Date

Steve Sayer
General Manager
(317)228-3100



April 18, 2017
Date

Beth Schrage
Quality Manager
(317)228-3100



April 18, 2017
Date

Anne Troyer
Technical Director
(317)228-3100

Table of Contents

1.0. INTRODUCTION AND ORGANIZATIONAL STRUCTURE

"Working together to protect our environment and improve our health"
Pace Analytical Services LLC - Mission Statement

1.1. Introduction to Pace

1.1.1. Pace Analytical Services, LLC is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. Pace offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, dioxins and coplanar PCB's by high resolution mass spectroscopy , radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. This document defines the Quality System and Quality Assurance (QA)/Quality Control (QC) protocols.

1.1.2. Pace laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. Methods are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, Standard Methods, and State Agencies. Section 11 of this document is a representative listing of general analytical protocol references.

1.2. Statement of Purpose

1.2.1. To meet the business needs of our customers for high quality, cost-effective analytical measurements and services.

1.3. Quality Policy Statement and Goals of the Quality System

1.3.1. Pace management is committed to maintaining the highest possible standard of service and quality for our customers by following a documented quality system that is compliant with all current applicable state, federal, and industry standards, such as the NELAC Standard, the TNI Standard, and ISO standards and is in accordance with the stated methods and customer requirements. The overall objective of this quality system is to provide reliable data of known quality through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

1.3.2. All personnel within the Pace network are required to be familiar with all facets of the quality system relevant to their position and implement these policies and procedures in their daily work.

1.4. Core Values

1.4.1. The following are the Pace Core Values:

- **Integrity**
- **Value Employees**
- **Know Our Customers**
- **Honor Commitments**

- **Flexible Response To Demand**
- **Pursue Opportunities**
- **Continuously Improve**

1.5. Code of Ethics and Standards of Conduct

1.5.1. **Code of Ethics**:

1.5.1.1. Each Pace employee is responsible for the propriety and consequences of his or her actions;

1.5.1.2. Each Pace employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where Pace does business or seeks to do business;

1.5.1.3. Each Pace employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.

1.5.1.4. Each Pace employee must recognize and understand that our daily activities in environmental laboratories affect public health as well as the environment and that environmental laboratory analysts are a critical part of the system society depends upon to improve and guard our natural resources:

1.5.2. **Standards of Conduct:**

1.5.2.1. **Data Integrity**

1.5.2.1.1. The accuracy and integrity of the analytical results and its supporting documentation produced at Pace are the cornerstones of the company. Employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations, and databases. Employees are prohibited from making false entries or misrepresentations of data for any reason.

1.5.2.1.2. Managerial staff must make every effort to ensure that personnel are free from any undue pressures that may affect the quality or integrity of their work including commercial, financial, over-scheduling, and working condition pressures.

1.5.2.1.3. The data integrity system includes in-depth, periodic monitoring of data integrity including peer data review and validation, internal raw data audits, proficiency testing studies, etc.

1.5.2.1.4. Any documentation related to data integrity issues, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be retained for a minimum of five years.

1.5.2.2. **Confidentiality**

1.5.2.2.1. Pace employees must not use or disclose confidential or proprietary information except when in connection with their duties at Pace. This is effective over the course of employment and for an additional period of two years thereafter.

1.5.2.2.2. Confidential or proprietary information, belonging to either Pace and/or its customers, includes but is not limited to test results, trade secrets, research and development

matters, procedures, methods, processes and standards, company-specific techniques and equipment, marketing and customer information, inventions, materials composition, etc.

1.5.2.3. **Conflict of Interest**

1.5.2.3.1. Pace employees must avoid situations that might involve a conflict of interest or could appear questionable to others. This includes participation in activities that conflict or appear to conflict with the employees' Pace responsibilities. This would also include offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced to behave or in a different manner than he would normally (such as bribes, gifts, kickbacks, or illegal payments).

1.5.2.3.2. Employees are not to engage in outside business or economic activity relating to a sale or purchase by the Company. Other problematic activities include service on the Board of Directors of a competing or supplier company, significant ownership in a competing or supplier company, employment for a competing or supplier company, or participation in any outside business during the employee's work hours.

1.5.3. Strict adherence by each Pace employee to this Code of Ethics and to the Standards of Conduct is essential to the continued vitality of Pace and to continue the pursuit of our common mission to protect our environment and improve our health.

1.5.4. Failure to comply with the Code of Ethics and Standards of Conduct will result in disciplinary action up to and including termination and referral for civil or criminal prosecution where appropriate. An employee will be notified of an infraction and given an opportunity to explain, as prescribed under current disciplinary procedures.

1.5.5. Compliance: all employees undergo annual Data Integrity/Ethics training which includes the concepts listed above. All employees also sign an annual Ethic Policy statement.

1.6. Anonymous Compliance Alertline

1.6.1. An ethical and safe workplace is important to the long-term success of Pace and the well-being of its employees. Pace has a responsibility to provide a work environment where employees feel safe and can report unethical or improper behavior in complete confidence. With this in mind, Pace has engaged Lighthouse Services, Inc. to provide all employees with access to an anonymous ethics and compliance alertline for reporting possible ethics and compliance violations. The purpose of this service is to ensure that any employee can report anonymously and without fear of retaliation.

1.6.2. Lighthouse Services provides a toll-free number along with several other reporting methods, all of which are available 24 hours a day, seven days a week for use by employees and staff.

1.6.3. Telephone: English speaking USA and Canada: (844)-970-0003.

1.6.4. Telephone: Spanish speaking North America: (800)-216-1288.

1.6.5. Website: www.lighthouse-services.com/pacelabs.

1.6.6. Email: reports@lighthouse-services.com (must include company name with report).

1.7. Laboratory Organization

1.7.1. Each laboratory within the system operates with local management, but all labs share common systems and receive support from the Corporate Office. See Attachment III for the Corporate Organizational structure.

1.7.2. A Senior General Manager (SGM) oversees all laboratories and service centers in their assigned region. Each laboratory or facility in the company is then directly managed by an SGM, a General Manager (GM), an Assistant General Manager (AGM), or an Operations Manager (OM). Quality Managers (QM) or Senior Quality Managers (SQM) at each laboratory report directly to the highest level of local laboratory management, however named, that routinely makes day-to-day decisions regarding that facility's operations. The QMs and SQMs will also receive guidance and direction from the corporate Director of Environmental Quality.

1.7.3. The SGM, GM, AGM or OM, or equivalent functionality in each facility, bears the responsibility for the laboratory operations and serves as the final, local authority in all matters. In the absence of these managers, the SQM/QM serves as the next in command, unless the manager in charge has assigned another designee. He or she assumes the responsibilities of the manager, however named, until the manager is available to resume the duties of their position. In the absence of both the manager and the SQM/QM, management responsibility of the laboratory is passed to the Technical Director, provided such a position is identified, and then to the most senior department manager until the return of the lab manager or SQM/QM. The most senior department manager in charge may include the Client Services Manager (CSM) or the Administrative Business Manager (ABM) at the discretion of the SGM/GM/AGM/OM.

1.7.4. A Technical Director who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director to temporarily perform this function. The laboratory SGM/GM/AGM/OM or SQM/QM has the authority to make this designation in the event the existing Technical Director is unable to do so. If this absence exceeds 35 consecutive calendar days, the primary accrediting authority shall be notified in writing.

1.7.5. The SQM/QM has the responsibility and authority to ensure the Quality System is implemented and followed at all times. In circumstances where a laboratory is not meeting the established level of quality or following the policies set forth in this Quality Assurance Manual, the SQM/QM has the authority to halt laboratory operations should he or she deem such an action necessary. The SQM/QM will immediately communicate the halting of operations to the SGM/GM/AGM/OM and keep them posted on the progress of corrective actions. In the event the SGM/GM/AGM/OM and the SQM/QM are not in agreement as to the need for the suspension, the Chief Operating Officer (COO) and Director of Environmental Quality will be called in to mediate the situation.

1.7.6. The technical staff of the laboratory is generally organized into the following functional groups:

- Organic Extractions
- Wet Chemistry Analysis
- Metals Analysis
- Volatiles Analysis
- Semi-volatiles Analysis

1.7.7. The organizational structure for Pace – Indianapolis is listed in Attachment II. In the event of a change in SGM/GM/AGM/OM, SQM/QM, or any Technical Director, the laboratory will notify its

accrediting authorities per their individual required timeframes, not to exceed 30 days. The QAM will remain in effect until the next scheduled revision.

1.8. Laboratory Job Descriptions

1.8.1. Senior General Manager

- Oversees all functions of all the operations within their designated region;
- Oversees the development of local GMs/AGMs/OMs within their designated region;
- Oversees and authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Oversees the preparation of budgets and staffing plans for all operations within their designated region;
- Ensures compliance with all applicable state, federal and industry standards;
- Works closely with Regional Sales Management.

1.8.2. General Manager

- Oversees all functions of their assigned operations;
- Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Prepares budgets and staffing plans;
- Monitors the Quality Systems of the laboratory and advises the SQM/QM accordingly;
- Presents the Ethics/Data Integrity training annually to all employees in their facilities as an instructor-led training.
- Ensures compliance with all applicable state, federal and industry standards.

1.8.4. Quality Manager

- Responsible for implementing, maintaining and improving the quality system while functioning independently from laboratory operations. Reports directly to the highest level of local laboratory facility management, however named, that routinely makes day-to-day decisions regarding laboratory operations, but receives direction and assistance from the Corporate Director of Environmental Quality;
- Ensures that communication takes place at all levels within the lab regarding the effectiveness of the quality system and that all personnel understand their contributions to the quality system;
- Monitors QA/QC activities to ensure that the laboratory achieves established standards of quality (as set forth by the Corporate Environmental Quality office). The QM is responsible for reporting the lab's level of compliance to these standards to the Corporate Director of Environmental Quality on a quarterly basis;
- Maintains records of quality control data and evaluates data quality;
- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives;
- Reviews select laboratory data and final reports;
- Reviews tenders, contracts and QAPPs to ensure the laboratory can meet the data quality objectives for any given project;
- Reviews and maintains records of proficiency testing results;

- Maintains the document control system;
- Assists in development and implementation of appropriate training programs;
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements;
- Maintains certifications from federal and state programs;
- Ensures compliance with all applicable state, federal and industry standards;
- Maintains the laboratory training records, including those in the Learning Management System (LMS), and evaluates the effectiveness of training;
- Monitors corrective and preventive actions;
- Maintains calibration of support equipment such as balances and thermometers;
- Maintains the currency of the Quality Manual.

1.8.5. **Technical Director**

- Monitors the standards of performance in quality assurance and quality control data;
- Monitors the validity of analyses performed and data generated;
- May review tenders, contracts and QAPPs to ensure the laboratory can meet the data quality objectives for any given project;
- Serves as the manager of the laboratory in the absence of the SGM/GM/AGM/OM and SQM/QM;
- Provides technical guidance in the review, development, and validation of new methodologies.

1.8.6. **Administrative Business Manager**

- Responsible for financial and administrative management for the entire facility;
- Provides input relative to tactical and strategic planning activities;
- Organizes financial information so that the facility is run as a fiscally responsible business;
- Works with staff to confirm that appropriate processes are put in place to track revenues and expenses;
- Provide ongoing financial information to the SGM/GM/AGM/OM and the management team so they can better manage their business;
- Utilizes historical information and trends to accurately forecast future financial positions;
- Works with management to ensure that key measurements are put in place to be utilized for trend analysis—this will include personnel and supply expenses, and key revenue and expense ratios;
- Works with SGM/GM/AGM/OM to develop accurate budget and track on an ongoing basis;
- Works with entire management team to submit complete and justified capital budget requests and to balance requests across departments;
- Works with project management team and administrative support staff to ensure timely and accurate invoicing.

1.8.7. **Client Services Manager**

- Oversees all the day to day activities of the Client Services Department which includes Project Management and, possibly, Sample Control;
- Responsible for staffing and all personnel management related issues for Client Services;

- Serves as the primary senior consultant to customers on all project related issues such as set up, initiation, execution and closure;
- Performs or is capable of performing all duties listed for that of Project Manager.

1.8.8. Project Manager

- Coordinates daily activities including taking orders, reporting data and analytical results;
- Serves as the primary technical and administrative liaison between customers and Pace;
- Communicates with operations staff to update and set project priorities;
- Provides results to customers in the requested format (verbal, hardcopy, electronic, etc.);
- Works with customers, laboratory staff, and other appropriate Pace staff to develop project statements of work or resolve problems of data quality;
- Responsible for solicitation of work requests, assisting with proposal preparation and project initiation with customers and maintain customer records;
- Mediation of project schedules and scope of work through communication with internal resources and management;
- Responsible for preparing routine and non-routine quotations, reports and technical papers;
- Interfaces between customers and management personnel to achieve customer satisfaction;
- Manages large-scale complex projects;
- Supervises less experienced project managers and provide guidance on management of complex projects;
- Arranges bottle orders and shipment of sample kits to customers;
- Verifies login information relative to project requirements and field sample Chains-of-Custody;
- Enters project and sample information in the Laboratory Information Management System (LIMS) for scheduling, tracking and reporting purposes.

1.8.9. Department Manager/Supervisor

- Oversees the day-to-day production and quality activities of their assigned department;
- Ensures that quality assurance and quality control criteria of analytical methods and projects are satisfied;
- Assesses data quality and takes corrective action when necessary;
- Approves and releases technical and data management reports;
- Trains analysts or oversees training of analysts in laboratory operations and analytical procedures;
- Ensures compliance with all applicable state, federal and industry standards.

1.8.10. Quality Assurance Analyst

- Assists the SQM/QM in the performance of quality department responsibilities as delegated by the SQM/QM;
- Reviews select laboratory data and final reports;
- Generates and reviews QC data validation packages;
- Assists in monitoring QA/QC data;
- Assists in internal audits;
- Assists in maintaining training records;
- Assists in maintaining the document control system.

1.8.11. Group Supervisor/Leader

- Trains analysts in laboratory operations and analytical procedures;
- Organizes and schedules analyses with consideration for sample holding times;
- Implements data verification procedures by assigning data verification duties to appropriate personnel;
- Evaluates instrument performance and supervises instrument calibration and preventive maintenance programs;
- Reports non-compliance situations to laboratory management including the SQM/QM.

1.8.12. Laboratory Analyst

- Performs detailed preparation and analysis of samples according to published methods and laboratory procedures;
- Processes and evaluates raw data obtained from preparation and analysis steps;
- Generates final results from raw data, performing primary review against method criteria;
- Monitors quality control data associated with analysis and preparation. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks;
- Reports data in LIMS, authorizing for release pending secondary approval;
- Conducts routine and non-routine maintenance of equipment as required;
- Performs or is capable of performing all duties associated with that of Laboratory Technician.

1.8.13. Laboratory Technician

- Prepares standards and reagents according to published methods or in house procedures;
- Performs preparation and analytical steps for basic laboratory methods;
- Works under the direction of a Laboratory Analyst on complex methodologies;
- Assists Laboratory Analysts on preparation, analytical or data reduction steps for complex methodologies;
- Monitors quality control data as required or directed. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks.

1.8.14. Field Technician

- Prepares and samples according to published methods, PACE Quality Assurance Manual and/or customer directed sampling objectives;
- Capable of the collection of representative environmental or process samples;
- Reviews project documentation for completeness, method compliance and contract fulfillment;
- Train less experienced environmental technicians and provide guidance on sampling and analysis;
- Responsible for project initiation and contact follow-up;
- Develop sampling plans and prepare test plan documents.

1.8.15. **Sample Receiving Personnel**

- Signs for incoming samples and verifies the data entered on the Chain of custody forms;
- Stages samples according to EPA requirements;
- Assists Project Managers and Coordinators in filling bottle orders and sample shipments;
- May enter project and sample information in the Laboratory Information Management System (LIMS) for scheduling, tracking and reporting purposes;
- Manages sample storage areas and sample disposal procedures.

1.8.16. **Systems Administrator or Systems Manager**

- Assists with the creation and maintenance of electronic data deliverables (EDDs);
- Coordinates the installation and use of all hardware, software and operating systems;
- Performs troubleshooting on all aforementioned systems;
- Trains new and existing users on systems and system upgrades;
- Maintains all system security passwords;
- Maintains the electronic backups of all computer systems.

1.8.17. **Safety/Chemical Hygiene Officer**

- Maintains the laboratory Chemical Hygiene Plan;
- Plans and implements safety policies and procedures;
- Maintains safety records;
- Organizes and/or performs safety training;
- Performs safety inspections and provides corrective/preventative actions;
- Assists personnel with safety issues.

1.8.18. **Hazardous Waste Coordinator**

- Evaluates waste streams and helps to select appropriate waste transportation and disposal companies;
- Maintains complete records of waste disposal including waste manifests and state reports;
- Assists in training personnel on waste-related issues such as waste handling and storage, waste container labeling, proper satellite accumulation, secondary containment, etc.;
- Conducts a weekly inspection of the waste storage areas of the laboratory.

1.9. Training and Orientation

1.9.1. Training for Pace employees is managed through web-based training systems. Employees are provided with several training activities for their particular job description and scope of duties. These training activities may include:

- Hands-on training led by supervisors;
- Job-specific training checklists and worksheets;
- Lectures and instructor-led training sessions;
- Method-specific training;
- External conferences and seminars;
- Reading Standard Operating Procedures (SOPs);

- Reading the Quality Assurance Manual and Safety Manual/Chemical Hygiene Plan;
- Core training modules (basic lab skills, etc.);
- Quality system training modules (support equipment use, corrective actions/root causes, etc.);
- Data Integrity/Ethics training;
- Specialized training by instrument manufacturers;
- On-line courses.

1.9.2. All procedures and training records are maintained and available for review during laboratory audits. Additional information can be found in SOP S-IN-Q-153 *Training Procedures* or its equivalent revision or replacement.

1.10. Laboratory Safety and Waste

1.10.1. It is the policy of Pace to make safety and waste compliance an integral part of daily operations and to ensure that all employees are provided with safe working conditions, personal protective equipment, and requisite training to do their work without injury. Each employee is responsible for his/her own safety as well as those working in the immediate area by complying with established company rules and procedures. These rules and procedures as well as a more detailed description of the employees' responsibilities are contained in the local Safety Manual/Chemical Hygiene Plan.

1.11. Security and Confidentiality

1.11.1. Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by Pace staff. Keyless door locks are accessible only to authorized personnel through the use of assigned key fobs. All visitors, including PACE staff from other facilities, must sign the Visitor's Logbook maintained by the receptionist. A staff member will accompany them during the duration of their stay on the premises unless the SGM/GM/AGM/OM, SQM/QM, or Technical Director specify otherwise. In this instance, the staff member will escort the visitor back to the reception area at the end of his/her visit where he/she signs out.

1.11.2. Additional security is provided where necessary, (e.g., specific secure areas for sample, data, and customer report storage), as requested by customers, or cases where national security is of concern. These areas are lockable within the facilities, or are securely offsite. Access is limited to specific individuals or their designees.

1.11.3. All information pertaining to a particular customer, including national security concerns will remain confidential. Data will be released to outside agencies only with written authorization from the customer or where federal or state law requires the company to do so.

1.12. Communications

1.12.1. Management within each lab bears the responsibility of ensuring that appropriate communication processes are established and that communication takes place regarding the effectiveness of the management/quality system. These communication processes may include email, regular staff meetings, senior management meetings, etc.

1.12.2. Corporate management bears the responsibility of ensuring that appropriate communication processes are established within the network of facilities and that communication takes place at a company-wide level regarding the effectiveness of the management/quality systems of all Pace facilities. These communication processes may include email, quarterly continuous improvement conference calls for all lab departments, and annual continuous improvement meetings for all department supervisors, quality managers, client services managers, and other support positions.

2.0. SAMPLE CUSTODY

2.1. Project Initiation

2.1.1. Prior to accepting new work, the laboratory reviews its performance capability. The laboratory confirms that sufficient personnel, equipment capacity, analytical method capability, etc., are available to complete the required work. Customer needs, certification requirements, and data quality objectives are defined and the appropriate sampling and analysis plan is developed to meet the project requirements by project managers or sales representatives. Members of the management staff review current instrument capacity, personnel availability and training, analytical procedures capability, and projected sample load. Management then informs the sales and client services personnel whether or not the laboratory can accept the new project via written correspondence, email, and/or daily operations meetings.

2.1.2. Additional information regarding specific procedures for reviewing new work requests can be found in SOP S-IN-C-006 *Review of Analytical Requests* or its equivalent revision or replacement.

2.2. Sampling Materials and Support

2.2.1. Each individual Pace laboratory provides shipping containers, properly preserved sample containers, custody documents, and field quality control samples to support field-sampling events. Guidelines for sample container types, preservatives, and holding times for a variety of methods are listed in Attachment VII. Note that all analyses listed are not necessarily performed at all Pace laboratories and there may be additional laboratory analyses performed that are not included in these tables. Customers are encouraged to contact their local Pace Project Manager for questions or clarifications regarding sample handling. Pace may provide pick-up and delivery services to their customers when needed.

2.2.2. Some Pace facilities provide sampling support through a Field Services department. Field Services operates under the Pace Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services. All procedures and methods used by Field Services are documented in SOPs and Procedure Manuals.

2.3. Chain of Custody

2.3.1. A chain of custody (COC) provides the legal documentation of samples from time of collection to completion of analysis.

2.3.2. Field personnel or client representatives must complete a COC for all samples that are received by the laboratory. Samplers are required to properly complete a COC. This is critical to efficient sample receipt and to ensure the requested methods are used to analyze the correct samples. If sample shipments are not accompanied by the correct documentation, the Sample Receiving department notifies a Project Manager. The Project Manager then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

2.3.3. The COC is filled out completely and legibly with indelible ink. Errors are corrected by drawing a single line through the initial entry and initialing and dating the change. All transfers of samples are recorded on the chain of custody in the “relinquished” and “received by” sections. All information except signatures is printed.

2.3.4. Additional information can be found in SOP S-IN-C-001 *Sample Management* or its equivalent revision or replacement.

2.4. Sample Acceptance Policy

2.4.1. In accordance with regulatory guidelines, Pace complies with the following sample acceptance policy for all samples received.

2.4.2. If the samples do not meet the sample receipt acceptance criteria outlined below, the laboratory is required to document all non-compliances, contact the customer, and either reject the samples or fully document any decisions to proceed with analyses of samples which do not meet the criteria. Any results reported from samples not meeting these criteria are appropriately communicated to the client.

2.4.3. Sample Acceptance Policy requirements:

- Sample containers must have unique client identification designations that are clearly marked with indelible ink on durable, water-resistant labels. The client identifications must match those on the chain-of-custody (COC).
- There must be clear documentation on the COC, or related documents, that lists the unique sample identification, sampling site location, date and time of sample collection, and name of the sample collector.
- There must be clear documentation on the COC, or related documents, that lists the requested analyses, the preservatives used, and any special remarks concerning the samples (i.e., data deliverables, samples are for evidentiary purposes, field filtration, etc.).
- Samples must be in appropriate sample containers. If the sample containers show signs of damage (i.e., broken or leaking) or if the samples show signs of contamination, the samples will not be processed without prior client approval.
- Samples must be correctly preserved upon receipt, unless the method requested allows for laboratory preservation. If the samples are received with inadequate preservation, and the samples cannot be preserved by the lab appropriately, the samples will not be processed without prior client approval.
- Samples must be received within required holding time. Any samples with hold times that are exceeded will not be processed without prior client approval.
- Samples must be received with sufficient sample volume or weight to proceed with the analytical testing. If insufficient sample volume or weight is received, analysis will not proceed without client approval.
- All samples that require thermal preservation are considered acceptable if they are received at a temperature within 2°C of the required temperature, or within the method-specified range. For samples with a required temperature of 4°C, samples with a temperature ranging from just above freezing to 6°C are acceptable. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice. Any samples that are not received at the required temperature will not be processed without prior client approval.
- Samples for **drinking water** analyses will be rejected at the time of receipt if they are not received in a secure manner, are received in inappropriate containers, are received outside the required temperature range, are received outside the recognized holding time, are received with inadequate identification on sample containers or COC, or are improperly

preserved (with the exception of VOA samples- tested for pH at time of analysis and TOC- tested for pH in the field).

- Some specific clients may require custody seals. **For these clients**, samples or coolers that are not received with the proper custody seals will not be processed without prior client approval.

Note 1: Temperature will be read and recorded based on the precision of the measuring device. For example, temperatures obtained from a thermometer graduated to 0.1°C will be read and recorded to ± 0.1°C. Measurements obtained from a thermometer graduate to 0.5°C will be read to ± 0.5°C. Measurements read at the specified precision are not to be rounded down to meet the ≤ 6°C limit. Please reference the Support Equipment SOP for more information.

Note 2: Some microbiology methods allow sample receipt temperatures of up to 10°C. Consult the specific method for microbiology samples received above 6°C prior to initiating corrective action for out of temperature preservation conditions.

2.4.4. Upon sample receipt, the following items are also checked and recorded:

- Presence of custody seals or tapes on the shipping containers;
- Sample condition: Intact, broken/leaking, bubbles in VOA samples;
- Sample holding time;
- Sample pH and residual chlorine when required;
- Appropriate containers.

2.4.5. Additional information can be found in SOP S-IN-C-001 *Sample Management* or its equivalent revision or replacement.

2.5. Sample Log-in

2.5.1. After sample inspection, all sample information on the COC is entered into the Laboratory Information Management System (LIMS). The lab's permanent records for samples received include the following information:

- Customer name and contact
- Customer number
- Pace Analytical project number
- Pace Analytical Project Manager
- Sample descriptions
- Due dates
- List of analyses requested
- Date and time of laboratory receipt
- Field ID code
- Date and time of collection
- Any comments resulting from inspection for sample rejection

2.5.2. If the time collected for any sample is unspecified and Pace is unable to obtain this information from the customer, the laboratory will use 08:00 as the time sampled. All hold times will be based on this sampling time and qualified accordingly if exceeded.

2.5.3. The LIMS automatically generates a unique identification number for each sample created in the system. The LIMS sample number follows the general convention of 50XXXXXX. This unique identification number is placed on the sample container as a durable label and becomes the link between the laboratory's sample management system and the customer's field identification; it will be a permanent reference number for all future interactions.

2.5.4. Sample labels are printed from the LIMS and affixed to each sample container.

2.5.5. Additional information can be found in SOP S-IN-C-001 *Sample Management* or its equivalent revision or replacement.

2.6. Sample Storage

2.6.1. Additional information on sample storage can be found in SOP S-IN-C-001 *Sample Management* or its equivalent revision or replacement and in SOP S-IN-W-002 *Waste Handling and Management* or its equivalent revision or replacement.

2.6.2. **Storage Conditions**

2.6.2.1. Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross contamination. Volatile samples are stored separately from other samples. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

2.6.2.2. Storage blanks are stored with volatile samples and are used to measure cross-contamination acquired during storage. Laboratories must have documented procedures and criteria for evaluating storage blanks, appropriate to the types of samples being stored.

2.6.2.3. Additional information can be found in SOP S-IN-Q-018 *Monitoring Temperature Controlled Units***.**

2.6.3. **Temperature Monitoring**

2.6.3.1. Samples are taken to the appropriate storage location immediately after sample receipt and check-in procedures are completed. All sample storage areas are located in limited access areas and are monitored to ensure sample integrity.

2.6.3.2. The temperature of each refrigerated storage area is maintained at $\leq 6^{\circ}C$ but above freezing unless state, method or program requirements differ. The temperature of each freezer storage area is maintained at $\leq -10^{o}C$ unless state, method or program requirements differ. The temperature of each storage area is checked and documented each day of use. If the temperature falls outside the acceptable limits, the following corrective actions are taken and appropriately documented:

- The temperature is rechecked after a period of time, usually two hours, to verify temperature exceedance. Corrective action is initiated and documented if necessary.
- The SQM/QM and/or laboratory management are notified if the problem persists.
- The samples are relocated to a proper environment if the temperature cannot be maintained after corrective actions are implemented.
- The affected customers are notified and/or documentation is provided on the final report, if necessary.

2.6.3.3. Additional information can be found in SOP S-IN-Q-018 *Monitoring Temperature Controlled Units*.

2.6.4. **Hazardous Materials**

2.6.4.1. Samples designated by clients upon receipt as pure product or potentially heavily contaminated samples, or samples found to be designated as such following analysis, must be tagged as "hazardous" or "lab pack" and stored separately from other samples.

2.6.5. **Foreign/Quarantined Soils**

2.6.5.1. Foreign soils and soils from domestic USDA quarantined areas must be adequately segregated to prevent cross-contamination and enable proper sample disposal. The USDA requires these samples and by-products to be properly identified and handled and to be treated by an approved procedure prior to disposal or as part of disposal.

2.6.5.2. Additional information regarding USDA regulations and sample handling can be found in the laboratory's SOP S-IN-C-007 *USDA Regulated Soil Handling and Disposal* or its equivalent revision or replacement.

2.7. Subcontracting Analytical Services

2.7.1. Every effort is made to perform all analyses for Pace customers within the laboratory that receives the samples. When subcontracting to a laboratory other than the receiving laboratory, whether inside or outside the Pace network, becomes necessary, a preliminary verbal communication with that laboratory is undertaken. Customers are notified in writing of the laboratory's intention to subcontract any portion of the testing to another laboratory. Work performed under specific protocols may involve special considerations. When possible, subcontracting will be to a TNI-accredited laboratory.

2.7.2. Potential subcontract laboratories must be approved by Pace based on the criteria listed in SOP S-IN-C-003 *Subcontracting Samples* or its equivalent revision or replacement. All sample reports from the subcontracted labs are appended to the applicable Pace final reports.

2.7.3. Any Pace work sent to other labs within the Pace network is handled as inter-regional work and all final reports are labeled clearly with the name of the laboratory performing the work. Any non-TNI work is clearly identified. Pace will not be responsible for analytical data if the subcontract laboratory was designated by the customer.

2.7.4. Additional information can be found in SOP S-IN-C-003 *Subcontracting Samples* or its equivalent revision or replacement.

2.8. Sample Retention and Disposal

2.8.1. Samples, extracts, digestates, and leachates must be retained by the laboratory for the period of time necessary to protect the interests of the laboratory and the customer.

2.8.2. The minimum sample retention time is 45 days from receipt of the samples. Samples requiring thermal preservation may be moved to ambient temperature storage when the hold time is expired, when the report has been delivered, and/or when allowed by the customer, program, or contract. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

2.8.3. After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposal of **hazardous** samples is to return the excess sample to the customer. If it is not feasible to return samples, or the customer requires Pace to dispose of excess samples, proper arrangements will be made for disposal by an approved contractor.

2.8.4. Additional information can be found in SOP S-IN-W-002 *Waste Handling and Management* and SOP S-IN-C-001 *Sample Management* or their equivalent revisions or replacements.

3.0. QUALITY CONTROL PROCEDURES

3.1. Quality Control Samples

3.1.1. The quality control samples described in this section are analyzed per batch as applicable to the method used. Acceptance criteria must be established for all quality control samples and if the acceptance criteria are not met, corrective actions must be performed and samples reanalyzed, or the final report must be appropriately qualified.

3.1.2. Quality control samples must be processed in the same manner as associated client samples.

3.1.3. Please reference the glossary of this Quality Manual for definitions of all quality control samples mentioned in this section.

3.1.4. Any deviations to the policies and procedures governing quality control samples must be approved by the QM/SQM.

3.2. Method Blank

3.2.1. A method blank is a negative control used to assess the preparation/analysis system for possible contamination and is processed through all preparation and analytical steps with its associated client samples. The method blank is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples. Method blanks are not applicable for certain analyses (i.e., pH, flash point, temperature, etc.).

3.2.2. Each method blank is evaluated for contamination. Corrective actions for blank contamination may include the re-preparation and re-analysis of all samples (where possible) and quality control samples. Data qualifiers must be applied to results that are affected by contamination in a method blank.

3.2.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for method blanks.

3.3. Laboratory Control Sample

3.3.1. The Laboratory Control Sample (LCS) is a positive control used to assess the performance of the entire analytical system including preparation and analysis. The LCS is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples.

3.3.2. The LCS contains all analytes required by a specific method or by the customer or regulatory agency, which may not include the full list of target compounds. In the absence of specified components, the laboratory will spike the LCS with the following compounds:

- For multi-peak analytes (e.g. PCBs, technical chlordane, toxaphene), a representative standard will be processed.
- For methods with long lists of analytes, a representative number of target analytes may be chosen. The following criteria is used to determine the number of LCS compounds used:
    - For methods with 1-10 target compounds, the laboratory will spike with all compounds;
    - For methods with 11-20 target compounds, the laboratory will spike with at least 10 compounds or 80%, whichever is greater;

- For methods with greater than 20 compounds, the laboratory will spike with at least 16 compounds.

3.3.3. The LCS is evaluated against the method default or laboratory-derived acceptance limits. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Any sample containing a compound that was 'out-of-control' in the associated LCS must either be re-analyzed with a successful LCS or reported with the appropriate data qualifier. When the result of the LCS exceeds the upper control limit, indicating high bias, associated samples determined to be non-detect may be reported without qualification.

3.3.4. For LCSs containing a large number of analytes, it is statistically likely that a few recoveries will be outside of control limits. This does not necessarily mean that the system is out of control, and therefore no corrective action would be necessary (except for proper documentation). TNI has allowed for a minimum number of marginal exceedances, defined as recoveries that are beyond the LCS control limits (3X the standard deviation) but within than the marginal exceedance limits (4X the standard deviation). The number of allowable exceedances depends on the number of compounds in the LCS. If more analyte recoveries exceed the LCS control limits than is allowed (see below) or if any one analyte exceeds the marginal exceedance limits, then the LCS is considered non-compliant and corrective actions are necessary. The number of allowable exceedances is as follows:

- >90 analytes in the LCS- 5 analytes
- 71-90 analytes in the LCS- 4 analytes
- 51-70 analytes in the LCS- 3 analytes
- 31-50 analytes in the LCS- 2 analytes
- 11-30 analytes in the LCS- 1 analyte
- <11 analytes in the LCS- no analytes allowed out)

3.3.5. A matrix spike (MS) can be used in place of a non-compliant LCS in a batch as long as the MS passes the LCS acceptance criteria. When this happens, full documentation must be made available to the data user. If this is not allowed by a customer or regulatory body, the associated samples must be rerun with a compliant LCS when possible or reported with appropriate data qualifiers.

3.3.6. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for LCSs.

3.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.4.1. A matrix spike (MS) is a positive control used to determine the effect of the sample matrix on compound recovery for a particular method. A matrix spike/matrix spike duplicate (MS/MSD) set or matrix spike/sample duplicate set is processed at a frequency specified in a particular method or as determined by a specific customer request. The MS and MSD consist of the sample matrix that is spiked with known concentrations of target analytes.

3.4.2. The MS and MSD contain all analytes required by a specific method or by the customer or regulatory agency. In the absence of specified components, the laboratory will spike the MS/MSD with the same number of compounds as previously discussed in the LCS section.

3.4.3. A matrix spike and sample duplicate will be performed instead of a matrix spike and matrix spike duplicate when specified by the customer or method or when limited sample volume or weight prohibits the analysis of an MS/MSD set.

3.4.4. The MS and MSD are evaluated against the method or laboratory derived limits. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance; however, is based on method blank and LCS performance, not on MS/MSD recoveries. The spike recoveries give the data user a better understanding of the final results based on their site-specific information.

3.4.5. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for MS/MSDs.

3.5. Sample Duplicate

3.5.1. A sample duplicate is a second portion of sample that is prepared and analyzed in the laboratory along with the first portion. It is used to measure the precision associated with preparation and analysis. A sample duplicate is processed at a frequency specified by the particular method or as determined by a specific customer.

3.5.2. The sample and duplicate are evaluated against the method or laboratory limits for relative percent difference (RPD). Any duplicate that is outside of these limits is considered to be 'out of control' and must be qualified appropriately.

3.5.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for sample duplicates.

3.6. Surrogates

3.6.1. Surrogates are compounds that reflect the chemistry of target analytes and are added to samples for most organic analyses to measure the extraction or purge efficiency and to monitor the effect of the sample matrix on compound recovery.

3.6.2. The surrogates are evaluated against the method or laboratory derived acceptance limits. Any surrogate compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Samples with surrogate failures are typically re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systemic error. An exception to this would be samples that have surrogate recoveries that exceed the upper control limit but have no reportable hits for target compounds. These samples would be reported and qualified to indicate the implied high bias would not affect the final results.

3.6.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for surrogates.

3.7. Internal Standards

3.7.1. Internal Standards are method-specific analytes that are added, as applicable, to every standard, QC sample, and client sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes.

3.7.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for internal standards.

3.8. Limit of Detection (LOD)

3.8.1. Pace laboratories use a documented procedure to determine a limit of detection (LOD) for each analyte of concern in each matrix reported. Unless otherwise noted in a published method, the procedure used by Pace laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. All sample processing steps of the preparation and analytical methods are included in the LOD determination including any clean ups.

3.8.2. Additional information can be found in SOP S-IN-Q-004 *Determination of LOD and LOQ* or its equivalent revision or replacement.

3.9. Limit of Quantitation (LOQ)

3.9.1. A limit of quantitation (LOQ) for every analyte of concern must be determined. For Pace laboratories, this LOQ is referred to as the RL, or Reporting Limit. The RL may or may not be based on the lowest calibration standard concentration used in the initial calibration. Results below the lowest calibration level may not be reported without qualification since the results would not be substantiated by a calibration standard. For methods with a determined LOD, results can be reported below the LOQ but above the LOD if they are properly qualified (e.g., J flag).

3.9.2. Additional information can be found in SOP S-IN-Q-004 *Determination of LOD and LOQ* or its equivalent revision or replacement.

3.10. Estimate of Analytical Uncertainty

3.10.1. Pace can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling or sample matrix. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, Pace laboratories base this estimation on the recovery data obtained from the Laboratory Control Samples (LCS). The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the SOP S-IN-Q-031 *Estimation of Measurement Uncertainty* or its equivalent revision or replacement.

3.10.2. The measurement of uncertainty is provided only on request by the customer, as required by specification or regulation and when the result is used to determine conformance within a specification limit.

3.11. Proficiency Testing (PT) Studies

3.11.1. Pace laboratories participate in a defined proficiency testing (PT) program. PT samples are obtained from NIST-approved providers and analyzed and reported at a minimum of two times per year for the relevant fields of testing per matrix.

3.11.2. The laboratory initiates an investigation whenever PT results are determined to be "Not Acceptable" by the PT provider. All findings and corrective actions taken are reported to the SQM/QM or their designee. A corrective action plan is initiated and, when required, this report is sent to the appropriate state accreditation agencies for their review. Additional PTs will be analyzed and reported as needed for certification purposes.

3.11.3. Additional information can be found in SOP S-IN-Q-010 *Proficiency Testing Program* or its equivalent revision or replacement.

3.12. Rounding and Significant Figures

3.12.1. In general, the Pace laboratories report data to no more than three significant figures. Therefore, all measurements made in the analytical process must reflect this level of precision. In the event that a variable that contributes to the final result has less than three significant figures of precision, the final result must be reported with no more significant figures than that of the parameter in question. The rounding rules listed below are descriptive of the LIMS and not necessarily of any supporting program such as Excel.

3.12.2. **Rounding:** Pace - Indianapolis follows the odd / even guidelines for rounding numbers:

- If the figure following the one to be retained is less than five, that figure is dropped and the retained ones are not changed (with three significant figures, 2.544 is rounded to 2.54).
- If the figure following the ones to be retained is greater than five, that figure is dropped and the last retained one is rounded up (with three significant figures, 2.546 is rounded to 2.55).
- If the figure following the ones to be retained is five and if there are no figures other than zeros beyond that five, then the five is dropped and the last figure retained is unchanged if it is even and rounded up if it is odd (with three significant figures, 2.525 is rounded to 2.52 and 2.535 is rounded to 2.54).

3.12.3. **Significant Figures**

3.12.3.1. Pace - Indianapolis follows the following convention for reporting to a specified number of significant figures. Unless specified by federal, state, or local requirements or on specific request by a customer, the laboratory reports:

Values > 10 – Reported to 3 significant figures
Values ≤ 10 – Reported to 2 significant figures

3.13. Retention Time Windows

3.13.1. When chromatographic conditions are changed, retention times and analytical separations are often affected. As a result, two critical aspects of any chromatographic method are the determination and verification of retention times and analyte separation. Retention time windows must be established for the identification of target analytes. The retention times of all target analytes in all calibration verification standards must fall within appropriately determined retention time windows. If an analyte falls outside the retention time window in an ICV or CCV, new absolute

retention time windows must be calculated, unless instrument maintenance fixes the problem. New retention time windows must be established when column geometry is affected by maintenance.

3.13.2. Please reference method-specific SOPs for the proper procedure for establishing retention time windows.

3.14. Analytical Method Validation and Instrument Validation

3.14.1. In some situations, Pace develops and validates methodologies that may be more applicable to a specific problem or objective. When non-standard methods are required for specific projects or analytes of interest, when the laboratory develops or modifies a method, or when the laboratory brings new instrumentation online, the laboratory validates the method and/or instrument prior to applying it to customer samples. Method validity is established by meeting criteria for precision and accuracy as established by the data quality objectives specified by the end user of the data. The laboratory records the validation procedure, the results obtained and a statement as to the usability of the method. The minimum requirements for method or instrument validation include evaluation of sensitivity, quantitation, precision, bias, and selectivity of each analyte of interest.

3.15. Regulatory and Method Compliance

3.15.1. It is Pace policy to disclose in a forthright manner any detected noncompliance affecting the usability of data produced by our laboratories. The laboratory will notify customers within 30 days of fully characterizing the nature of the nonconformance, the scope of the nonconformance and the impact it may have on data usability.

4.0. DOCUMENT MANAGEMENT AND CHANGE CONTROL

4.1. Document Management

4.1.1. Additional information can be found in SOP S-IN-Q-002 *Document Control and Management* or its equivalent revision or replacement. Information on Pace's policy for electronic signatures can also be found in this SOP.

4.1.2. Pace has an established procedure for managing documents that are part of the quality system.

4.1.3. A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents.

4.1.4. Each managed document is uniquely identified to include the date of issue, the revision identification, page numbers, the total number of pages and the issuing authorities. For complete information on document numbering, refer to SOP S-ALL-Q-003 *Document Numbering*.

4.1.5. **Quality Assurance Manual (QAM):** The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for Pace. The base QAM template is distributed by the Corporate Environmental Quality Department to each of the SQMs/QMs. The local management personnel modify the necessary and permissible sections of the base template then applicable lab staff will sign the Quality Assurance Manual. Each SQM/QM is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual template is reviewed on an annual basis and revised accordingly by the Corporate Quality office.

4.1.6. **Standard Operating Procedures (SOPs)**

4.1.6.1. SOPs are reviewed every two years at a minimum; although, a more frequent review may be required by some state or federal agencies or customers. If no revisions are made based on this review, documentation of the review itself is made by the addition of new signatures on the cover page. If revisions are made, documentation of the revisions is made in the revisions section of each SOP and a new revision number is applied to the SOP. This provides a historical record of all revisions.

4.1.6.2. All copies of superseded SOPs are removed from general use and the original copy of each SOP is archived for audit or knowledge preservation purposes. This ensures that all Pace employees use the most current version of each SOP and provides the SQM/QM with a historical record of each SOP.

4.1.6.3. Additional information can be found in SOP S-IN-Q-001 *Preparation of SOPs* or its equivalent revision or replacement.

4.2. Document Change Control

4.2.1. Additional information can be found in SOP S-IN-Q-002 *Document Control and Management* or its equivalent revision or replacement.

4.2.2. Changes to managed documents are reviewed and approved in the same manner as the original review. Any revision to a document requires the approval of the applicable signatories. After

revisions are approved, a revision number is assigned and the previous version of the document is officially retired.

4.2.3. All copies of the previous document are replaced with copies of the revised document and the superseded copies are destroyed or archived. All affected personnel are advised that there has been a revision and any necessary training is scheduled.

5.0. EQUIPMENT AND MEASUREMENT TRACEABILITY

5.1. Standards and Traceability

5.1.1. Each Pace facility retains pertinent information for standards, reagents, and chemicals to assure traceability to a national standard. This includes documentation of purchase, receipt, preparation, and use.

5.1.2. Upon receipt, all purchased standard reference materials are recorded into a standard logbook or database and assigned a unique identification number. The entries include the facility's unique identification number, the chemical name, manufacturer name, manufacturer's identification numbers, receipt date, and expiration date. Vendor's certificates of analysis for all standards, reagents, or chemicals are retained for future reference.

5.1.3. Subsequent preparations of intermediate or working solutions are also documented in a standard logbook or database. These entries include the stock standard name and lot number, the manufacturer name, the solvents used for preparation, the solvent lot number and manufacturer, the preparation steps, preparation date, expiration dates, preparer's initials, and a unique Pace identification number. This number is used in any applicable sample preparation or analysis logs so the standard can be traced back to the standard preparation record. This process ensures traceability back to the national standard.

5.1.4. Prepared standard or reagent containers include the Pace identification number, the standard or chemical name, and expiration date. The date of preparation, concentration with units, and the preparer's initials can be determined by tracing the standard or reagent ID through the standard log database.

5.1.5. All initial calibrations must be verified with a standard obtained from a second manufacturer or a separate lot prepared independently by the same manufacturer, unless client-specific QAPP requirements state otherwise.

5.1.6. Additional information concerning the procurement of standards and reagent and their traceability can be found in the SOP S-IN-Q-025 *Standard and Reagent Management and Traceability* or its equivalent revision or replacement.

5.2. General Analytical Instrument Calibration Procedures

5.2.1. All applicable instrumentation are calibrated or checked before use to ensure proper functioning and verify that laboratory, client and regulatory requirements are met. All calibrations are performed by, or under the supervision of, an experienced analyst at scheduled intervals against either certified standards traceable to recognized national standards or reference standards whose values have been statistically validated.

5.2.2. Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in a narrative. The highest calibration standard is the highest concentration for which quantitative data may be reported. Data reported above this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in the narrative.

5.2.3. Results from all calibration standards analyzed must be included in constructing the calibration curve with the following exceptions:

5.2.3.1. The lowest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done on an individual analyte basis. The reporting limit must be adjusted to the lowest concentration included in the calibration curve;

5.2.3.2. The highest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done an individual analyte basis. The upper limit of quantitation must be adjusted to the highest concentration included in the calibration curve;

5.2.3.3. Multiple points from either the high end or the low end of the calibration curve may be excluded as long as the remaining points are contiguous in nature and the minimum number of levels remains as established by method or standard operating procedure. The reporting limit or quantitation range, whichever is appropriate, must be adjusted accordingly;

5.2.3.4. Results from a concentration level between the lowest and highest calibration levels can only be excluded from an initial calibration curve for a documentable and acceptable cause with approval from the SGM/GM, AGM, or the local SQM/QM. An acceptable cause is defined as an obvious sample introduction issue that resulted in no response or very low response, documentation of an incorrectly prepared standard, or a documented response of a single standard that is more than twice the expected value of that standard. The results for all analytes in the calibration standard are to be excluded and the remaining number of concentration levels must meet the minimum number of standards established by the method and standard operating procedure.

5.2.4. Instrumentation or support equipment that cannot be calibrated to specification or is otherwise defective is clearly labeled as out-of-service until it has been repaired and tested to demonstrate it meets the laboratory's specifications. All repair and maintenance activities including service calls are documented in the maintenance log. Equipment sent off-site for calibration testing is packed and transported to prevent breakage and is in accordance with the calibration laboratory's recommendations.

5.2.5. In the event that recalibration of a piece of test equipment indicates the equipment may have been malfunctioning during the course of sample analysis, an investigation is performed. The results of the investigation along with a summary of the information reviewed are documented and maintained by the quality manager. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed. This allows for sufficient investigation and review of documentation to determine the impact on the analytical results. Instrumentation found to be consistently out of calibration is either repaired and positively verified or taken out of service and replaced.

5.2.6. Raw data records are retained to document equipment performance. Sufficient raw data is retained to reconstruct the instrument calibration and explicitly connect the continuing calibration verification to the initial calibration.

5.2.7. Please reference method-specific SOPs for more detailed calibration information.

5.3. Support Equipment Calibration and Verification Procedures

5.3.1. All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use, as applicable. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until brought back into control. Additional information regarding calibration and maintenance of support equipment can be found in SOP S-IN-Q-157 *Support Equipment* or its equivalent revision or replacement.

5.3.2. On each day of use, balances, ovens, refrigerators, incubators, freezers and water baths are checked in the expected range of use with NIST traceable references in order to ensure the equipment meets laboratory specifications. These checks are documented appropriately. This applies mainly to thermometers within temperature-controlled units and balances.

5.3.3. **Analytical Balances**

5.3.3.1. Each analytical balance is calibrated or verified at least annually by a qualified service technician. The calibration of each balance is verified each day of use with weights traceable to NIST bracketing the range of use. Working calibration weights are ASTM Class 1 or other class weights that have been calibrated against a reference weight set that is re-certified every 5 years, at a minimum, by the manufacturer or other qualified vendor, against a NIST traceable reference. If balances are calibrated by an external agency, verification of their weights must be available. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the local Quality department.

5.3.4. **Thermometers**

5.3.4.1. Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and are re-certified every 3 years, at a minimum by the manufacturer or other qualified vendor with equipment directly traceable to NIST.

5.3.4.2. Working thermometers and temperature sensors are compared with the reference thermometers annually according to corporate metrology procedures. Alternatively, working thermometers may be replaced with new thermometers annually in lieu of verification or may be verified annually by the manufacturer or other qualified vendor. Each thermometer is individually numbered and assigned a correction factor, when applicable, based on the NIST reference source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

5.3.4.3. Laboratory thermometer inventory and calibration data are maintained in the local Quality department.

5.3.5. **pH/Electrometers**

5.3.5.1. The meter is calibrated before use each day, at a minimum, using fresh buffer solutions.

5.3.5.2. The pH electrode is inspected daily and cleaned, filled or replaced as needed.

5.3.6. **Spectrophotometers**

5.3.6.1. During use, spectrophotometer performance is checked at established frequencies in analysis sequences against initial calibration verification (ICV) and continuing calibration verification (CCV) standards.

5.3.7. **Mechanical Volumetric Dispensing Devices**

5.3.7.1. Mechanical volumetric dispensing devices including bottle top dispensers dispensing critical volumes, pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis.

5.3.7.2. Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP S-IN-Q-157 *Support Equipment* or its equivalent revision or replacement.

5.4. Instrument/Equipment Maintenance

5.4.1. The objectives of the Pace Analytical maintenance program are twofold: to establish a system of instrument care that maintains instrumentation and equipment at required levels of calibration and sensitivity, and to minimize loss of productivity due to repairs.

5.4.2. The Operations Manager and/or department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems, and coordinate instrument repair and maintenance. Analysts have the primary responsibility to perform routine maintenance.

5.4.3. To minimize downtime and interruption of analytical work, preventative maintenance may routinely performed on each analytical instrument. Up-to-date instructions on the use and maintenance of equipment are available to staff in the department where the equipment is used.

5.4.4. Department manager/supervisors are responsible for maintaining an adequate inventory of spare parts required to minimize equipment downtime. This inventory includes parts and supplies that are subject to frequent failure, have limited lifetimes, or cannot be obtained in a timely manner should a failure occur.

5.4.5. All major equipment and instrumentation items are uniquely identified to allow for traceability. Equipment/instrumentation is, unless otherwise stated, identified as a system and not as individual pieces. The laboratory maintains equipment records that include the following:

- The name of the equipment and its software
- The manufacturer's name, type, and serial number
- Approximate date received and date placed into service
- Current location in the laboratory
- Condition when received (new, used, etc.)
- Copy of any manufacturer's manuals or instructions
- Dates and results of calibrations and next scheduled calibration (if known)
- Details of past maintenance activities, both routine and non-routine
- Details of any damage, modification or major repairs

5.4.6. All instrument maintenance is documented in maintenance logbooks that are assigned to each particular instrument or system.

5.4.7. The maintenance log entry must include a summary of the results of that analysis and verification by the analyst that the instrument has been returned to an in-control status. In addition, each entry must include the initials of the analyst making the entry, the dates the maintenance actions were performed, and the date the entry was made in the maintenance logbook, if different from the date(s) of the maintenance.

5.4.8. Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used to analyze customer samples until it has been repaired and shown to perform satisfactorily. In the event of instrumentation failure, to avoid hold time issues, the lab may subcontract the necessary samples to another Pace lab or to an outside subcontract lab if possible.

6.0. CONTROL OF DATA

Analytical results processing, verification, and reporting are procedures employed that result in the delivery of defensible data. These processes include, but are not limited to, calculation of raw data into final concentration values, review of results for accuracy, evaluation of quality control criteria and assembly of technical reports for delivery to the data user.

All analytical data undergo a documented multi-tier review process prior to being reported to the customer. This section describes procedures used for translating raw analytical data into accurate final sample reports as well as Pace data storage policies.

When analytical, field, or product testing data is generated, it is documented appropriately. The resulting logbooks and other laboratory records are kept in accordance with each facility's SOP for documentation storage and archival In this case, the laboratory must ensure that there are sufficient redundant electronic copies so no data is lost due to unforeseen computer issues

6.1. Primary Data Review

6.1.1. The primary analyst is responsible for initial data reduction and data review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting non-conformances in logbooks or as footnotes or narratives, and uploading analytical results into the LIMS. Data review checklists, either hardcopy or electronic, are used to document the primary data review process. The primary analyst must be clearly identified in all applicable logbooks, spreadsheets, LIMS fields, and data review checklists.

6.1.2. The primary analyst compiles the initial data for secondary data review. This compilation must include sufficient documentation for secondary data review.

6.1.3. Additional information regarding data review procedures can be found in SOP S-IN-Q-016 *Data Review* or its equivalent revision or replacement, as well as in SOP S-ALL-Q-016 *Manual Integration* or its equivalent revision or replacement.

6.2. Secondary Data Review

6.2.1. Secondary data review is the process of examining data and accepting or rejecting it based on pre-defined criteria. This review step is designed to ensure that reported data are free from calculation and transcription errors, that quality control parameters are evaluated, and that any non-conformances are properly documented.

6.2.2. The completed data from the primary analyst is sent to a designated qualified secondary data reviewer, which must be someone other than the primary analyst. The secondary data reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations, data quantitation and applicable data qualifiers. The reviewer validates the data entered into the LIMS and documents approval of manual integrations. Data review checklists, either hardcopy or electronic, are used to document the secondary data review process.

6.2.3. Additional information regarding data review procedures can be found in SOP S-IN-Q-016 *Data Review* or its equivalent revision or replacement, as well as in SOP S-ALL-Q-016 *Manual Integration* or its equivalent revision or replacement.

6.3. Data Reporting

6.3.1. Data for each analytical fraction pertaining to a particular Pace project number are delivered to the Project Manager for assembly into the final report. All points mentioned during technical and QC reviews are included in data qualifiers on the final report or in a separate case narrative if there is potential for data to be impacted.

6.3.2. Final reports are prepared according to the level of reporting required by the customer and can be transmitted to the customer via hardcopy or electronic deliverable. A standard Pace final report consists of the following components:

6.3.2.1. A title which designates the report as “Final Report”, “Laboratory Results”, “Certificate of Results”, etc.;

6.3.2.2. Name and address of laboratory and/or subcontractor laboratories, if used;

6.3.2.3. Phone number and name of laboratory contact to where questions can be referred;

6.3.2.4. A unique identification number for the report. The pages of the report are numbered and a total number of pages is indicated;

6.3.2.5. Name and address of customer and name of project;

6.3.2.6. Unique identification of samples analyzed as well as customer sample IDs;

6.3.2.7. Date and time of sample collection, sample receipt and sample analysis;

6.3.2.8. Identification of the test methods used;

6.3.2.9. Qualifiers to the analytical data, if needed or applicable;

6.3.2.10. Identification of whether results are reported on a dry-weight or wet-weight basis;

6.3.2.11. Reporting limits used;

6.3.2.12. Final results or measurements;

6.3.2.13. A signature and title, electronic or otherwise, of person accepting responsibility for the content of the report;

6.3.2.14. Date report was issued;

6.3.2.15. A statement clarifying that the results of the report relate only to the samples tested or to the samples as they were received by the laboratory;

6.3.2.16. A statement indicating that the report must not be reproduced except in full, without the written approval of the laboratory;

6.3.3. Any changes made to a final report shall be designated as “Revised” or equivalent wording. The laboratory must keep sufficient archived records of all laboratory reports and revisions. For higher levels of data deliverables, a copy of all supporting raw data is sent to the customer along with a final report of results. Pace will provide electronic data deliverables (EDD) as required by contracts or upon customer request.

6.3.4. Customer data that requires transmission by telephone, telex, facsimile or other electronic means undergoes appropriate steps to preserve confidentiality.

6.3.5. The following positions are the only approved signatories for Pace final reports:

- Senior General Manager
- General Manager
- Assistant General Manager
- Senior Quality Manager
- Quality Manager
- Client Services Manager
- Project Manager
- Project Coordinator

6.3.6. Additional information regarding final reports and data deliverables can be found in SOP S-IN-Q-046 *Final Report and Data Deliverable Contents* or its equivalent revision or replacement.

6.4. Data Security

6.4.1. All data including electronic files, logbooks, extraction/digestion/distillation worksheets, calculations, project files and reports, and any other information used to produce the technical report are maintained secured and retrievable by the Pace facility.

6.5. Data Archiving

6.5.1. All records compiled by Pace are archived in a suitable, limited-access environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. TNI-related records will be made readily available to accrediting authorities. Access to archived data is controlled by the Quality Department.

6.5.2. Records that are computer-generated have either a hard copy or electronic backup copy. Hardware and software necessary for the retrieval of electronic data is maintained with the applicable records. Archived electronic records are stored protected against electronic and/or magnetic sources.

6.5.3. In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained per the purchase agreement. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

6.6. Data Disposal

6.6.1. Data that has been archived for the facility's required storage time may be disposed of in a secure manner by shredding, returning to customer, or utilizing some other means that does not jeopardize data confidentiality. Records of data disposal will be archived for a minimum of five years unless superseded by federal, contractual, and/or accreditation requirements. Data disposal includes any preliminary or final reports, raw analytical data, logs or logbooks, and electronic files.

7.0. QUALITY SYSTEM AUDITS AND REVIEWS

7.1. Internal Audits

7.1.1. Responsibilities

7.1.1.1. The SQM/QM is responsible for managing, assigning and/or conducting internal audits in accordance with a predetermined schedule and procedure. Since internal audits represent an independent assessment of laboratory functions, the auditor must be independent from laboratory operations to ensure objectivity. The auditor must be trained, qualified, and familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation. The SQM/QM evaluates audit observations and verifies the completion of corrective actions. In addition, a periodic corporate audit will be conducted. The corporate audits will focus on the effectiveness of the Quality System as outlined in this manual but may also include other quality programs applicable to an individual laboratory.

7.1.1.2. Additional information can be found in SOP S-IN-Q-011 *Internal and External Audits* or its equivalent revision or replacement.

7.1.2. Scope and Frequency of Internal Audits

7.1.2.1. The complete internal audit process consists of the following four sections, at a minimum:

- Raw Data Review audits- conducted according to a schedule per local SQM/QM. A certain number of these data review audits may be conducted per quarter to accomplish this yearly schedule;
- Quality System audits- considered the traditional internal audit function and includes analyst interviews to help determine whether practice matches method requirements and SOP language;
- Final Report reviews;
- Corrective Action Effectiveness Follow-up

7.1.2.2. Internal systems audits are conducted annually at a minimum. The scope of these audits includes evaluation of specific analytical departments or a specific quality related system as applied throughout the laboratory.

7.1.2.3. Where the identification of non-conformities or departures cast doubt on the laboratory's compliance with its own policies and procedures, the lab must ensure that the appropriate areas of activity are audited as soon as possible.

7.1.2.4. Certain projects may require an internal audit to ensure laboratory conformance to site work plans, sampling and analysis plans, QAPPs, etc.

7.1.2.5. The laboratory, as part of their overall internal audit program, ensures that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery and reporting of potential data integrity issues are handled in a confidential manner. All investigations that result in findings of inappropriate activity are fully documented, including the source of the problem, the samples and customers affected the impact on the data, the corrective actions taken by the laboratory, and identification of final reports that were re-issued. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed.

7.1.3. **Internal Audit Reports and Corrective Action Plans**

7.1.3.1. A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. The Quality Department auditor writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

7.1.3.2. When audit findings cast doubt on the effectiveness of the operations or on the correctness of validity of the laboratory's environmental test results, the laboratory will take timely corrective action and notify the customer in writing within three business days, if investigations show that the laboratory results may have been affected.

7.1.3.3. Additional information can be found in SOP S-IN-Q-011 *Internal and External Audits* or its equivalent revision or replacement.

7.2. External Audits

7.2.1. Pace laboratories are audited regularly by regulatory agencies to maintain laboratory certifications and by customers to maintain appropriate specific protocols.

7.2.2. External audit teams review the laboratory to assess the effectiveness of quality systems. The SQM/QM host the external audit team and assist in facilitation of the audit process. After the audit, the external auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the SQM/QM, who provides a written response to the external audit team. The SQM/QM follows-up with the laboratory staff to ensure corrective actions are implemented and that the corrective action was effective.

7.3. Annual Managerial Review

7.3.1. A managerial review of Management and Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements. Additional information can be found in SOP S-ALL-Q-015 *Review of Laboratory Management System* or its equivalent revision or replacement.

7.3.2. The managerial review must include the following topics of discussion:

- Suitability of quality management policies and procedures
- Manager/Supervisor reports
- Internal audit results
- Corrective and preventive actions
- External assessment results
- Proficiency testing studies
- Sample capacity and scope of work changes
- Customer feedback, including complaints
- Recommendations for improvement,
- Other relevant factors, such as quality control activities, resources, staffing, and safety/waste activities.

7.3.3. This managerial review must be documented for future reference by the SQM/QM and copies of the report are distributed to laboratory staff. Results must feed into the laboratory planning system and must include goals, objectives, and action plans for the coming year. The laboratory shall ensure that any actions identified during the review are carried out within an appropriate and agreed upon timescale.

8.0. CORRECTIVE ACTION

Additional information can be found in SOP S-IN-Q-012 *Corrective and Preventive Actions* or its equivalent revision or replacement.

During the process of sample handling, preparation, and analysis, or during review of quality control records, or during reviews of non-technical portions of the lab, certain occurrences may warrant the necessity of corrective actions. These occurrences may take the form of analyst errors, deficiencies in quality control, method deviations, or other unusual circumstances. The Quality System of Pace provides systematic procedures for the documentation, monitoring, completion of corrective actions, and follow-up verification of the effectiveness of these corrective actions. This can be done using Pace's LabTrack system or other system that lists at a minimum, the deficiency by issue number, the deficiency source, responsible party, root cause, resolution, due date, and date resolved.

8.1. Corrective and Preventive Action Documentation

8.1.1. The following items are examples of sources of laboratory deviations or non-conformances that may warrant some form of documented corrective action:

- Internal Laboratory Non-Conformance Trends
- Proficiency Testing Sample Results
- Internal and External Audits
- Data or Records Review
- Client Complaints
- Client Inquiries
- Holding Time violations

8.1.2. Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency or it may be a more formal documentation. This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

8.1.3. The person who discovers the deficiency or non-conformance initiates the corrective action documentation within LabTrack. The documentation must include the affected projects and sample numbers, the name of the applicable Project Manager, the customer name, and any other pertinent information. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

8.1.4. **Root Cause Analysis**: Laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance, assigning responsibilities, determining if the condition is significant, and investigating the root cause of the nonconformance. General non-conformance investigative techniques follow the path of the sample through the process looking at each individual step in detail. The root cause must be documented within LabTrack.

8.1.5. Based on the determined root cause(s), the lab implements applicable corrective actions and verifies their effectiveness. In the event that analytical testing or results do not conform to documented

laboratory policies or procedures Project Management will notify the customer of the situation and will advise of any ramifications to data quality if impacted.

8.2. Corrective Action Completion

8.2.1. Internal Laboratory Non-Conformance Trends

8.2.1.1. There are several types of non-conformance trends that may occur in the laboratory that would require the initiation of a corrective action report. Laboratories may choose to initiate a corrective action for all instances of one or more of these categories if they so choose, however the intent is that each of these would be handled according to its severity; one time instances could be handled with a footnote or qualifier whereas a systemic problem with any of these categories may require an official corrective action process. These categories, as defined in the Corrective Action SOP are as follows:

- Login error
- Preparation Error
- Contamination
- Calibration Failure
- Internal Standard Failure
- LCS Failure
- Laboratory accident
- Spike Failure
- Instrument Failure
- Final Reporting/Data Entry error

8.2.2. PE/PT Sample Results

8.2.2.1. Any PT result assessed as "not acceptable" requires an investigation and applicable corrective actions. The operational staff is made aware of the PT failures and they are responsible for reviewing the applicable raw data and calibrations and list possible causes for error. The SQM/QM reviews their findings and initiates another external PT sample or an internal PT sample to try and correct the previous failure. Replacement PT results must be monitored by the SQM/QM and reported to the applicable regulatory authorities.

8.2.2.2. Additional information, such as requirements regarding time frames for reporting failures to states, makeup PTs, and notifications of investigations, can be found in SOP S-IN-Q-010 *Proficiency Testing Program* or its equivalent revision or replacement.

8.2.3. Internal and External Audits

8.2.3.1. The SQM/QM or designee is responsible for documenting all audit findings and their corrective actions. This documentation must include the initial finding, the persons responsible for the corrective action, the due date for responding to the auditing body, the root cause of the finding, and the corrective actions needed for resolution. The SQM/QM or designee is also responsible for providing any back-up documentation used to demonstrate that a corrective action has been completed.

8.2.4. **Data Review**

8.2.4.1. In the course of performing primary and secondary review of data or in the case of raw data reviews, errors may be found which require corrective actions. Any finding that affects the quality of the data requires some form of corrective action, which may include revising and re-issuing of final reports.

8.2.5. **Client Complaints**

8.2.5.1. Project Managers are responsible for issuing corrective action requests, when warranted, for customer complaints. As with other corrective actions, the possible causes of the problem are listed and the form is passed to the appropriate analyst or supervisor for investigation. After potential corrective actions have been determined, the Project Manager reviews the corrective action form to ensure all customer needs or concerns are being adequately addressed.

8.2.6. **Client Inquiries**

8.2.6.1. When an error on the customer's final report is discovered, the Project Manager is responsible for initiating a formal corrective action form that describes the failure (e.g., incorrect analysis reported, reporting units are incorrect, or reporting limits do not meet objectives). The Project Manager is also responsible for revising the final report if necessary and submitting it to the customer.

8.2.7. **Holding Time Violations**

8.2.7.1. In the event that a holding time has been missed due to laboratory error, the analyst or supervisor must complete formal corrective action. The Project Manager and the SQM/QM must be made aware of all holding time violations due to laboratory error.

8.2.7.2. The Project Manager must contact the customer in order that appropriate decisions are made regarding the hold time excursion and the ultimate resolution is then documented and included in the customer project file.

9.0. GLOSSARY

The source of some of the definitions is indicated previous to the actual definition (e.g., TNI, DoD).

Terms and Definitions	
3P Program	The Pace continuous improvement program that focuses on Process, Productivity, and Performance. Best Practices are identified that can be used by all Pace labs.
Acceptance Criteria	TNI- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. DoD- Refers to accreditation in accordance with the DoD ELAP.
Accreditation Body (AB)	TNI- The organization having responsibility and accountability for environmental laboratory accreditation and which grants accreditation under this program. DoD- Entities recognized in accordance with the DoD-ELAP that are required to operate in accordance with ISO/IEC 17011, *Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies*. The AB must be a signatory, in good standing, to the International Laboratory Accreditation Cooperation (ILAC) mutual recognition arrangement (MRA) that verifies, by evaluation and peer assessment, that its signatory members are in full compliance with ISO/IEC 17011 and that its accredited laboratories comply with ISO/IEC 17025.
Accuracy	TNI- The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Activity, Absolute	TNI- Rate of nuclear decay occurring in a body of material, equal to the number of nuclear disintegrations per unit time. NOTE: Activity (absolute) may be expressed in becquerels (Bq), curies (Ci), or disintegrations per minute (dpm), and multiples or submultiples of these units.
Activity, Areic	TNI- Quotient of the activity of a body of material and its associated area.
Activity, Massic	TNI- Quotient of the activity of a body of material and its mass; also called specific activity.
Activity, Volumic	TNI- Quotient of the activity of a body of material and its volume; also called activity concentration. NOTE: In this module [TNI Volume 1, Module 6], unless otherwise stated, references to activity shall include absolute activity, areic activity, massic activity, and volumic activity.
Activity Reference Date	TNI- The date (and time, as appropriate to the half-life of the radionuclide) to which a reported activity result is calculated. NOTE: The sample collection date is most frequently used as the Activity Reference Date for environmental measurements, but different programs may specify other points in time for correction of results for decay and ingrowth.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.

American Society for Testing and Materials (ASTM)	An international standards organization that develops and publishes voluntary consensus standards for a wide range of materials, products, systems and services.
Analysis	DoD- A combination of sample preparation and instrument determination.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI- The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.
Analyte	TNI- A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed. DoD- The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together.
Analytical Method	DoD- A formal process that identifies and quantifies the chemical components of interest (target analytes) in a sample.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Annual (or Annually)	Defined by Pace as every 12 months ± 30 days.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- An all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance conducted on-site.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	A process in which a sample is converted to free atoms.
Audit	TNI- A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Batch	TNI- Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.
Batch, Radiation Measurements (RMB)	TNI- An RMB is composed of 1 to 20 environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameter, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections). The maximum time between the start of processing of the first and last in an RMB is 14 calendar days.
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI and DoD- A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results (See Method Blank). DoD- Blank samples are negative control samples, which typically include field blank samples (e.g., trip blank, equipment (rinsate) blank, and temperature blank) and laboratory blank samples (e.g., method blank, reagent blank, instrument blank, calibration blank, and storage blank).
Blind Sample	A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.

Calibration	TNI- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.
Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.
Calibration Range	DoD- The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of Custody Form (COC)	TNI- Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100

Confirmation	TNI- Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second-column confirmation; alternate wavelength; derivatization; mass spectral interpretation; alternative detectors; or additional cleanup procedures. DoD- Includes verification of the identity and quantity of the analyte being measured by another means (e.g., by another determinative method, technology, or column). Additional cleanup procedures alone are not considered confirmation techniques.
Conformance	An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.
Congener	A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a Calibration Verification Standard (CVS) in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Continuous Improvement Plan (CIP)	The delineation of tasks for a given laboratory department or committee to achieve the goals of that department.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)

Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Correction	DoD- Action taken to eliminate a detected non-conformity.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence. A root cause analysis may not be necessary in all cases.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Critical Value	TNI- Value to which a measurement result is compared to make a detection decision (also known as critical level or decision level). NOTE: The Critical Value is designed to give a specified low probability α of false detection in an analyte-free sample, which implies that a result that exceeds the Critical Value, gives high confidence (1 – α) that the radionuclide is actually present in the material analyzed. For radiometric methods, α is often set at 0.05.
Customer	DoD- Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.
Data Integrity	TNI- The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form.
Definitive Data	DoD- Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.
Demonstration of Capability (DOC)	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate analytical results by a specific method that meet measurement quality objectives (e.g., for precision and bias).
Department of Defense (DoD)	An executive branch department of the federal government of the United States charged with coordinating and supervising all agencies and functions of the government concerned directly with national security.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.

Detection Limit (DL) for Safe Drinking Water Act (SDWA) Compliance	TNI- Laboratories that analyze drinking-water samples for SDWA compliance monitoring must use methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25.c as the radionuclide concentration, which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).
Deuterated Monitoring Compounds (DMCs)	DoD- SIM specific surrogates as specified for GC/MS SIM analysis.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat and acid) to convert the target analytes in the sample to a more easily measured form.
Document Control	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.
Documents	DoD- Written components of the laboratory management system (e.g., policies, procedures, and instructions).
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	A solvent used to carry the components of a mixture through a stationary phase.
Elute	To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental Protection Agency (EPA)	An agency of the federal government of the United States which was created for the purpose of protecting human health and the environment by writing and enforcing regulations based on laws passed by Congress.

Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Extracted Internal Standard Analyte	Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution methods.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	DoD- A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.
False Positive	DoD- A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Field of Proficiency Testing (FoPT)	TNI- Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges and acceptance criteria have been established by the PTPEC.

Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive, negative, or neutral and is normally accompanied by specific examples of the observed condition. The finding must be linked to a specific requirement (e.g., this standard, ISO requirements, analytical methods, contract specifications, or laboratory management systems requirements).
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation to analysis, as appropriate.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Improper Actions	DoD- Intentional or unintentional deviations from contract-specified or method-specified analytical practices that have not been authorized by the customer (e.g., DoD or DOE).
Incremental Sampling Method (ISM)	Soil preparation for large volume (1 kg or greater) samples.

In-Depth Data Monitoring	TNI- When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	DoD- Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.
Injection Internal Standard Analyte	Isotopically labeled analogs of analytes of interest (or similar in physiochemical properties to the target analytes but with a distinct response) to be quantitated. Added to all blanks, standards, samples and batch QC after extraction and prior to analysis.
Instrument Blank	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
Interference, spectral	Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.
Internal Standard	TNI and DoD- A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

International Organization for Standardization (ISO)	An international standard-setting body composed of representatives from various national standards organizations.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.
Laboratory	A body that calibrates and/or tests.
Laboratory Control Sample (LCS)	TNI- (also known as laboratory fortified blank (LFB), spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	DoD- The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
LabTrack	Database used by Pace to store and track corrective actions and other laboratory issues.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody (COC) Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.
Limit(s) of Detection (LOD)	TNI- The minimum result, which can be reliably discriminated from a blank with predetermined confidence level. DoD- The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.

Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.
Linear Dynamic Range	DoD- Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	TNI- A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.
Management	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	System to establish policy and objectives and to achieve those objectives.
Manager (however named)	The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
May	EPA – The word "may" is used to provide guidance on aspects of the method that are useful but not essential.
Measurement Performance Criteria (MPC)	DoD- Criteria that may be general (such as completion of all tests) or specific (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined criteria.

Measurement Quality Objective (MQO)	TNI- The analytical data requirements of the data quality objectives are project- or program-specific and can be quantitative or qualitative. MQOs are measurement performance criteria or objectives of the analytical process. Examples of quantitative MQOs include statements of required analyte detectability and the uncertainty of the analytical protocol at a specified radionuclide activity, such as the action level. Examples of qualitative MQOs include statements of the required specificity of the analytical protocol, e.g., the ability to analyze for the radionuclide of interest given the presence of interferences.
Measurement System	TNI- A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the sample preparation and test and the operator(s).
Measurement Uncertainty	DoD- An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty.
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI- A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	TNI- One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Method of Standard Additions	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration.

Minimum Detectable Activity (MDA)	TNI- Estimate of the smallest true activity that ensures a specified high confidence, 1 – β, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods, β is often set at 0.05. NOTE 1: The MDS is a measure of the detection capability of a measurement process and as such, it is an a priori concept. It may be used in the selection of methods to meet specified MQOs. Laboratories may also calculate a "sample specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability. However, the MDA must never be used instead of the Critical Value as a detection threshold. NOTE 2: For the purpose of this Standard, the terms MDA and minimum detectable concentration (MDC) are equivalent.
MintMiner	Program used by Pace to review large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
Must	EPA – The word "must" is used to indicate aspects of the method that are considered essential to its performance, based on sound analytical practices.
National Environmental Laboratory Accreditation Conference (NELAC)	See definition of The NELAC Institute (TNI).
National Institute of Occupational Safety and Health (NIOSH)	National institute charged with the provision of training, consultation and information in the area of occupational safety and health.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.

Operator Aid	DoD- A technical posting (such as poster, operating manual, or notepad) that assists workers in performing routine tasks. All operator aids must be controlled documents (i.e., a part of the laboratory management system).
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Physical Parameter	TNI- A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical and biological components.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Positive Control	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI- The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	TNI and DoD- Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Primary Accreditation Body (Primary AB)	TNI- The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing (PT)	TNI- A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.
Proficiency Testing Program (PT Program)	TNI- The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Proficiency Testing Provider (PT Provider)	TNI- A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT Program.
Proficiency Testing Provider Accreditor (PTPA)	TNI- An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.

Proficiency Testing Reporting Limit (PTRL)	TNI- A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.
Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Proficiency Testing (PT) Study	TNI- a) Scheduled PT Study: A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants; b) Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard [TNI] but that does not have a pre-determined opening date and closing date.
Proficiency Testing Study Closing Date	TNI- a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider; b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.
Proficiency Testing Study Opening Date	TNI- a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT Provider; b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.
Protocol	TNI- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Qualitative Analysis	DoD- Analysis designed to identify the components of a substance or mixture.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.
Quality Control (QC)	TNI- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.

Quality Manual	TNI- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality System	TNI and DoD- A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.
Quality System Matrix	TNI and DoD- These matrix definitions shall be used for purposes of batch and quality control requirements and may be different from a field of accreditation matrix: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin. • **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	DoD- The range of values (concentrations) in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard used to relate instrument response to analyte concentration. The quantitation range (adjusted for initial sample volume/weight, concentration/dilution and final volume) lies within the calibration range.
Quantitative Analysis	DoD- Analysis designed to determine the amounts or proportions of the components of a substance.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.

Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.
Reagent Blank (method reagent blank)	A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Records	DoD- The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Method	TNI- A published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a "standard method", that term is equivalent to "reference method"). When a laboratory is required to analyze by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is no regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.
Reference Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e., statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.
Reporting Limit Verification Standard (RLVS)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	Denotes a mandatory specification; often designated by the term "shall".

Retention Time	The time between sample injection and the appearance of a solute peak at the detector.
Revocation	TNI- The total or partial withdrawal of a laboratory's accreditation by an accreditation body.
Sample	Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Condition Upon Receipt Form (SCURF)	Form used by sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a chain-of-custody form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selected Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector. DoD- Using GC/MS, characteristic ions specific to target compounds are detected and used to quantify in applications where the normal full scan mass spectrometry results in excessive noise.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	TNI- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	EPA – The word "shall" is used to indicate aspects of the method that are considered essential to its performance, based on sound analytical practices.
Should	EPA – The word "should" is used to provide guidance on aspects of the method that are useful but not essential.
Signal-to-Noise Ratio (S/N)	DoD- A measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of the noise on the relative error of a measurement increases.

Source Water	TNI- When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.
Spike	A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI- The document describing the elements of a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.
Standard (Chemical)	Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.
Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Storage Blank	DoD- A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A storage blank is used to record contamination attributable to sample storage at the laboratory.
Supervisor	The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
Surrogate	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Suspension	TNI- The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed 6 months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.

Target Analytes	DoD- Analytes or chemicals of primary concern identified by the customer on a project-specific basis.
Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
Test Method	DoD- A definitive procedure that determines one or more characteristics of a given substance or product.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Test Source	TNI- A radioactive source that is tested, such as a sample, calibration standard, or performance check source. A Test Source may also be free of radioactivity, such as a Test Source counted to determine the subtraction background, or a short-term background check.
The NELAC Institute (TNI)	A non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. Previously known as NELAC (National Environmental Laboratory Accreditation Conference).
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty, Counting	TNI- The component of Measurement Uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts (MARLAP). Older references sometimes refer to this parameter as Error, Counting Error or Count Error (c.f., Total Uncertainty).
Uncertainty, Expanded	TNI- The product of the Standard Uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand (c.f., Standard Uncertainty). NOTE: Radiochemical results are generally reported in association with the Total Uncertainty. Either if these estimates of uncertainty can be reported as the Standard Uncertainty (one-sigma) or as an Expanded Uncertainty (k-sigma, where k $>$ 1).
Uncertainty, Measurement	TNI- Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.
Uncertainty, Standard	TNI- An estimate of the Measurement Uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).
Uncertainty, Total	TNI- An estimate of the Measurement Uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar items (c.f., Counting Uncertainty).
Unethical actions	DoD- Deliberate falsification of analytical or quality control results where failed method or contractual requirements are made to appear acceptable.
United States Department of Agriculture (USDA)	A department of the federal government that provides leadership on food, agriculture, natural resources, rural development, nutrition and related issues based on public policy, the best available science, and effective management.
United States Geological Survey (USGS)	Program of the federal government that develops new methods and tools to supply timely, relevant, and useful information about the Earth and its processes.
Unregulated Contaminant Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminants in drinking water.
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
Verification	TNI- Confirmation by examination and objective evidence that specified requirements have been met. In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

Voluntary Action Program (VAP)	A program of the Ohio EPA that gives individuals a way to investigate possible environmental contamination, clean it up if necessary and receive a promise from the State of Ohio that no more cleanup is needed.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

10.0. REFERENCES

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11.0. REVISIONS

The Pace Corporate Environmental Quality Office files an electronic version of a Microsoft Word document with tracked changes detailing all revisions made to previous versions of the Quality Assurance Manual. This document is available upon request. All current revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual 19.0	General: made administrative edits that do not affect the policies or procedures within the document (including revising company name to Pace Analytical Services, LLC). Cover page: removed corporate approval signature lines and revised document control format. Table of Contents: added Attachment VII – Pace COC Old Section 3: moved to other sections of the QAM as applicable and deleted entire section (All section references below reflect the new section numbers). Section 1.1.2: replaced with section 3.1.1. Sections 1.3, 1.4, 1.11: removed extraneous language. Sections 1.5: added language from old section 1.6. Section 1.6: revised anonymous reporting information. Section 1.8: removed job descriptions for non-applicable personnel. Section 1.8.4: added tasks to QM job description. Section 1.8.8: added tasks to PM job description. Section 1.11.1: added keyless entry using key fobs detail. Section 2: rearranged existing sections. Section 2.4: reworded to match existing Sample Acceptance policy document. Section 2.6.3.2: added some detail regarding temperature monitoring corrective action. Section 2.6.5.1: added by-products of USDA soils. Section 3.2.2: added basic evaluation criteria. Section 3.4.3: added MS and Dup as optional alternative to MS/MSD. Section 3.5.2: added basic evaluation criteria. Section 3.9.1: added that RL may be based on calibration standard. Section 3.14: added new instrumentation as requiring validation. Section 4: in general, for each QC type, removed language regarding frequency and corrective actions and referenced lab-specific SOPs. Section 5: in general, removed extraneous language and Management of Change section. Section 5.1, 5.2: reorganized into Primary and Secondary Review sections and removed extraneous language. Section 5.3.2: specified types of support equipment to be monitored daily. Section 5.3.3.1: specified “working” weights. Section 5.3.4.2: added temperature sensors and added alternatives to annual in-house verification. Section 5.3.5: added pH electrode inspection/maintenance. Section 6: removed extraneous language including Quarterly Report section. Section 8.2.3.1: added “or designee”. Section 9 (glossary): revised and added definitions based on 2016 TNI Standard. Added “may, must, shall and should” based on SW-846 definition. Section 10: Added EPA DW Manual and revised references as applicable. Attachment III: updated corporate organizational chart. Old Attachment IV: removed floor plan attachment. Old Attachment VII: removed COC (available in SOPs). Indy added back in.	22Mar2017

ATTACHMENT I- QUALITY CONTROL CALCULATIONS

PERCENT RECOVERY (%REC)

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

NOTE: The SampleConc is zero (0) for the LCS and Surrogate Calculations

PERCENT DIFFERENCE (%D)

$$\%D = \frac{MeasuredValue - TrueValue}{TrueValue} * 100$$

where:
TrueValue = Amount spiked (can also be the $\overline{CF}$ or $\overline{RF}$ of the ICAL Standards)
Measured Value = Amount measured (can also be the CF or RF of the CCV)

PERCENT DRIFT

$$\%Drift = \frac{CalculatedConcentration - TheoreticalConcentration}{TheoreticalConcentrtion} * 100$$

RELATIVE PERCENT DIFFERENCE (RPD)

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

where:
R1 = Result Sample 1
R2 = Result Sample 2

CORRELATION COEFFICIENT (R)

$$CorrCoeff = \frac{\sum_{i=1}^{N} W_i * (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} W_i * (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} W_i * (Y_i - \overline{Y})^2\right)}}$$

With:		
	N	Number of standard samples involved in the calibration
	i	Index for standard samples
	Wi	Weight factor of the standard sample no. i
	Xi	X-value of the standard sample no. i
	X(bar)	Average value of all x-values
	Yi	Y-value of the standard sample no. i
	Y(bar)	Average value of all y-values

ATTACHMENT I- QUALITY CONTROL CALCULATIONS (CONTINUED)

STANDARD DEVIATION (S)

$$S = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \overline{X})^2}{(n-1)}}$$

where:
n = number of data points
X_i = individual data point
$\overline{X}$ = average of all data points

AVERAGE ($\overline{X}$)

$$\overline{X} = \frac{\sum_{n=1}^{i} X_i}{n}$$

where:
n = number of data points
X_i = individual data point

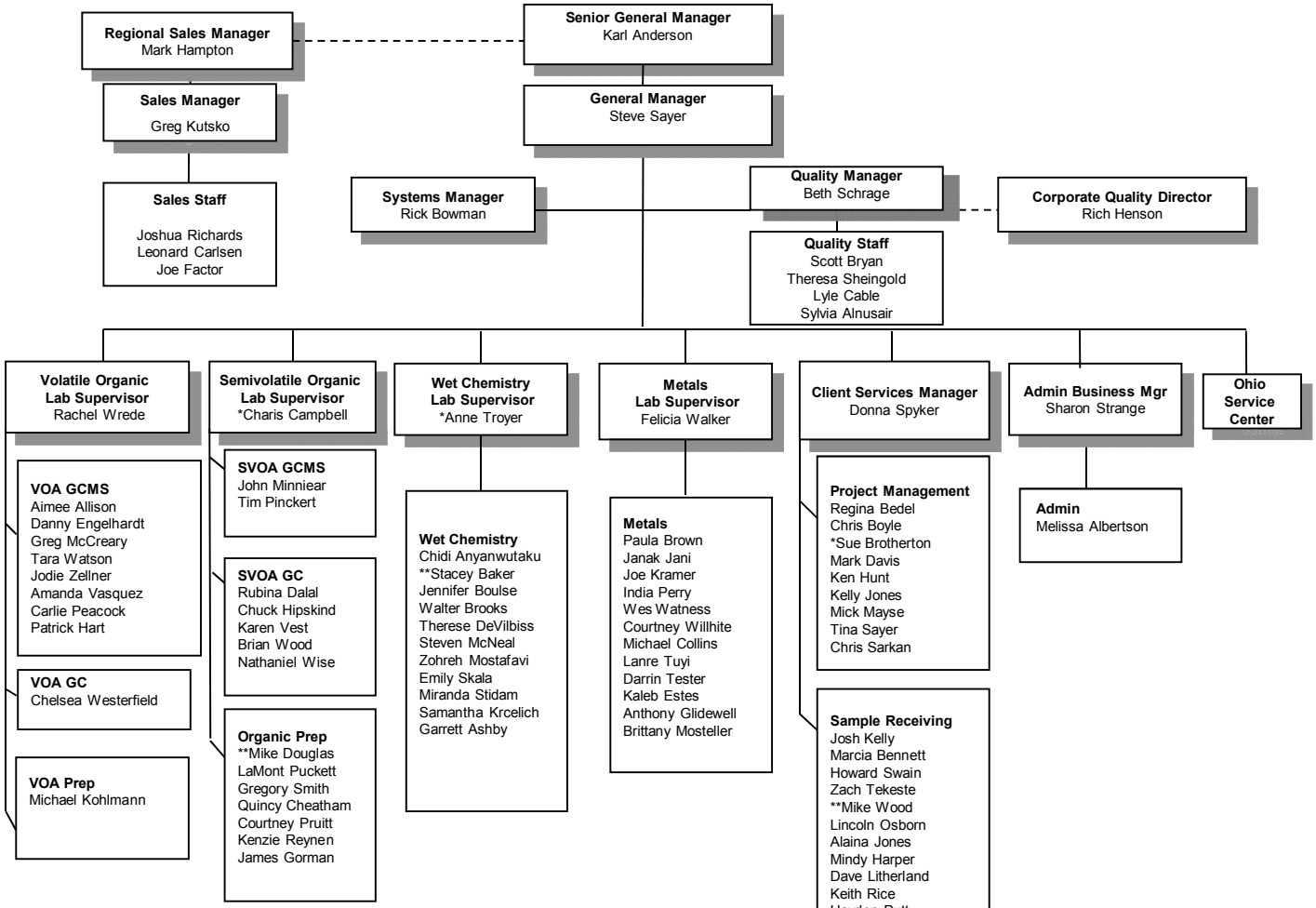
RELATIVE STANDARD DEVIATION (RSD)

$$RSD = \frac{S}{\overline{X}} * 100$$

where:
S = Standard Deviation of the data points
$\overline{X}$ = average of all data points

ATTACHMENT II- LABORATORY ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)

PACE ANALYTICAL SERVICES - INDIANAPOLIS

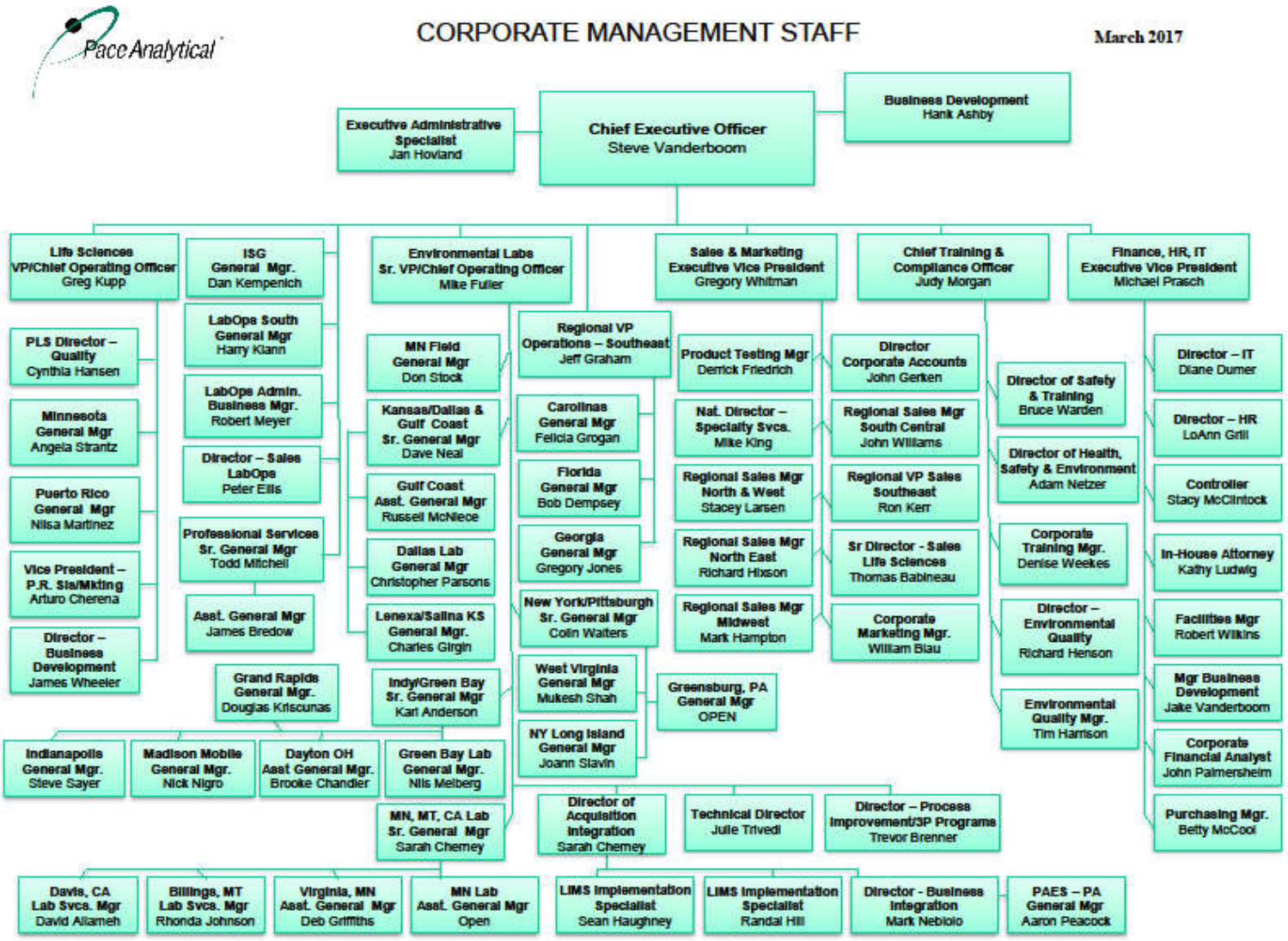


*TNI TECHNICAL DIRECTOR

**DEPT LEAD

Last Revised 3/22/2017

ATTACHMENT III- CORPORATE ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)

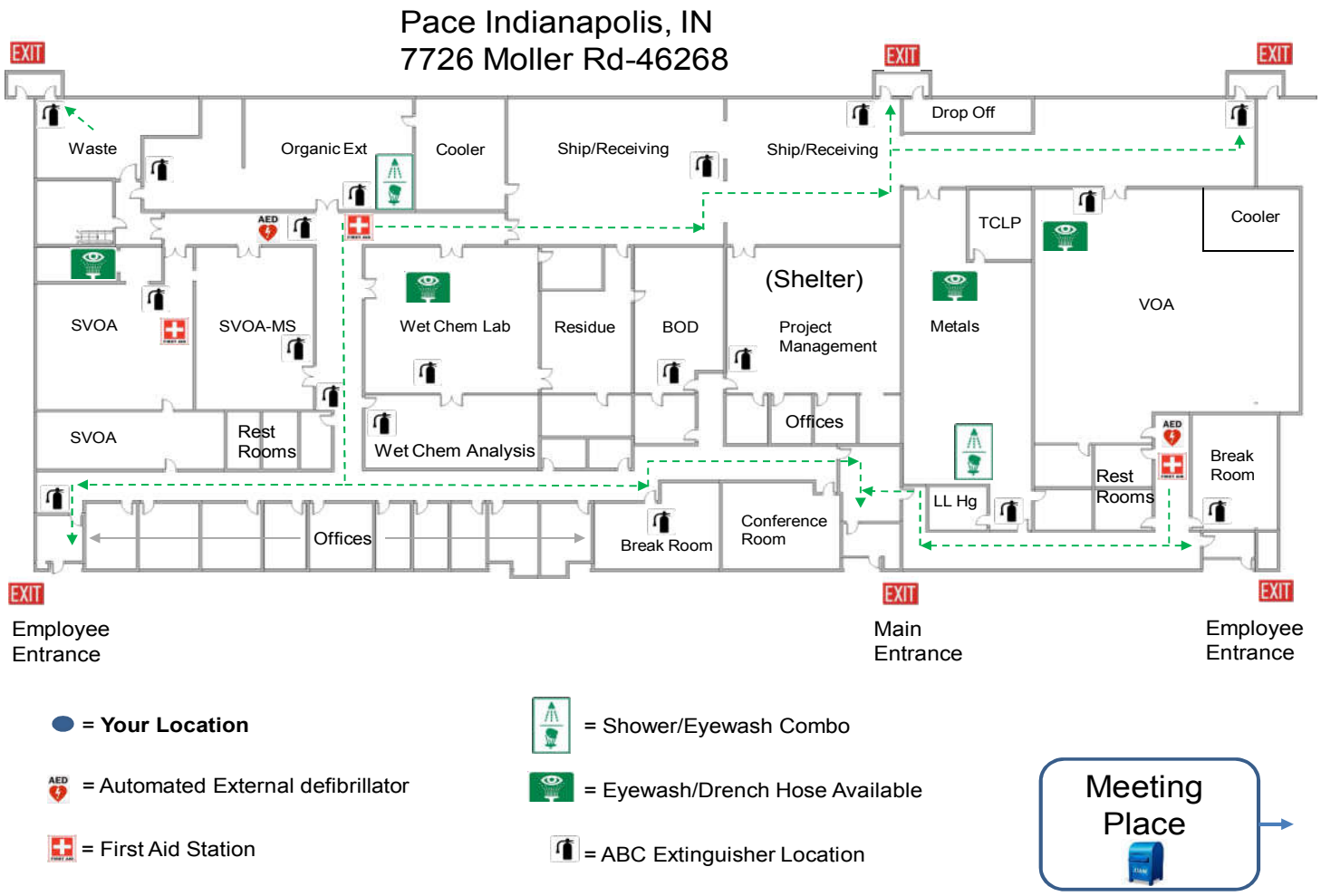


ATTACHMENT IV- EQUIPMENT LIST (CURRENT AS OF ISSUE DATE)

Pace Indianapolis Equipment/Instrumentation List

INSTRUMENT	MANUFACTURER	MODEL NUMBER	DETECTOR	AUTOSAMPLER	SERVICE ANALYSIS	YEAR
GC/MS	Agilent	6890	MS 5973	Centurion W/S	8260/624 VOC	2003
GC/MS	Agilent	6890	MS 5973	Centurion	8260/624/524.2 VOC	2007
GC/MS	Agilent	6890	MS 5973	Centurion W/S	8260/624 VOC	2003
GC/MS	Agilent	6850N	MS 5975	Centurion	8260/624/524.2 VOC	2007
GC/MS	Agilent	6890	MS 5973	Centurion W/S	8260/624 VOC	2004
GC/MS	Agilent	6850N	MS 5975	Centurion	8260/624 VOC	2010
GC/MS	Agilent	6890	MS 5973	OI	8260/624/524.2 VOC	2007
GC/MS	Agilent	7890	MS 5975C	Archon	8260	2008
GC/MS	Agilent	6890	MS 5975	OI	8260/624/524.2 VOC	2007
GC/MS	Agilent	6890	5975	Centurion	8260/624 VOC	2008
GC/MS	Hewlett-Packard	6890	MS 5973	7683	8270 PAH SIM	2000
GC/MS (2)	Agilent	7890	MS 5975	7683	8270/625 BNA	2008
GC/MS (2)	Agilent	6890	MS 5975	7683	8270 PAH SIM	2009
GC/MS (3)	Agilent	6890	MS 5973	7683	8270/625 BNA	2008
GC/MS	Agilent	7890	MS 5975	7683	8270 PAH SIM	2009
GC/MS (2)	Hewlett-Packard	5890	MS 5971	7673	Solvent Screen	2007
Gas Chromatograph	Agilent	6890	FID	7683	8015 Alcohols	2006
Gas Chromatograph	Hewlett-Packard	6890	FID	6890	8015 Glycols	2008
Gas Chromatograph	Agilent	7890A	FID	7693	8015 DRO/ERO	2009
Gas Chromatograph	Agilent	7890A	Dual ECD	7693	8082/608 PCBs/8011 EDB/DBCP	2009/2013
Gas Chromatograph	Hewlett-Packard	5890	FID	6890	Benzene	2006
Gas Chromatograph	Hewlett-Packard	5890	FID	8100	8015 GRO	2011
Gas Chromatograph	Hewlett-Packard	5890	FID	EST LGX50	RSK175 Dissolved gases	2006
Gas Chromatograph	Agilent	6890N	FID	8100	8015 GRO	2008
Gas Chromatograph	Agilent	6890	Dual NPD	7683	Pesticides	2008
Gas Chromatograph (2)	Agilent	6890	Dual ECD	7683	PCBs	2008
Gas Chromatograph	Hewlett-Packard	6890	Dual ECD	7683	Herbicides	2008
Gas Chromatograph	Agilent	7890	Dual ECD	7693	Pesticides	2010
Microwave Extractors (2)	CEM	230/60	n/a	n/a	soil extraction	2008/2011
Spe-Dex	Horizon	4790	n/a	n/a	1664A Oil & Grease	2008
Trace ICP (3)	Thermo Scientific	ICAP 6500	n/a	n/a	6010/200.7 Metals	2008/2011
ICP/MS (2)	Agilent	7700	n/a	n/a	6010/200.7 Metals	2012/2014
Mercury Analyzer	CETAC	M-6100	n/a	n/a	7470/7471/245 Mercury	2012/2010
Mercury Analyzer	Teledyne Leeman	M-7600	n/a	n/a	7470/7471/245 Mercury	2016
Low-Level Mercury Analyzer	CETAC	M-8000	n/a	n/a	Low-Level Mercury	2015
Auto Analyzer (2)	Lachat	Quick Chem	n/a	n/a	NO3,Cl,Phenol, NH3,TKN	2010/2012
Titrosampler	Metrohm	855	n/a	n/a	Alkalinity, Acidity	2014
Automated Flash Point	Tanaka	APM-8	n/a	n/a	flash point	2010
Spectrophotometer	Spec 20	Labtronics	n/a	n/a	Sulfide	2002
Spectrophotometer	Hach	DR5000	n/a	n/a	Sulfate,Cr6+,Fe2+, PO4	2007
Spectrophotometer	Thermo	AquaMatePlus	n/a	n/a	Surfactants, COD	2005
pH/ISE Meter (2)	Accumet	AR25/XL25	n/a	n/a	pH, Fluoride, Redox	2003/2010
pH/ISE Meter	Thermo Orion Star	A214	n/a	n/a	pH, Fluoride, Redox	2013
Dissolved Oxygen/pH Meter	Hach	HQ440d	n/a	n/a	BOD, cBOD	2014
BOD Analyzer	Thermo	AutoEz	n/a	n/a	BOD, cBOD	2013
TOC Analyzer	Shimadzu	TOC-Vwp	n/a	n/a	TOC, DOC	2008
TOC Analyzer	Teledyne	Phoenix 8000	n/a	n/a	TOC, DOC	2005
Discrete Analyzer	Smart Chem	200	n/a	n/a	Cyanide, Phosphorus	2006
Ion Chromatogram	Dionex	IC3000	n/a	n/a	Cl-, F-, SO4-, Br-, NO3/NO2	2008

ATTACHMENT V- LABORATORY FLOOR PLAN (CURRENT AS OF ISSUE DATE)



ATTACHMENT VI- LABORATORY CERTIFICATION LIST (CURRENT AS OF ISSUE DATE)

Pace Analytical – Indianapolis Certifications

Accrediting Authority	Program Category	Accrediting Agency	Certification #	Expiration Date
Illinois	Hazardous Waste	IL-EPA	003971	10/12/2017
Illinois	Non-Potable Water	IL-EPA	003971	10/12/2017
Indiana	Drinking Water	ISDH	C-49-06	05/06/2018
Kansas (TNI)	Hazardous Waste	KDHE	E-10177	04/30/2017
Kansas (TNI)	Non-Potable Water	KDHE	E-10177	04/30/2017
Kentucky	UST	KDEP	80226	04/30/2017
Kentucky	Wastewater	KDEP	98019	12/31/2017
Ohio VAP	Hazardous Waste	OH-EPA	CL0065	01/28/2018
Ohio VAP	Non-Potable Water	OH-EPA	CL0065	01/28/2018
Oklahoma	Non-Potable Water	OK DEQ	2016-075	08/31/2017
Oklahoma	Solids	OK DEQ	2016-075	08/31/2017
Texas	Non-Potable Water	TX CEQ	T104704355-16-10	01/31/2018
Texas	Solid Chemical Mat.	TX CEQ	T104704355-16-10	01/31/2018
West Virginia	Hazardous Waste	WV-DEP	330	10/31/2017
West Virginia	Non-Potable Water	WV-DEP	330	10/31/2017
Wisconsin	Non-Potable Water	WI DNR	999788130	08/31/2017
Wisconsin	Waste, Soil, Tissue	WI DNR	999788130	08/31/2017
USDA	Foreign Soil	USDA	P330-16-00257	08/19/2019

ATTACHMENT VII- PACE CHAIN-OF-CUSTODY (CURRENT AS OF ISSUE DATE)

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Pace Analytical™ www.pacelabs.com

Page: of

Section A Required Client Information:
Company:
Address:
Email To: Fax:
Phone:
Requested Due Date/TAT:

Section B Required Project Information:
Report To:
Copy To:
Purchase Order No.:
Project Name:
Project Number:

Section C Invoice Information:
Attention:
Company Name:
Address:
Pace Quote Reference:
Pace Project Manager:
Pace Profile #:

REGULATORY AGENCY
☐ NPDES ☐ GROUND WATER ☐ DRINKING WATER
☐ UST ☐ RCRA ☐ OTHER

Site Location STATE:

Section D Required Client Information

SAMPLE ID (A-Z, 0-9 / ,-) Sample IDs MUST BE UNIQUE

Matrix Codes (MATRIX / CODE): Drinking Water DW; Water WT; Waste Water WW; Product P; Soil/Solid SL; Oil OL; Wipe WP; Air AR; Tissue TS; Other OT

ITEM #	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED: COMPOSITE START DATE	COMPOSITE START TIME	COMPOSITE END/GRAB DATE	COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Analysis Test ↓	Y/N	Requested Analysis Filtered (Y/N)	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
1																					
2																					
3																					
4																					
5																					
6																					
7																					
8																					
9																					
10																					
11																					
12																					

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS	Temp in °C	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER:

SIGNATURE of SAMPLER:

DATE Signed (MM/DD/YY):

ATTACHMENT VIII- METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE (CURRENT AS OF ISSUE DATE)

THE HOLDING TIME INDICATED IN THE CHART BELOW IS THE MAXIMUM ALLOWABLE TIME FROM COLLECTION TO EXTRACTION AND/OR ANALYSIS PER THE ANALYTICAL METHOD. FOR METHODS THAT REQUIRE PROCESSING PRIOR TO ANALYSIS, THE HOLDING TIME IS DESIGNATED AS 'PREPARATION HOLDING TIME/ANALYSIS HOLDING TIME'.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Acid Base Accounting	Sobek	Solid	Plastic/Glass	None	N/A
Acidity	SM2310B	Water	Plastic/Glass	≤ 6°C	14 Days
Acid Volatile Sulfide	Draft EPA 1629	Solid	8oz Glass	≤ 6°C	14 Days
Actinides	HASL-300	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Actinides	HASL-300	Solid	Plastic/Glass	None	180 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass (NY requires separate bottle filled to the exclusion of air)	≤ 6°C	14 Days
Alkylated PAHs		Water	1L Amber Glass	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved
Alkylated PAHs		Solid	8oz Glass	≤ 10°C	1 Year/40 Days
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0/300.1/SM4110B	Water	Plastic/Glass	≤ 6°C; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0	Solid	Plastic/Glass	≤ 6°C	All analytes 28 days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	$\leq 6^oC$	48 hours
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Asbestos	EPA 600/R-93/116	Solid	Plastic/Glass; bulk- 2" square; popcorn ceiling- 2tbsp; soil- 4oz	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	$\leq 6^oC$; $Na_2S_2O_3$	24 Hours
Base/Neutrals and Acids	8270	Solid	8oz Glass	$\leq 6^oC$	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.2	Water	1L Amber Glass	pH<2 HCl; $\leq 6^oC$; Na sulfite if Cl present	14/30 Days
Biomarkers		Water	$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	$\leq 6^oC$; pH<2 1:1 HCl (optional)
Biomarkers		Solid	$\leq 10^oC$	1 Year/40 Days	$\leq 10^oC$
BOD/cBOD	SM5210B	Water	Plastic/Glass	$\leq 6^oC$	48 hours
Boiling Range Distribution of Petroleum Fractions	ASTM D2887-98	Product	10mL glass vials	$\leq 6^oC$	N/A
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	28 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	72 Hours
Carbamates	531.1	Water	Glass	$Na_2S_2O_3$, Monochloroacetic acid pH <3; $\leq 6^oC$	28 Days
Carbamates	8318	Water	Glass	Monochloroacetic acid pH 4-5; $\leq 6^oC$	7/40 Days
Carbamates	8318	Solid	Glass	$\leq 6^oC$	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Carbon Specific Isoptope Analysis (CSIA)	AM24	Water	40mL clear VOA vial with TLS	≤ 6°C, trisodium phosphate or HCl	N/A
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Cation Exchange	9081	Solid	8oz Glass	None	unknown
Cations (Ferrous Iron, Ferric Iron, Divalent Manganese)	7199 modified	Water	40mL clear VOA vials with mylar septum	≤ 6°C; HCl	48 Hours
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days
Chlorinated Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes
Chlorophyll	SM10200H	Water	Opaque bottle or aluminum foil	≤ 6°C	48 Hours to filtration
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	24 Hours
Coliform, Fecal	SM9221E	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9221E	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	24 Hours
Coliform, Total	SM9222B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total	SM9221B	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total, Fecal and E. coli	Colilert/ Quanti-tray	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total and E. coli	SM9223B	Drinking Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	30 Hours
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6°C	48 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	180 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/ 9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6°C; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present- applies to SM4500CN only)
Diesel Range Organics- Alaska DRO	AK102	Solid	8oz Glass	≤ 6°C	14/40 Days
Diesel Range Organics- Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Diesel Range Organics- TPH DRO	8015	Tissue	1L Amber Glass	≤ - 10°C	1 Year if frozen/40 Days
Diesel Range Organics- TPH DRO	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Water	1L Amber Glass	pH <2 HCl; ≤ 6°C	14/40 Days; 7 Days from collection to extraction if unpreserved
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	≤ 6°C	10/47 Days
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass	≤ 6°C; pH <2 HCl	14/40 Days
Dioxins and Furans	1613B	Solid	8oz Glass	≤ 6°C	1 year
Dioxins and Furans	1613B	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	≤ 6°C	1 year
Dioxins and Furans	8290	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	30/45 Days
Dioxins and Furans	8290	Solid	8oz Glass	≤ 6°C	30/45 Days
Dioxins and Furans	8290	Fish/ Tissue	Not specified	< -10°C	30/45 Days
Dioxins and Furans	TO-9	Air	PUF	None	7/40 Days
Diquat/Paraquat	549.2	Water	Amber Plastic	≤ 6°C; $Na_2S_2O_3$	7/21 Days
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days
Endothall	548.1	Water	Amber Glass	≤ 6°C; $Na_2S_2O_3$	7/14 Days
Enterococci	EPA 1600	Water	100mL Plastic	≤ 10°C	8 Hours
Enterococci	Enterolert	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Explosives	8330/8332	Water	1L Amber Glass	≤ 6°C	7/40 Days
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Water	1L Amber Glass	pH < 2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Solid	4oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6°C	7/40 Days
Fecal Streptococci	SM9230B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Ferrous Iron	SN3500Fe-D; Hach 8146	Water	Glass	None	Immediate

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Flashpoint/ Ignitability	1010	Liquid	Plastic/Glass	None	28 Days
Florida PRO	FL PRO DEP (11/1/95)	Liquid	Glass, PTFE lined cap	$\leq 6^oC$; pH <2 H_2SO_4 or HCl	7/40 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days
Gasoline Range Organics (C3-C10)	8260B modified	Water	40mL vials	$\leq 6^oC$; HCl	14 Days
Gasoline Range Organics (C3-C10)	8260B modified	Solid	4oz Glass Jar	$\leq 6^oC$	14 Days
Gasoline Range Organics- Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)
Gasoline Range Organics- Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; $\leq 6^oC$	14 Days
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; $\leq 6^oC$	7 Days unpreserved; 14 Days preserved
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	$\leq 6^oC$; packed jars with no headspace	14 Days
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Water	40mL vials	pH<2 HCl; $\leq 6^oC$	14 Days
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	$\leq 6^oC$ in MeOH	21 Days
Glyphosate	547	Water	Glass	$\leq 6^oC$; $Na_2S_2O_3$	14 Days (18 Months frozen)
Grain Size	ASTM D422	Solid	Not specified	Ambient	N/A
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; $\leq 6^oC$	14/7 Days if extracts stored $\leq 6^oC$ or 14/14 Days if extracts stored at $\leq -10^oC$

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Heterotrophic Plate Count (SPC/HPC)	SM9215B	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Heterotrophic Plate Count (SPC/HPC)	SimPlate	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Herbicides, Chlorinated	8151	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14/28 Days
Hexavalent Chromium	7196/218.6/ SM3500Cr-B, C	Water	Plastic/Glass	$\leq 6^oC$	24 Hours (see note 4)
Hexavalent Chromium	218.6/SM3500Cr-B, C	Water	Plastic/Glass	Ammonium Buffer pH 9.3-9.7	28 Days (see note 4)
Hexavalent Chromium	218.6/218.7	Drinking Water	Plastic/Glass	Ammonium Buffer pH >8	14 Days (see note 4)
Hexavalent Chromium	7196 (with 3060A)	Solid	Glass	$\leq 6^oC$	30 Days from collection to extraction and 7 days from extraction to analysis
Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Hydrogen by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Light Hydrocarbons by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Light Hydrocarbons in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Lipids	Pace Lipids	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen
Mercury, Low-Level	1631E	Solid	Glass	None	28 Days
Mercury, Low-Level	1631E	Water	Fluoropolymer bottles (Glass if Hg is only analyte being tested)	12N HCl or BrCl	48 Hours for preservation or analysis; 28 Days to preservation if sample oxidized in bottle; 90 Days for analysis if preserved
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Mercury	7471	Solid	8oz Glass Jar	≤ 6°C	28 Days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Mercury	7471/245.6	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	180 Days
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	180 Days
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	≤ -10°C	180 Days if frozen
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175; PM01/AM20GAx	Water	20mL vials	HCl; or trisodium phosphate or benzalkonium chloride and ≤ 6°C	14 Days; 7 Days unpreserved
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	28 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
Methanol, Ethanol	8015 modified	Water	40mL vials	≤ 6°C	14 Days
Methanol, Ethanol	8015 modified	Solid	2oz Glass	≤ 6°C	14 Days
Methyl Mercury	1630	Water	Teflon/ fluoropolymer	Fresh water- 4mL/L HCl; Saline water- 2mL/L H_2SO_4 (must be preserved within 48 hours of collection)	6 months

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Methyl Mercury	1630	Tissue	2-4oz glass jar	≤ 0°C	28 Days; ethylated distillate 48 hours
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Total Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Total Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	≤ 6°C	24 Hours preferred
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	≤ 6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	28 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	72 Hours
Odor	SM2150B	Water	Glass	≤ 6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Oil and Grease/HEM	9071	Solid	Glass	≤ 6°C	28 Days
Oil Range Organics	8015	Solid	Glass	≤ 6°C	14/40 Days
Oil Range Organics	8015	Water	Glass	≤ 6°C	7/40 Days
Organic Matter	ASA 29-3.5.2	Solid	Plastic/Glass	None; samples air-dried and processed prior to analysis	N/A
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Oxygenates on Product (GCMS SIM)	1625 modified	Product	10mL glass vial	≤ 6°C	14 Days (7 Days from extraction)
PBDEs	1614	Water	1L Amber Glass	≤ 6°C	1 Year/1 Year
PBDEs	1614	Solid	Wide Mouth Jar	≤ 6°C	1 Year/1 Year
PBDEs	1614	Tissue	Aluminum Foil	≤ -10°C	1 Year/1 Year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days
PCBs and Pesticides, Organochlorine (OC)	608	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	Pest: 7/40 Days; PCB: 1 Year/1 Year
PCBs, Pesticides (OC), Herbicides	508.1	Water	Glass	Na2SO3; pH<2 HCl; $\leq 6^oC$	14/30 Days
PCBs, total as Decachlorobiphenyl	508A	Water	1L Glass, TFE lined cap	$\leq 6^oC$	14/30 Days
Perchlorate	331	Water	Plastic/Glass	$\geq$0-6^oC, field filtered with headspace	28 Days
Permanent Gases (O2, N2, CO2)	RSK-175; PM01/AM20GAx	Water	40mL vials	benzalkonium chloride and $\leq 6^oC$	14 Days
Permanent Gases by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Permanent Gases in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	$\leq -10^oC$	1 Year if frozen/40 Days
Pesticides, Organophosphorous (OP)	8141	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Pesticides, Organophosphorous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
PCBs (Aroclors)	8082	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	1 Year/1 Year
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	$\leq 6^oC$	1 Year/1 Year
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	$\leq -10^oC$	1 Year if frozen/1 Year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
PCB Congeners	1668A	Water	1L Amber Glass	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Solid	4-8oz Glass Jar	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	≤ -10°C	1 Year/1 Year
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particle Size	ASA 15-5 modified	Solid	Plastic/Glass (100g sample)	None	N/A
Particulates	PM-10	Air	Filters	None	180 Days
Permanent Gases	EPA 3C	Air	Summa Canister	None	28 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
pH	SM4500H+B/9040	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	7 Days
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	≤ 6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Total	365.4	Solid	Plastic/Glass	≤ 6°C	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/40 Days
Purgeable Organic Halides (POX)	9021	Water	Glass; no headspace	≤ 6°C	14 Days
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320	Solid	Plastic/Glass		
Residual Range Organics- Alaska RRO	AK103	Solid	8oz Glass	≤ 6°C	14/40 Days
Saturated Hydrocarbons		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Saturated Hydrocarbons		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤ 6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total	SM2540G	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6°C	28 Days
Stationary Source Dioxins and Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	180 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	180 Days
Sulfate	SM4500SO4/9036/9038/375.2/ASTM D516	Water	Plastic/Glass	$\leq 6^oC$	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; $\leq 6^oC$	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	$\leq 6^oC$	48 Hours
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Total Alpha Radium (see note 3)	9315	Solid	Plastic/Glass	None	180 days
Total Inorganic Carbon (TIC)	PM01/AM20GAx	Water	40mL VOA vial with mylar septum	$\leq 6^oC$	14 Days
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4 or HCl; $\leq 6^oC$	28 Days
Total Organic Carbon (TOC)	9060/Walkley Black/Lloyd Kahn	Solid	Glass	$\leq 6^oC$	14 Days
Total Organic Halogen (TOX)	SM5320/9020	Water	Glass; no headspace	$\leq 6^oC$	14 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Water	40mL vials	pH<2 HCl, no headspace, $\leq 6^oC$	7 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Solid	Glass	$\leq 6^oC$	14 days
Tritium	906.0	Water	Glass	None	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	$\leq 6^oC$	48 Hours
Total Uranium	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HNO_3	180 days
UCMR Metals	200.8	Water	Plastic or glass	pH<2 HNO_3	28 Days
UCMR Hexavalent Chromium	218.7	Water	HDPE or propylene	Na_2CO_3/$NaHCO_3$/ $(NH_4)_2SO_4$; pH>8	14 Days
UCMR Chlorate	300.1	Water	Plastic or glass	EDA	28 Days
UCMR Perfluorinated Compounds	537	Water	Polypropylene	Trizma	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
UCMR 1, 4 Dioxane	522	Water	Glass	Na_2SO_3, $NaHSO_4$; pH<4	28 Days
UV254	SM5910B	Water	Glass	≤ 6°C	48 Hours
Vermiculite	EPA 600/R-93/116	Solid	Plastic/Glass	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Volatile Fatty Acids	AM21G	Water	40mL clear VOA vials	≤ 6°C	21 Days
Volatile Fatty Acids (low level)	AM23G	Water	40mL clear VOA vials	≤ 6°C with benzalkonium chloride	14 Days
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days preserved
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Solid	4-8oz Glass Jar	≤ 6°C; packed jars with no headspace	7/28 Days
Volatiles	TO-14	Air	Summa Canister	None	28 Days
Volatiles	TO-14	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	TO-15	Air	Summa Canister or Tedlar Bag	None	28 Days
Volatiles	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Volatiles	TO-18/8260	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	8260	Solid	5035 vial kit	See note 1 (analyze for acrolein and acrylonitrile per local requirements)	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤ 6°C	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present (or unpreserved if run within 7 days of collection) (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days (7 Days for aromatics if unpreserved)
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; ≤ 6°C; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days
Whole Oil	ASTM D3328 (prep); ASTM D5739	Product	10mL glass vials	≤ 6°C	N/A

[1] **5035/5035A Note**: 5035 vial kit typically contains 2 vials water, preserved by freezing **or**, 2 vials aqueous sodium bisulfate preserved at 4°C, **and** one vial methanol preserved at ≤6°C **and** one container of unpreserved sample stored at ≤6°C.

[2] Method 524.2 lists ascorbic acid as the preservative when residual chlorine is suspected, unless gases or Table 7 compounds are NOT compounds of interest and then sodium thiosulfate is the preservative recommended.

[3] Methods 9315 and 9320 both state that if samples are unpreserved, the samples should be brought to the lab within 5 days of collection, preserved in the lab, and then allowed to sit for a minimum of 16 hours before sample preparation/analysis.

[4] The holding time for hexavalent chromium may be extended by the addition of the ammonium buffer listed in EPA 218.6 per the 2012 EPA Method Update Rule. Although Method 218.6 stipulates a different pH range (9.0 to 9.5) for buffering, this method requirement was modified in the Method Update Rule to a pH range of 9.3 to 9.7.For non-potable waters, adjust the pH of the sample to 9.3 to 9.7 during collection with the method required ammonium sulfate buffer to extend the holding time to 28 days. For potable waters, addition of the buffer during collection will extend the holding time for 14 days per EPA 218.7 and the EPA UCMR program.

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF SOIL AND WASTE pH

REFERENCE METHOD: EPA SW-846 METHOD 9045C

SOP NUMBER:	S-IN-I-069-rev.12
EFFECTIVE DATE:	February 13, 2017
SUPERSEDES:	S-IN-I-069-rev.11

APPROVAL

General Manager — February 7, 2017 Date

Quality Manager — February 7, 2017 Date

Department Manager — February 7, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining pH in solid samples while meeting the requirements specified in EPA method 9045C.

2. Summary of Method

2.1. The sample is mixed with reagent water and then the pH of the mixture is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode. The measuring device is calibrated using a series of standard solutions of known pH.

3. Scope and Application

3.1. This method is applicable for the measurement of pH in soils, sludges and solid wastes. If water is present, it must be less than 20% of the total volume of the sample.

3.2. Reporting limits, control limits, volumes used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted for use by, or under the supervision of, analysts experienced in the use of pH analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This procedure is applicable to extracts prepared from many types of solid waste and soil samples.

5. Limits of Detection and Quantitation

5.1. Refer to LIMS for method detection limits.

6. Interferences

6.1. Coatings of oily material or particulate matter on the electrode can impair response. This can be minimized by gently wiping off the electrode or washing with detergent followed by reagent water. An additional treatment with hydrochloric acid (1:9 ratio) may be necessary to remove any remaining film.

6.2. Samples with very low or very high pH may give incorrect readings on the pH meter. This error can be minimized by using a low-sodium-error electrode.

6.3. Temperature can have effects on results. To compensate for temperature interferences, the analyst should always calibrate the pH meter at the same temperature as the samples and they should record the sample temperature and pH at the time of analysis. Alternately, an automatic temperature compensating pH meter can be used.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Solids	>20g in 4oz glass container	None	Ambient	Analyze immediately**

** There is no holding time requirement listed in the method. SW-846 Chapter Three lists the holding time as "analyze immediately." All samples will be qualified as being analyzed outside recommended holding time.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Description / Comments
pH/Ion meter	Accumet AB15 or equivalent equipped with a combination pH probe and temperature sensor.
Analytical balance	OHaus AV212 or equivalent capable of weighing to 0.1g

9.2. General Supplies

Item	Description
Magnetic stirrer	With Teflon coated stir bars. Fisher Brand or equivalent
Disposable beakers	50mL capacity. Fisher Brand or equivalent
Graduated cylinder	Class A for measuring 20mL reagent water volume. Fisher Brand or equivalent
Digestion tubes	50mL capacity with lids. Environmental Express or equivalent
Filter paper	Fisher Brand P4 or equivalent
Tumbling apparatus	

10. Reagents and Standards

10.1. Reagents and Standards

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
pH 2 buffer	Fisher/ catalog # SB-96-1, or equivalent
pH 4 buffer	Fisher/ catalog # SB101-4 or equivalent
pH 7 buffer (CCV)	Fisher/ catalog # SB107-4 or equivalent
pH 10 buffer	Fisher/ catalog # SB115-4 or equivalent
pH 12 buffer	Ricca/ catalog #161532 or equivalent
pH 7 buffer (ICV)	Ricca/ catalog #155141 or equivalent

11. Calibration and Standardization

11.1. Refer to manufacturer's instructions for pH electrode and meter. If using a refillable electrode, uncover the fill hole on the pH electrode and add the appropriate fill solution to the electrode up to the fill hole. The level of the fill solution must always be above the reference junction. The fill hole should remain open when the electrode is in use. Store electrode in KCl filling solution when not in use.

11.2. Each instrument/electrode system must be calibrated on each analysis day with a minimum of two calibration points that bracket the expected pH of the samples. The two points should be at least three pH units apart. Place the appropriate buffer solution into a clean beaker using sufficient volume to cover the sensing elements of the electrodes and to give the magnetic stir bar some clearance to move.

11.3. Press and release the **mode** key until the digital display indicates pH mode.

11.4. Press the **setup** key twice and then press the **enter** key to clear an existing standardization.

11.5. Rinse the electrode with reagent water and immerse the rinsed electrode into the first buffer solution while gently stirring the buffer.

11.6. Press **std** to access Standardization mode. Wait for the reading to stabilize. Press **std** again to initiate standardization. The meter will automatically recognize the buffer and then return to the Measure screen. Record the pH reading of the buffer. Rinse off the electrode with reagent water and gently wipe dry.

11.7. Repeat steps 11.5-11.6 for each of the remaining buffer solutions.

11.8. Do not clear the previous buffers. Readings should be within 0.05 pH units from the listed values for the buffer solutions. When the three buffers have been standardized, record the slope from the meter. The slope must be 90-102%.

11.9. Check calibration with a second source pH 7 buffer (ICV). Record the pH. Results must be within +/- 0.10 pH units of the true value.

11.10. If either of the requirements of steps 11.8 or 11.9 are not met, repeat the meter calibration using fresh buffers. If either of the requirements are still not met, replace electrode filling solution and clean the electrode or replace the electrode.

11.11. Once calibrated, the meter is ready to analyze samples when in the **Measure** mode.

12. Procedures

12.1. Place 20g of sample into a 50mL digestion tube and add 20mL of reagent water. Cap the tube and tumble the suspension continuously for 5 minutes. Additional dilutions are allowed for problematic samples.

12.2. Filter or centrifuge the sample to separate the aqueous phase.

12.3. Decant the aqueous portion of prepared sample or transfer to a disposable beaker.

12.4. Immerse the electrode into the sample and stir gently at a constant rate. Record the sample pH to the nearest 0.1 pH units and record the temperature to the nearest ^{o}C when **STABLE** appears on the screen. For normal use, the pH meter Stability Criteria is set to slow, allowing the highest precision.

12.5. If the pH of the sample exceeds the calibration range (i.e. between 4 and 10), an additional pH buffer solution must be analyzed to confirm the accuracy of the pH meter beyond the calibration range. Results should be within +/- 0.10 pH units. If the pH of the additional buffer is not within +/-0.10 pH units, re-standardize the pH meter using buffers that bracket the pH range of the samples and reanalyze the samples.

12.6. Measure and record the pH of the calibration source pH 7 buffer (CCV) at the beginning and ending of each analytical sequence and after every 10 samples. Results should be within +/-0.05 pH units. If the pH reading of the CCV is not within +/-0.05 pH units of the true value, repeat the CCV measurement using fresh CCV. If CCV still fails, replace electrode filling solution and clean the electrode or replace the electrode and repeat calibration per Section 11. Repeat any samples that are not bracketed by acceptable buffer readings.

12.7. Thoroughly rinse and gently blot the electrode between sample measurements.

NOTE 1: Certain customer technical specifications may stipulate that their samples must be analyzed within 4 hours of the calibration.

NOTE 2: For samples originating in West Virginia, the pH meter must be calibrated prior to use with pH buffer standards that bracket the value to be measured.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Sample Duplicate	Sample	One duplicate per every 10 samples analyzed	≤2% RPD	Consult with supervisor to see if another sample should be duplicated.

13.2. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

14. Data Analysis and Calculations

14.1. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11, 12, and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11, 12, and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11, 12, and 13.

18. Method Performance

18.1. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

18.2. The analyst must read and understand this procedure with written documentation maintained in his/her training file.

19. Method Modifications

19.1. Samples are gently stirred during pH measurement.

19.2. Buffers are purchased as certified standards.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves**,** lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, method 9045C.

25.2. Accumet AB-15 or equivalent User Manual

25.3. Pace Analytical Quality Manual; latest revision.

25.4. TNI Standard; Quality Systems section; 2003, 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-069-rev.09	1. Cover: revised method reference to 9045C 2. Section 1: revised method reference to 9045C 3. Table 8.2: added graduated cylinder 4. Section 10: revised to be more specific to Accumet AR 25 meter. Added guidance for determining a stable reading. 5. Section 11: Added guidance for determining a stable reading. 6. Inserted new Method Modifications section. 7. Section 16.1: revised method reference to 9045C	19Oct2012
S-IN-I-069-rev.10	1. Section 9: tables combined into single Table 9.1 2. Table 9.1: added pH 11 buffer and identified ICV and CCV standards. 3. Section 10: copied Section 10 of pH Waters SOP for consistency. 4. Section 10.5: added (ICV) for clarity. 5. Section 11.6: changed "second source" to "calibration source" and revised criteria to +/-0.05 standard units. 6. Section 16: added reference to the pH meter user manual	30Jul2013
S-IN-I-069-rev.11	1. Table 7.1 and caption: revised holding time to "analyze immediately" and indicated that all sample results would be qualified as outside holding time. 2. Section 11 Note 1: removed specific reference to BP. 3. Section 11 Note 2: added for West Virginia requirements.	29Oct2013
S-IN-I-069-rev.12	1. Cover page: updated phone number and document control format. 2. Converted to 27-section format. 3. Section 9.1: updated pH meter identification. 4. Section 9.2: added tumbling device, digestion tubes and filter paper. 5. Section 10.1: removed pH 11 buffer and updated pH 12 and ICV buffers. 6. Section 11: updated calibration instructions based on current meter in use, added electrode handling information and added corrective actions. 7. Section 12.1: changed procedure for the use of digestion tubes. 8. Section 12.2: changed procedure to filtration for separation of solids from liquid. 9. Table 13.1: changed RPD to $\leq$2%. 10. Section 25: updated user manual reference and TNI reference.	06Feb2017

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STANDARD OPERATING PROCEDURE

MEASUREMENT OF PERCENT MOISTURE, TOTAL SOLIDS AND VOLATILE SOLIDS IN SOLIDS AND SEMI-SOLIDS

REFERENCE METHODS: STANDARD METHODS 2540G (1997)

SOP NUMBER:	S-IN-I-094-rev.08
EFFECTIVE DATE:	January 25, 2016
SUPERSEDES:	S-IN-I-094-rev.07

APPROVALS

General Manager — January 22, 2016 Date

Quality Manager — January 22, 2016 Date

Department Manager — January 22, 2016 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for measuring Percent Moisture, Total Solids and/or Volatile Solids in solid and semi-solid samples while meeting the requirements specified in Standard Method 2540G (1997).

2. Summary of Method

2.1. A sample aliquot is weighed before and after heating to dryness at 103°-105°C. The weight loss is calculated as percent moisture.

2.2. If Fixed Solids or Volatile Solids are also determined, the dried sample is transferred to a muffle furnace at 550°C. Weight loss is reported as Volatile Solids.

3. Scope and Application

3.1. Percent moisture is used to correct results of inorganic and organic tests to dry weight basis.

3.2. This procedure is restricted to use by, or under the supervision of, analysts experienced in the measurement of percent moisture.

4. Applicable Matrices

4.1. This method is applicable to most solid and semi-solid samples, including sludges.

5. Limits of Detections and Quantitation

5.1. This method is applicable to samples containing at least 0.1% moisture.

6. Interferences

6.1. Non-representative materials, such as leaves and sticks, should be removed from the sample prior to measurement.

6.2. Measurements are subject to negative bias for samples containing significant quantities of ammonium carbonate, volatile organics, or other volatile materials that could be lost during drying.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Solids for Percent Moisture	>100g in a glass container with a tightly fitting lid.	None required	0°C to 6°C	Samples must be analyzed within 14 days of collection
Solids for Total and/or Volatile Solids	>100g in a glass container with a tightly fitting lid.	None required	0°C to 6°C	Samples must be analyzed within 7 days of collection

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment

Equipment	Model / Version	Description / Comments
Analytical Balance	OHaus AV212 or equivalent	Capable of weighing to 0.01g
Drying Oven	Fisher IsoTemp or equivalent	Able to maintain a temperature of 103° to 105°C
Muffle Furnace	Thermolyne or equivalent	Able to maintain a temperature of 550°C
Desiccator	Fisher or equivalent	

9.2. General Supplies

Item	Model / ID	Description
Desiccant	Drier-ite or equivalent	
Weighing dishes	Fisher or equivalent	Disposable, aluminum
Porcelain Crucibles	Fisher or equivalent	For use in muffle furnace
Wooden Tongue Depressors	Fisher or equivalent	Or other device for transfer of sample to weighing dish

10. Reagents and Standards

10.1. Reagents- Not applicable to this SOP.

10.2. Analytical Standards- Not applicable to this SOP.

11. Calibration

11.1. The analytical balance must be calibrated by an outside vendor at least annually and checked each day of use with Class 1 weights.

12. Procedure

12.1. Allow samples to warm to room temperature prior to processing.

12.2. For each sample, label and record the tare weight of a weighing dish to the nearest 0.01g. Samples for Volatile Solids analysis must be dried in porcelain crucibles.

12.3. Remove the top portion of material from the sample container and transfer approximately 10g of sample to the tared weighing dish.

12.4. Weigh the wet sample and dish, recording the weight to the nearest 0.01g.

12.5. Place the samples in the drying oven and dry at 103°C - 105°C for at least 4 hours, preferably overnight.

12.6. Remove the sample from the oven and place into a desiccator to cool.

12.7. After the sample has cooled, weigh the dry sample and dish to the nearest 0.01g.

12.8. If the sample was not dried overnight, return it to the oven and dry at 103° - 105°C for an additional hour. Remove the sample from the oven and place into a desiccator to cool. Weigh the sample and dish to the nearest 0.01g. Repeat this process until a constant weight is achieved or until weight change is less than 4% of the previous weight or 50mg, whichever is less.

12.9. Calculate Percent Moisture per equation in Section 14.1. Calculate Total Solids per equation in Section 14.2.

12.10. Volatile Solids Determination

12.10.1. Transfer the crucible with dried sample from Section 12.8 to a cool muffle furnace.

12.10.2. Heat the muffle furnace to 550°C and ignite sample for 1 hour.

12.10.3. After 1 hour, remove samples to a heat resistant surface and allow to cool slightly before placing in desiccator.

12.10.4. Cool sample in desiccator then weigh.

12.10.5. Repeat ignition for intervals of 30 minutes, cooling and weighing until a constant weight is achieved or until weight change is less than 4% or 50mg, whichever is less.

12.10.6. Calculate Percent Volatile Solids per equation in Section 14.2.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Duplicate	Sample	One per batch of up to 10 samples.	≤5%.RPD	Analyze another sample and duplicate. Analyst can still report data from original duplicate as long as it is properly qualified.

14. Data Analysis and Calculations

14.1. Calculate Percent Moisture:

$$\% \text{ Moisture} = \frac{(C - A) * 100}{(C - B)}$$

Where: A = Weight of dried residue + dish, mg
B = Weight of dish
C = Weight of wet sample + dish, mg

14.2. Calculate Percent Total Solids:

$$\% \text{ Total Solids} = \frac{(A - B) * 100}{C - B}$$

Where: A = Weight of dried residue + dish, mg
B = Weight of dish
C = Weight of wet sample + dish, mg

14.3. Calculate Percent Volatile Solids:

$$\% \text{ Volatile Solids} = \frac{(A - D) * 100}{A - B}$$

Where: A = Weight of dried residue + dish, before ignition, mg
B = Weigh of dish
D = Weight of dried residue + dish, after ignition, mg

14.4. RPD calculation:

$$\mathbf{RPD} = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Section 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Section 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Section 13.

18. Method Performance

18.1. The analyst must read and understand this procedure with written documentation maintained in his/her training file.

19. Method Modifications

19.1. A nominal sample amount of 10g is used instead of 25g-50g.

19.2. Constant weight is not determined on samples that are dried overnight.

19.3. A holding time of 14 days is observed for Percent Moisture.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.2. Equipment: Preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

24. Pollution Prevention

24.1. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24.2. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Standard Methods for the Examination of Wastewater and Water; Methods 2540G (1997).

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; latest revision.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-094-rev.06	1. Revised cover page to reflect periodic review format. 2. Table of Contents fixed to include sections 10 and 11. 3. Table 7.1: clarified storage and hold time requirements. 4. Section 8: updated information and added "or equivalent" where applicable. 5. Section 15: changed NELAC to TNI.	31May2011
S-IN-I-094-rev.07	1. Section 11.8: clarified constant weight requirement. 2. Section 11.9: clarified calculation and defined wet sample weight and dry sample weight to be consistent with %M LIMSlink format. 3. Inserted new Method Modification section.	24Sep2012
S-IN-I-094-rev.08	1. Converted to 27-section format. 2. Cover page: changed phone number, added Total Solids, Volatile Solids and changed method reference to SM 2540G, revised effective date format and revised document control format. 3. Section 1.1: added Total Solids, Volatile Solids and changed method reference to SM2540G. 4. Section 2: added summary of Volatile Solids determination. 5. Section 4.1: added semi-solid matrix. 6. Table 7.1: changed holding time from 28 days to 7 days. 7. Section 9: added muffle furnace and porcelain crucibles. 8. Section 12.2: added requirement to use porcelain crucibles for Volatile Solids. 9. Section 12.8: added constant weight as change of 4% or 50mg, whichever is less. 10. Section 12.10: added procedure for Volatile Solids. 11. Section 14.2: added calculation for % Total Solids and % Volatile Solids. 12. Section 25: added SM2540G reference and removed ASTM reference.	11Jan2016

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STANDARD OPERATING PROCEDURE

ACID DIGESTION OF AQUEOUS SAMPLES FOR ICP AND ICP-MS ANALYSIS

REFERENCE METHOD: EPA SW-846 METHODS 3005A, 3010A AND EPA METHOD 200.2

SOP NUMBER:	S-IN-M-030-rev.13
EFFECTIVE DATE:	November 13, 2017
SUPERSEDES:	S-IN-M-030-rev.12

LOCAL APPROVAL

General Manager — November 3, 2017 Date

Quality Manager — November 3, 2017 Date

Department Manager — November 3, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for acid digestion of aqueous samples for metals analysis while meeting the requirements specified in EPA methods 3005A and 3010A for analysis by ICP and in EPA Method 200.2 for analysis by ICP-MS.

2. Summary of Method

2.1. A portion of sample is digested with strong acid and heat in a block digester and then brought to volume with reagent water.

3. Scope and Application

3.1. This procedure is used to determine total metals and dissolved metals.

3.2. Volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of metals digestion equipment. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This digestion procedure is used for the preparation of aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection	Preservation	Storage	Hold time
Aqueous - Total	250mL in plastic container	- HNO_3 to pH <2 - Samples received at pH>2 must be preserved to pH<2 with HNO_3 and be allowed to equilibrate for 24 hours before being prepared for analysis.	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.
Aqueous - Dissolved	250mL in plastic container	- Filter; HNO_3 to pH<2 - For all dissolved elements by methods 200.7 or 200.8 and for **all dissolved elements by methods 200.7/200.8/6010/6020 for West Virginia**, samples must be filtered within 15 minutes of collection and before adding HNO_3, or data must be qualified that filtration occurred beyond 15 minutes of collection. - Samples filtered in the lab are preserved to pH<2 with HNO_3 and allowed to equilibrate for 24 hours before being prepared for analysis.	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Description / Comments
Hot Block Digester	Environmental Express or equivalent, adjustable and capable of maintaining a temperature of 92°C to 98°C.
Centrifuge	Fisher Centrific centrifuge Model 225 or equivalent
Vacuum pump	For lab filtration for dissolved elements.

9.2. General Supplies

Item	Description
Volumetric Flasks	Class A , various capacities
Volumetric Pipettors	Eppendorf or equivalent, various sizes
Digestion Tubes	Environmental Express or equivalent, volumetrically certified and contaminant free
Thermometer	Ever Safe or equivalent, calibrated, used for monitoring Hot Block temperature
Plunger Filters	Environmental Express or equivalent
Graduated Cylinders	Class A, various capacities
pH strips	Fisher or equivalent, full range
Filtration system	FlipMate or equivalent 0.45 um fiber filter disc caps and cups for lab filtration for dissolved elements.

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Nitric acid	Concentrated, trace metal analyzed or equivalent
Hydrochloric acid	Concentrated, trace metal analyzed or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Table 10.1 Standard Definitions

Standard	Description	Comments
Spiking Standard	This solution contains the target analytes and is generally prepared using a standard source secondary to the standards used for calibration.	Same solution can be used for the LCS and MS/MSD

10.2.2. Storage Conditions

Table 10.2 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
ICP Stock Spiking Standards	Inorganic Ventures; catalog #s PA-STD-1B; PA-STD-2B; PA-STD-3B, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
ICP Working Spiking Standard	Refer to Section 10.2.3.1.	Expires 6 months from date of preparation.	Same as stock standard
ICP-MS Stock Spiking Standard #1	Inorganic Ventures; catalog #HERT-CAL-2A or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
ICP-MS Stock Spiking Standard #2	Inorganic Ventures; catalog #HERT-CAL-2B or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions

10.2.3. Preparation Procedures

Table 10.3 – ICP Stock Spiking Standard Details

Analyte	Concentration (mg/L)
Inorganic Ventures PA-STD-1B	
Arsenic	200
Barium	200
Beryllium	200
Cadmium	200
Chromium	200
Cobalt	200
Copper	200
Lead	200
Lithium	200
Manganese	200
Nickel	200
Phosphorus	200
Selenium	200
Strontium	200
Thallium	200
Vanadium	200
Zinc	200
Inorganic Ventures PA-STD-2B	
Antimony	200
Boron	200
Molybdenum	200
Silicon	1000
Inorganic Ventures PA-STD-2B continued	
Silver	100
Tin	200
Titanium	200
Inorganic Ventures PA-STD-3B	
Aluminum	2000
Calcium	2000
Iron	2000
Magnesium	2000
Potassium	2000
Sodium	2000

Table 10.4 – ICP-MS Stock Spiking Standard Details

Analyte	Concentration (mg/L)
Inorganic Ventures HERT-CAL-2A	
Antimony	2
Molybdenum	2
Tin	2
Titanium	2
Inorganic Ventures HERT-CAL-2B	
Aluminum	20
Arsenic	2
Barium	2
Beryllium	2
Boron	2
Cadmium	2
Chromium	2
Cobalt	2
Copper	2
Lead	2
Manganese	2
Nickel	2
Selenium	2
Silver	2
Strontium	2
Thallium	2
Thorium	2
Uranium	2
Vanadium	2
Zinc	2

10.2.3.1. Working Spiking Standard Preparation

Dilute 25mL of each stock spiking standard (solutions 1B, 2B and 3B) to 100mL with reagent water for a final nominal concentration of 50mg/L.

11. Calibration

11.1. Not applicable to this SOP.

12. Procedures

12.1. If lower reporting limits are required, digestate concentration may be performed provided that final acid concentration and final spike concentration remain consistent with unconcentrated digestates. Refer to Section 12.4.

12.2. Lab Filtration for Dissolved Elements

12.2.1. Prepare the filtration apparatus by attaching a filter disc cap to a sample cup for each sample to be filtered.

12.2.2. To filter, attach the filtration apparatus to the vacuum pump and turn the pump on. Turn the vacuum pump off when filtration is complete and an adequate volume of filtrate has been collected.

12.2.3. Prepare a Method Blank by filtering reagent water through the filter disc cap and into a labeled sample cup. Filter enough volume to support all analyses requested.

12.2.4. Prepare an LCS by filtering an LCS prepared as described in Section 12.3.3 or 12.4.3 through the filter disc cap and into a labeled sample cup. Filter enough volume to support all analyses requested.

12.2.5. Filter samples by pouring the sample from the original sample container into the filter disc cap and collecting approximately 100mL of the sample filtrate in a labeled sample cup.

12.2.6. Preserve all filtrates to pH<2 with concentrated nitric acid. Hold preserved samples for a minimum of 24 hours before digestion and/or analysis.

12.2.7. Record all filtration information including sample cup lot number, filter disc cap lot number, and date and time of preservation in the metals digestion log.

12.3. Aqueous Sample Digestion for ICP

12.3.1. Transfer 50mL of well-mixed sample into a labeled digestion tube.

12.3.2. Prepare a Method Blank by transferring 50mL of reagent water to a digestion tube.

12.3.3. Prepare an LCS by adding 1mL of the ICP Working Spiking Standard (50mg/L nominal) to 50mL of reagent water for a nominal spike concentration of 1mg/L.

12.3.4. Prepare a Matrix Spike by adding 1mL of the ICP Working Spiking Standard (50mg/L nominal) to 50mL of sample for a nominal spike concentration of 1mg/L.

12.3.5. Add 2.5mL concentrated nitric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of 95^{o}C (+/- 3^{o}C) in the digestion tubes.

12.3.6. If digestate is generating brown fumes, add another 2.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

12.3.7. Evaporate without boiling to approximately 10mL. Do not allow samples to go dry.

12.3.8. Cool the samples then add 2mL concentrated hydrochloric acid, return the samples to the hot block and heat for 15 minutes to dissolve any precipitate then allow samples to cool.

12.3.9. Dilute the digestates to 50mL in the digestion tube with reagent water. If necessary, filter the digestates to remove particulates by using a plunger filter. If any sample digestates in a batch are filtered, the Method Blank and LCS must also be filtered. Alternatively, reduce the effect of particulates by placing the samples into a centrifuge for approximately 5 minutes at 3500 rpm.

12.4. Aqueous Sample Digestion for ICP-MS

12.4.1. Transfer 50mL of well-mixed sample into a labeled digestion tube.

12.4.2. Prepare a Method Blank by transferring 50mL of reagent water to a digestion tube.

12.4.3. Prepare an LCS by adding 1mL of the ICP-MS Stock Spiking Standard #1 (2mg/L) and 1mL of the ICP-MS Stock Spiking Standard #2 (2mg/L nominal) to 50mL of reagent water for a nominal spike concentration of 0.04mg/L.

12.4.4. Prepare a Matrix Spike by adding 1mL of the ICP-MS Stock Spiking Standard #1 (2mg/L) and

1mL of the ICP-MS Stock Spiking Standard #2 (2mg/L nominal) to 50mL of sample for a nominal spike concentration of 0.04mg/L.

12.4.5. Add 0.5mL concentrated nitric acid and 0.25mL concentrated hydrochloric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of 95°C (+/- 3°C) in the digestion tubes.

12.4.6. If digestate is generating brown fumes, add another 0.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

12.4.7. Evaporate without boiling to approximately 10mL. Do not allow samples to go dry.

12.4.8. Remove the samples from the hot block and allow them to cool.

12.4.9. Dilute the digestates to 50mL in the digestion tube with reagent water. If necessary, reduce the effect of particulates by placing the samples into a centrifuge for approximately 5 minutes at 3500 rpm.

12.5. Aqueous Sample Digestion with Concentration for ICP

12.5.1. Transfer 50mL of well-mixed sample into a labeled digestion tube.

12.5.2. Prepare a Method Blank by transferring 50mL of reagent water to a digestion tube.

12.5.3. Prepare an LCS by adding 0.2mL of the ICP Working Spiking Standard (50mg/L nominal) to 50mL of reagent water for a nominal spike concentration of 1mg/L.

12.5.4. Prepare a Matrix Spike by adding 0.2mL of the ICP Working Spiking Standard (50mg/L nominal) to 50mL of sample for a nominal spike concentration of 1mg/L.

12.5.5. Add 0.5mL concentrated nitric acid to each digestion tube. Place the tubes into the block digester and set the temperature to achieve 95°C (+/- 3°C) in the digestion tubes.

12.5.6. If digestate is generating brown fumes, add another 0.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

12.5.7. Evaporate without boiling to approximately 10mL. Do not allow samples to go dry.

12.5.8. Cool the samples then add 0.4mL concentrated hydrochloric acid, return the samples to the hot block and heat for 15 minutes to dissolve any precipitate then allow samples to cool.

12.5.9. Dilute the digestates to 10mL in the digestion tube with reagent water. If necessary, filter the digestates to remove particulates using a plunger filter. If any sample digestates in a batch are filtered, the Method Blank and LCS must also be filtered. Alternatively, reduce the effect of particulates by placing the samples into a centrifuge for approximately 5 minutes at 3500 rpm.

12.6. Record all preparation information including standard numbers, reagent numbers, digestion tube lot numbers, filter lot numbers, Hot Block number, thermometer ID and correction factor, and digestion temperature in the metals digestion log and deliver the digestates to the ICP analyst.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Refer to the SOP for the determinative method.	Refer to the SOP for the determinative method
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples, per matrix.	Refer to the SOP for the determinative method.	Refer to the SOP for the determinative method.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Refer to the SOP for the determinative method.	Refer to the SOP for the determinative method.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Action for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Samples are digested for the ICP analysis of Antimony, Boron, Lithium, Silicon, Silver, Strontium, Tin and Titanium in addition to the analytes listed in the method.

19.2. Samples are digested for the ICP-MS analysis of Titanium in addition to the analytes listed in the method.

19.3. Volumes of acid used for ICP digestion vary from those in the methods.

19.4. Method modified for use with Hot Block digesters and digestion tubes are never capped while heating.

19.5. A digestion temperature range of 95°C +/-3°C is observed.

20. Instrument/Equipment Maintenance

20.1. Refer to maintenance log and/or instrument manufacturer's instructions.

21. Troubleshooting

21.1. Refer to maintenance log and/or instrument manufacturer's instructions.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock metals standards are toxic and should be handled with extreme care. Also handle concentrated acids with care, making sure to wear appropriate personal protective equipment.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves**,** lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

22.3. Equipment: Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Preventions

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, latest revision, Methods 3005A and 3010A.

25.2. U.S. EPA, EMSL Method 200.2, Revision 2.8, 1994.

25.3. Pace Analytical Quality Manual; latest revision.

25.4. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-M-030-rev.11	1. Cover Page: changed phone number, changed method reference to 3010A and 200.2, add ICP-MS reference updated effective date format and changed SOP naming format from inorganics to metals. 2. Table of Contents: added new Section 14, Method Modifications. 3. Section 1.1: changed method reference to 3010A and 200.2 and added ICP-MS reference. 4. Table 7.1: added preservation for lab-filtered samples. 5. Table 8.1: updated required temperature range of Hot Block. 6. Section 9: added ICP-MS spiking standards. 7. Section 11: revised to include batch QC preparation, added guidance for non-standard final volumes, revised acid-addition to reflect current process, and separated process for ICP and ICP-MS digestion. Added specific procedure for digestate concentration to achieve lower limits. 8. Table 12.1: updated Method Blank corrective action. 9. Section 13: removed MDL requirement. 10. Section 14: new Method Modifications section added. 11. Section 16.1: changed method reference to 3010A and added 200.2 reference.	17Sep2015
S-IN-M-030-rev.12	1. Converted to 27 section format. 2. Table 7.1: added requirement to filter within 15 minutes of collection for methods 200.7 and 200.8 and revised storage temperature format. 3. Section 9.1: added centrifuge. 4. Section 12.2: changed final evaporation volume from 5mL to 10mL and added centrifuge as option to filter. 5. Section 12.3: changed final evaporation volume from 5mL to 10mL and changed filtration to centrifugation. 6. Section 12.4: changed final evaporation volume from 5mL to 10mL and added centrifuge as option to filter. 7. Table 13.1: referred to SOP for determinative method for acceptance criteria. 8. Section 25.4: added years 2003 and 2009 to TNI reference.	08Oct2017
S-IN-M-030-rev.13	1. Cover page: added reference to method 3005A. 2. Section 1.1: added reference to method 3005A. 3. Table 7.1: added requirement that all West Virginia samples for dissolved elements must be filtered within 15 minutes of collection. 4. Section 9.1: added vacuum pump. 5. Section 9.2: added filtration apparatus. 6. Section 12.2: added section to describe lab filtration procedure for dissolved elements. 7. Table 13.1: referred to determinative method for corrective actions. 8. Section 25.1 added reference to method 3005A.	24Oct2017

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STANDARD OPERATING PROCEDURE

ACID DIGESTION OF SOLID SAMPLES FOR ICP AND ICP-MS ANALYSIS

REFERENCE METHOD: EPA SW-846 METHOD 3050B

SOP NUMBER:	S-IN-M-031-rev.11
EFFECTIVE DATE:	November 2, 2015
SUPERSEDES:	S-IN-I-031-rev.10

APPROVAL

General Manager — October 27, 2015 Date

Quality Manager — October 27, 2015 Date

Department Manager — October 26, 2015 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide two specific laboratory procedures for acid digestion of solid samples and wipes for metals analysis while meeting the requirements specified in EPA method 3050B. The digestates are then analyzed by inductively coupled plasma (ICP) by Method 6010B or by inductively coupled plasma-mass spectrometry (ICP-MS) by Method 6020.

2. Summary of Method

2.1. A sample is digested with strong acid and hydrogen peroxide and heat in a block digester and then brought to volume with reagent water.

3. Scope and Application

3.1. This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available."

3.2. This method is applicable to the digestion of sediments, sludges, soils, solids and wipes. Other elements and matrices may be analyzed by this method if performance is demonstrated for the analytes of interest in the matrices of interest at the concentration of interest.

3.3. The extracts from these two procedures are not interchangeable and should only be used with the analytical determinations outlined.

3.4. Volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.5. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of metals digestion equipment. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. Not applicable to this SOP.

5. Safety

5.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock metals standards are toxic and should be handled with extreme care. Also handle concentrated acids with care, making sure to wear appropriate personal protective equipment.

5.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

5.3. Equipment: Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

6. Definitions

6.1 Refer to Glossary section of the Pace Quality Assurance Manual for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Solid	50 grams in glass or plastic container	No chemical preservation required	0°C to 6°C	Must be analyzed within 6 months of the collection date.
Wipe	Individual wipe in glass or plastic container	No chemical preservation required	0°C to 6°C	Must be analyzed within 6 months of the collection date.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment/Instrumentation

Equipment	Description / Comments
Hot Block Digester	Environmental Express or equivalent, adjustable and capable of maintaining a temperature of 92°C to 98°C.
Analytical Balance	Capable of weighing to 0.01g

8.2. General Supplies

Item	Description
Volumetric Flasks	Class A , various capacities
Volumetric Pipettors	Eppendorf or equivalent, various sizes
Digestion Tubes	Environmental Express or equivalent, volumetrically certified and contaminant free
Thermometer	Ever Safe or equivalent, calibrated, used for monitoring Hot Block temperature
Plunger Filters	Environmental Express FilterMate or equivalent
Graduated Cylinders	Class A, various capacities
Ghost wipes	Environmental Express mixed cellulose ester wipe or equivalent
Plastic beads	Environmental Express or equivalent for use as a simulated solid matrix

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Nitric acid	Concentrated, trace metal analyzed or equivalent
Hydrochloric acid	Concentrated, trace metal analyzed or equivalent
Hydrogen Peroxide	30% solution, trace metal analyzed or equivalent

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions

Standard	Description	Comments
Spiking Standard	This solution contains the target analytes and is generally prepared using a standard source secondary to the standards used for calibration.	Same solution can be used for the LCS and MS/MSD

9.2.2. Storage Conditions

Standard Type	Description	Expiration	Storage
ICP Stock Spiking Standard	Inorganic Ventures; catalog #s PA-STD-1B; PA-STD-2B; PA-STD-3B, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
ICP Working Spiking Standard	Refer to Section 9.2.3.1.	Expires 6 months from date of preparation.	Same as stock standard
ICP-MS Stock Spiking Standard #1	Inorganic Ventures; catalog #HERT-CAL-2A or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
ICP-MS Stock Spiking Standard #2	Inorganic Ventures; catalog #HERT-CAL-2B or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions

Table 9.3 – Analytical Standard Storage Conditions

9.2.3. Preparation Procedures

Table 9.3 – ICP Stock Spiking Standard Details

Analyte	Concentration (mg/L)
Inorganic Ventures PA-STD-1B	
Arsenic	200
Barium	200
Beryllium	200
Cadmium	200
Chromium	200
Cobalt	200
Copper	200
Lead	200
Lithium	200
Manganese	200

Inorganic Ventures PA-STD-1B continued	
Nickel	200
Phosphorus	200
Selenium	200
Strontium	200
Thallium	200
Vanadium	200
Zinc	200
Inorganic Ventures PA-STD-2B	
Antimony	200
Boron	200
Molybdenum	200
Silicon	1000
Silver	100
Tin	200
Titanium	200
Inorganic Ventures PA-STD-3B	
Aluminum	2000
Calcium	2000
Iron	2000
Magnesium	2000
Potassium	2000
Sodium	2000

Table 9.4 – ICP-MS Stock Spiking Standard Details

Analyte	Concentration (mg/L)
Inorganic Ventures HERT-CAL-2A	
Antimony	2
Molybdenum	2
Tin	2
Titanium	2
Inorganic Ventures HERT-CAL-2B	
Aluminum	20
Arsenic	2
Barium	2
Beryllium	2
Boron	2
Cadmium	2
Chromium	2
Cobalt	2
Copper	2
Lead	2
Manganese	2
Nickel	2
Selenium	2
Silver	2
Strontium	2
Thallium	2
Thorium	2
Uranium	2
Vanadium	2
Zinc	2

9.2.3.1. ICP Working Spiking Standard Preparation

Dilute 25mL of each stock spiking standard (solutions 1B, 2B and 3B) to 100mL with reagent water for a final nominal concentration of 50mg/L.

10. Calibration

10.1. Not applicable to this SOP.

11. Procedures

11.1. Solid Sample Preparation for ICP

11.1.1. Weigh 1-1.2g of well-mixed sample to the nearest 0.01g into a labeled digestion tube and add 5mL reagent water.

11.1.2. Prepare a Method Blank by weighing 1g of plastic beads into a digestion tube and adding 5mL reagent water.

11.1.3. Prepare an ICP LCS by weighing 1g of plastic beads into a digestion tube, adding 1mL of the ICP Working Spiking Standard (50mg/L) and 5mL reagent water for a nominal spike concentration of 50mg/Kg.

11.1.4. Prepare an ICP Matrix Spike by weighing 1g of well-mixed sample into a digestion tube, adding 1mL of the ICP Working Spiking Standard (50mg/L) and 5mL reagent water for a nominal spike concentration of 50mg/Kg.

11.2. Solid Sample Preparation for ICP-MS

11.2.1. Weigh 0.5-0.54g of well-mixed sample to the nearest 0.01g into a labeled digestion tube and add 2.5mL reagent water.

11.2.2. Prepare a Method Blank by weighing 0.5g of plastic beads into a digestion tube and adding 2.5mL reagent water.

11.2.3. Prepare an ICP-MS LCS by weighing 0.5g of plastic beads into a digestion tube, adding 1mL of the ICP-MS Stock Spiking Standard #1 (2mg/L) and 1mL of the ICP-MS Stock Spiking Standard #2 (2/20mg/L) and 5mL reagent water for a nominal spike concentration of 2mg/Kg.

11.2.4. Prepare an ICP-MS MS by weighing 0.5-0.54g of sample into a digestion tube, adding 1mL of the ICP-MS Stock Spiking Standard #1 (2mg/L) and 1mL of the ICP-MS Stock Spiking Standard #2 (2/20mg/L) and 2.5mL reagent water for a nominal spike concentration of 2mg/Kg

11.3. Wipe Sample Preparation

11.3.1. Place the wipe sample into a labeled digestion tube and add 20mL reagent water to completely cover the wipe.

11.3.2. Prepare a Method Blank by placing an unused wipe into a digestion tube and adding 20mL reagent water to completely cover the wipe.

11.3.3. Prepare an LCS by placing an unused wipe into a digestion tube and adding 20mL reagent water to completely cover the wipe. Add 1mL of the ICP Working Spiking Standard (50mg/L) for a nominal spike concentration of 50 Total ug.

11.4. Sample Digestion for ICP Analysis

11.4.1. Add 5mL of concentrated nitric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of 95°C (+/- 3°C) in the digestion tubes..

11.4.2. If brown fumes are generated, repeat step 11.4.1 until no brown fumes are given off by the sample, indicating the complete reaction with nitric acid.

11.4.3. Evaporate to approximately 5mL or heat without boiling for 2 hours then cool. Do not allow samples to go dry.

11.4.4. Add 1mL 30% hydrogen peroxide to each tube and warm them slowly on the digestion block until the effervescing stops**.**

11.4.5. Continue to add 30% hydrogen peroxide in 1mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 5mL 30% hydrogen peroxide.

11.4.6. Continue heating until the volume has been reduced to approximately 5mL or heat without boiling for 2 hours. Do not allow samples to go dry.

11.4.7. Add 5mL concentrated hydrochloric acid to each tube and heat for 15 minutes. Cool and dilute each sample to 50mL with reagent water. Filter the samples to remove particulates using a plunger filter. The associated Method Blank and LCS must also be filtered

11.5. Sample Digestion for ICP-MS Analysis

11.5.1. Add 2.5mL of concentrated nitric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of 95°C (+/- 3°C) in the digestion tubes.

11.5.2. Loosely cap and reflux samples for 10-15 minutes without boiling then remove from the block digester and allow them to cool.

11.5.3. Add 2.5mL concentrated nitric acid and reflux for 2 hours.

11.5.4. If brown fumes are generated, repeat step 11.5.3 until no brown fumes are given off by the sample indicating the complete reaction with nitric acid.

11.5.5. Add 1.0mL reagent water then add 1.5mL 30% hydrogen peroxide to each tube and warm them slowly on the digestion block until the effervescing stops.

11.5.6. Continue to add 30% hydrogen peroxide in 0.5mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 2.5mL 30% hydrogen peroxide.

11.5.7. Continue refluxing for 2 hours. Do not allow samples to go dry.

11.5.8. Cool and dilute each sample to 50mL with reagent water. Filter the samples to remove particulates using a plunger filter. The associated Method Blank and LCS must also be filtered.

11.6. Record all information including standard numbers, reagent numbers, digestion tube lot numbers, filter lot numbers, Hot Block number, thermometer ID and correction factor and digestion temperature in the metals digestion log and deliver the digestates to the ICP or ICP-MS analyst.

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	1g simulated solid matrix for solid samples or one unused ghost wipe for wipe samples	One per preparation batch of up to 20 samples, per matrix.	Refer to determinative method.	Re-digest and reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte + 1g simulated solid matrix for solid samples or one unused ghost wipe for wipe samples	One per preparation batch of up to 20 samples, per matrix.	80-120% Recovery	Re-digest and reanalyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte + sample	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	75-125% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

12.2. LCS calculation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.3. MS/MSD calculation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4. RPD calculation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

12.5. Wipe Calculation:

Total ug analyte per wipe = (analyte conc., ug/L) x (0.050 Liter/wipe)

13. Method Performance

13.1. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14. Method Modifications

14.1. Samples are digested for the ICP analysis of Arsenic, Boron, Lithium, Phosphorus, Selenium, Strontium, Tin and Titanium in addition to the analytes listed in the method.

14.2. Samples are digested for the ICP-MS analysis of Aluminum, Antimony, Barium, Boron, Copper, Manganese, Nickel, Silver, Strontium, Thorium, Tin, Titanium, Uranium, and Zinc in addition to the analytes listed in the method.

14.3. Method modified for use with Hot Block digesters.

14.4. Method modified for the digestion of wipes.

14.5. A digestion temperature range of 95°C +/-3°C is observed**.**

14.6. Progress of digestion is measured in terms of the digestate volume and not by elapsed time.

14.7. First aliquot of hydrogen peroxide is 1mL instead of 1.5mL for a 50mL final volume for the ICP digestion.

14.8. Plunger filters are used with digestion cups instead of traditional gravity filtration. Plunger filter is more retentive than the filter described in the method.

15. Pollution Prevention and Waste Management

15.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

15.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

15.3. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16. References

16.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, latest revision, Method 3050B.

16.2. Pace Analytical Quality Manual; latest revision.

16.3. TNI Standard; Quality Systems section; latest revision.

17. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1. Not applicable to this SOP.

18. Revisions

Document Number	Reason for Change	Date
S-IN-I-031-rev.9	1. Table of Contents: fixed to include Sections 10 and 11. 2. Sections 8 and 9: revised to be in list form and removed catalog number references. 3. Section 9.2.3.2: reworded for clarity. 4. Table 9.3: changed working standard expiration to 6 months. 5. Table 12.1: added 10x rule exception for Method Blank corrective action.	04Jan2010
S-IN-I-031-rev.10	1. Cover page revised to most recent format. 2. Table 7.1: revised storage requirements and volume required and added wipes. 3. Section 8: converted to a tabular format and updated information, added wipes. 4. Section 9: converted to a tabular format and updated information. 5. Section 11.1: added "well-mixed" and procedure for wipes 6. Section 11.3: updated peroxide volumes added. 7. Section 11.6: added LCS after method blank in last sentence. 8. Section 11.7: added Hot Block number and digestion temperature as information to be recorded. 9. Section 12: revised Table 12.1 and added preparation instructions for Method Blank, LCS and Matrix Spike. 10. Section 13: removed SOP references. 11. Section 15: changed NELAC to TNI.	30Aug2011
S-IN-M-031-rev.11	1. Cover Page: added address and phone number, added ICP-MS, updated effective date format and changed SOP naming format from inorganics to metals. 2. Table of Contents: added new Section 14, Method Modifications. 3. Section 3.2: added "solids" as an applicable matrix. 4. Table 8.1: updated required temperature range of Hot Block. 5. Table 8.2: added simulated solid matrix and removed pH strips. 6. Section 9.2: added spiking standards for ICP-MS, added Table 9.4 and updated ICP standards 7. Section 11: revised to include batch QC preparation for ICP and ICP-MS, separated solid and wipe prep and digestion processes, added digestion process for ICP-MS. 8. Table 12.1: changed Teflon chips to simulated solid matrix and updated Method Blank corrective action. 9. Section 13: removed MDL requirement. 10. Section 14: new Method Modifications section added.	26Oct2015

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP)
REFERENCE METHOD: EPA SW-846 METHOD 6010B

SOP NUMBER:	S-IN-M-019-rev.11
EFFECTIVE DATE:	January 11, 2016
SUPERSEDES:	S-IN-I-019-rev.10

APPROVAL

General Manager — January 5, 2016 Date

Quality Manager — December 24, 2015 Date

Department Manager — December 28, 2015 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of metals in aqueous and solid environmental samples while meeting the requirements specified in SW-846 method 6010B.

2. Summary of Method

2.1. 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.

2.2. This method describes multielement determinations by Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES). 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 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.

2.3. Background correction may be required to compensate for spectral interferences. Background is measured adjacent to analyte lines at a wavelength selected to be free of spectral interference and which reflects the same change in background intensity as occurs at the wavelength measured. Background correction is not required in cases of line broadening where a correction would actually degrade the analytical result.

3. Scope and Application

3.1. This method is applicable to the determination of most trace elements, including metals, in solution.

3.2. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of ICP systems and interpretation of ICP data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable to groundwater, surface water, wastewater, extract, leachate, soil, sediment, sludge and other solid samples.

5. Limits of Detection and Quantitation

5.1. Refer to Table 1 for the list of metals and reporting limits. Refer to the LIMS for method detection limits.

6. Interferences

6.1. **Spectral interferences:** Overlap of emission lines from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and stray light can contribute to spectral interferences. These interferences can typically be minimized by careful selection of quantitation wavelengths, inter-element corrections, and background correction.

6.2. **Physical interferences:** Changes in sample viscosity, surface tension, or other effects associated with sample transport and nebulization can produce significant inaccuracies, especially in samples containing high concentrations of dissolved solids and acids. Dissolved solids may build up on the nebulizer tip, altering the sample flow rate and causing instrument drift. These effects can be minimized by sample

dilution or use of a specially designed high-solids nebulizer.

6.3. High Salt Concentrations: high salt concentrations in sample digestates can cause signal suppression and confuse interference tests.

6.4. Chemical interferences: Molecular compound formation, ionization effects, and solute vaporization effects are typically not significant with ICP determinations. If observed, they can be minimized by careful selection of plasma and spectrometer operating parameters.

6.5. Memory interferences: Sample deposition on the nebulizer tubing, spray chamber, and plasma torch can cause apparent sample carryover. Memory interferences can be minimized by flushing the system with rinse blanks between samples. If memory interference is suspected for a sample, the sample must be re-analyzed after a sufficient rinse period.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous - Total	250mL in plastic container	- HNO_3 to pH of <2 - Samples received at pH>2 must be preserved to pH<2 with HNO_3 and equilibrate for 24 hours before being prepared for analysis. Record date/time of preservation in preservation logbook.	Ambient or 0°C to 6°C	Must be analyzed within 6 months of the collection date.
Aqueous - Dissolved	250mL in plastic container	- Filter; HNO_3 to pH<2	Ambient or 0°C to 6°C	Must be analyzed within 6 months of the collection date.
Solid	50 grams in glass or plastic container	- No chemical preservation	Ambient or 0°C to 6°C	Must be analyzed within 6 months of the collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Vendor	Description / Comments
ICP-AES	Thermo-Fisher iCAP6500 or equivalent	Equipped with and autosampler and data system

9.2. General Supplies

Item	Vendor	Description
Volumetric Flasks	Class A	Various capacities
Volumetric Pipettors	Eppendorf or equivalent	Various sizes
Autosampler Vials	Environmental Express or equivalent	
Analytical Balance	Ohaus or eqivalent	Capable of weighing to 0.01g
Graduated Cylinders	Class A	Various capacities
pH strips	Fisher or equivalent	Full range

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Argon	High purity, liquefied
Nitric acid	Concentrated, trace metal analyzed or equivalent
Hydrochloric acid	Concentrated, trace metal analyzed or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	ICAL
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains the target analytes and is used to spike MS/MSD sets..	Same solution can be used for the LCS and MS/MSD
Internal Standard	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	Yttrium
Interference Check Standards	Prepared to contain a known amount of interfering elements that will provide an accurate test of the interelement correction factors. If the ICP will display overcorrection as a negative number, the additional spiking with interfered elements is not necessary..	ICSA (ICSAB for BP Samples only)

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Calibration Standards	SPEX; catalog #'s MIXSTD1-100; MIXSTD2-100; MIXSTD3-100; MIXSTD4-100; MIXSTD5-100; PLS19-2Y; CLSN2-2Y; PLTI9-2Y; PLLI-2Y; PLP9-3Y; CLAG2-2Y or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Calibration Standards	Refer to Section 10.2.3.2	Must be prepared fresh weekly	Same as stock standards
Stock ICV Standard	Inorganic Ventures; catalog #s PA-STD-1B; PA-STD-2B; PA-STD-3B or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working ICV Standard	Refer to Section 10.2.3.4	Must be prepared fresh weekly	Same as stock standard
Working Second Source Spiking Solution	Refer to Section 10.2.3.5	Expires 6 months from date of preparation.	Same as stock standard

Standard Type	Description	Expiration	Storage
Stock Interference Check Standard A	SPEX; catalog # INT-A1 or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Interference Check Standard A (ICSA)	Refer to Section 10.2.3.6	Must be prepared fresh weekly	Same as stock standards
Stock Interference Check Standard AB	SPEX; catalog #INT-A1, Mix-1B, Mix-2B, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Interference Check Standard AB (ICSAB)	Refer to Section 10.2.3.8	Must be prepared fresh weekly	Same as stock standards
Stock CRDL standards	SPEX individual standards for each element; 1000 or 10,000 mg/L, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate CRDL Standard	Refer to Section 10.2.3.11	Expires 6 months from date of preparation.	Same as stock standards
Working CRDL standard	Refer to Section 10.2.3.12	Must be prepared fresh weekly	Same as stock standards
Stock Internal Standard	SPEX; catalog # PLY2-2X; 1000mg/L yttrium or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Internal Standard	Refer to Section 10.2.3.13	Must be prepared fresh weekly	Same as stock standards

10.2.3. Standard Preparation Procedures

10.2.3.1. Stock Calibration Standard Details

The following table shows the seven stock standard mixes that may be used to prepare the initial calibration and calibration check standards:

Analyte	Concentration (mg/L)
Catalog # MIXSTD1-100	
Lead	500
Selenium	200
Cadmium	150
Zinc	150
Manganese	100
Beryllium	50
Catalog # MIXSTD2-100	
Iron	10,000
Barium	100
Cobalt	100
Copper	100
Vanadium	100
Catalog # MIXSTD3-100 + silicon PLSI9-2Y	
Arsenic	500
Molybdenum	100
Silicon	100
Catalog #MIXSTD4-100	
Calcium	1000
Potassium	400

Catalog #MIXSTD4-100 Cont'd	
Aluminum	200
Sodium	200
Chromium	20
Nickel	20
Catalog #MIXSTD5-100	
Magnesium	1000
Antimony	200
Thallium	200
Boron	100
Silver	50
Mix #6(combines: CLSN2-2Y; PLTI9-2Y, PLLI-2Y, PLP9-3Y)	
Lithium	1000
Phosphorus	10,000
Tin	1000
Titanium	1000
Mix #7- Catalog #CLAG2-2Y	
Silver	1000

10.2.3.2. Working Calibration Standards Preparation

Prepared fresh weekly and diluted from the stock standard mixes listed above, using a reagent water mixture that is 5% nitric acid and 2% hydrochloric acid unless otherwise noted.

Working Std. ID	Stock Standard	Vol. of Stock Std.	Final Volume
Calibration Std. Mix 1	MIXSTD1-100	2mL	100mL
Calibration Std. Mix 2	MIXSTD2-100	1mL	100mL
Calibration Std. Mix 3	MIXSTD3-100 Silicon PLSI9-2Y	2mL 0.8mL	100mL
Calibration Std. Mix 4	MIXSTD4-100	5mL	100mL
Calibration Std. Mix 5	MIXSTD5-100	2mL	100mL
Calibration Std. Mix 6	Lithium PLLI-2Y Phosphorus PLP9-3Y Tin CLSN2-2Y Titanium PLTI9-2Y	1mL 0.1mL 1mL 1mL	100mL
Calibration Std. Mix 7	Silver CLAG2-2Y	0.2mL	100mL in 10% HCl solution

10.2.3.3. Stock ICV Standard Details

The following table shows the concentrations of the stock standards purchased from Inorganic Ventures as three mixes:

Analyte	Concentration (mg/L)
Inorganic Ventures PA-STD1B	
Arsenic	200
Barium	200
Beryllium	200
Cadmium	200
Cobalt	200
Chromium	200
Copper	200
Manganese	200

Inorganic Ventures PA-STD1B Cont'd	
Nickel	200
Phosphorus	200
Lead	200
Selenium	200
Thallium	200
Lithium	200
Strontium	200
Vanadium	200
Zinc	200
Inorganic Ventures PA-STD2B	
Silicon	1000
Boron	200
Molybdenum	200
Antimony	200
Tin	200
Titanium	200
Zirconium	200
Silver	100
Inorganic Ventures PA-STD3B	
Aluminum	2000
Calcium	2000
Iron	2000
Potassium	2000
Magnesium	2000
Sodium	2000

10.2.3.4. Working ICV Standard Preparation

Add 0.5mL of each Stock ICV Standard mix to a 100mL volumetric flask and dilute to volume with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

10.2.3.5. Working Second Source Spiking Solution

Add 25.0 mL of each Stock ICV Standard mix to a 100 mL volumetric flask and dilute to volume with reagent water solution that is 2% nitric acid.

10.2.3.6. Stock Interference Check Standard A (ICSA) Details

SPEX Interference Check Standard A (ICSA)	
Aluminum	5000
Calcium	5000
Magnesium	5000
Iron	2000

10.2.3.7. Working Interference Check Standard A (ICSA) Preparation

Dilute 10mL of the Stock ICSA Standard to 100mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

10.2.3.8. Stock Interference Check Standard AB (ICSAB) Details

SPEX INT-A1	
Al, Ca, Mg	5000
Fe	2000
SPEX Mix 1B	
As, Ba, Be, B, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Tl, V, Zn	100
SPEX Mix 2B	
Mo, Sb, Sn, Ti	100
Ag	50
Si	500

10.2.3.9. Working Interference Check Standard AB (ICSAB) Preparation

Dilute 10mL of the stock INT-A1 standard and 0.5mLof the stock Mix 1B and Mix 2B to 100mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

10.2.3.10. Stock CRDL Standards Detail

When specified by client or program requirements, a low-level check standard, also known as a CRDL standard, must be analyzed prior to sample analysis and at the end of each analytical batch to bracket the client samples. Acceptance limits for all target elements is 50-150% recovery. The Stock CRDL standards are as follows:

Element	Conc. (ug/mL)	SPEX Catalog #	Element	Conc. (ug/mL)	SPEX Catalog #
Aluminum	1000	CLAL2-2Y	Manganese	1000	CLMN2-2Y
Antimony	1000	CLSB7-2Y	Molybdenum	1000	CLMO9-2Y
Arsenic	1000	CLAS2-2Y	Nickel	1000	CLNI2-2Y
Barium	1000	CLBA2-2Y	Phosphorus	10,000	PLP9-3Y
Beryllium	1000	CLBE2-2Y	Potassium	10,000	PLK2-3Y
Boron	1000	PLB9-2Y	Selenium	1000	CLSE2-2Y
Cadmium	1000	CLCD2-2Y	Silicon	1000	PLSI9-2Y
Calcium	10,000	PLCA2-3Y	Silver	1000	CLAG2-2Y
Chromium	1000	CLCR2-2Y	Sodium	10,000	PLNA2-3Y
Cobalt	1000	PLCO2-2Y	Strontium	1000	PLSR2-2Y
Copper	1000	CLCU2-2Y	Thallium	1000	CLTL2-2Y
Iron	1000	CLFE2-2Y	Tin	1000	CLSN2-2Y
Lead	1000	CLPB2-2Y	Titanium	1000	CLTI9-2Y
Lithium	1000	PLLI2-2Y	Vanadium	1000	CLV2-2Y
Magnesium	10,000	PLMG2-3Y	Zinc	1000	CLZN2-2Y

10.2.3.11. Intermediate CRDL Standard Preparation

Dilute the following volumes of the stock CRDL standards to 50mL with a reagent water solution that is 2% nitric acid:

Element	Volume (mL)	Final Conc. (mg/L)	Element	Volume (mL)	Final Conc. (mg/L)
Aluminum	2.5	50	Manganese	0.125	2.5
Antimony	0.075	1.5	Molybdenum	0.125	2.5
Arsenic	0.125	2.5	Nickel	0.125	2.5
Barium	0.125	2.5	Phosphorus	1.25	250
Beryllium	0.05	1	Potassium	1.25	250
Boron	1.25	25	Selenium	0.125	2.5
Cadmium	0.025	0.5	Silicon	2.5	50
Calcium	1.25	250	Silver	0.125	2.5
Chromium	0.125	2.5	Sodium	1.25	250
Cobalt	0.125	2.5	Strontium	0.125	2.5
Copper	0.125	2.5	Thallium	0.125	2.5
Iron	1.25	25	Tin	0.125	2.5
Lead	0.125	2.5	Titanium	0.125	2.5
Lithium	0.25	5	Vanadium	0.125	2.5
Magnesium	1.25	250	Zinc	0.25	5

10.2.3.12. Working CRDL Standard Preparation

Dilute 1mL of the Intermediate CRDL Standard to 250mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid. Final concentrations are shown below.

Element	Final Conc. (ug/L)	Element	Final Conc. (ug/L)
Aluminum	200	Manganese	10
Antimony	6	Molybdenum	10
Arsenic	10	Nickel	10
Barium	10	Phosphorus	1000
Beryllium	4	Potassium	1000
Boron	100	Selenium	10
Cadmium	2	Silicon	200
Calcium	1000	Silver	10
Chromium	10	Sodium	1000
Cobalt	10	Strontium	10
Copper	10	Thallium	10
Iron	100	Tin	10
Lead	10	Titanium	10
Lithium	20	Vanadium	10
Magnesium	1000	Zinc	20

10.2.3.13. Working Internal Standard Preparation

Dilute 5mL of yttrium stock standard (1000mg/L) to 1L with a reagent water solution that is 2% nitric acid for a final concentration of 5mg/L.

11. Calibration

11.1. Initial Calibration: Calibrate the ICP each working day according to the instrument manufacturer's recommended procedures. Flush the system with the Calibration Blank solution prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. The calibration curve must consist of a minimum of a calibration blank and a standard.

11.2. Linear Calibration: Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The analyst may employ a regression equation that does not pass through the origin. If a multi-point calibration is performed, the regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995.

11.3. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.4. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110% and the RSD of replicate readings must be <5%.

11.5. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.6. Initial Calibration Blank (ICB): The ICB consists of a reagent water solution that is 5% HNO_3 and 2% HCl. An ICB must be analyzed immediately following the ICV. If the ICB result is above the reporting limit, another ICB may be analyzed. If the second ICB fails, then a new initial calibration curve must be analyzed. Samples associated with a failed ICB must be reanalyzed. **Exception:** If the ICB is >RL, associated samples determined to be <RL are reportable. **If required by client or program, the ICB must be evaluated as follows: If the ICB result exceeds ½ the RL, the ICB is considered to be unacceptable. Only samples determined to be <RL are reportable. If the absolute value of a negative concentration exceeds twice the established MDL, the ICB is considered to be unacceptable. Samples associated with a failed ICB must be re-analyzed unless the concentration of the target analyte is greater than 10 times the absolute value of the ICB result.**

11.7. Continuing Calibration Verification (CCV): A CCV must be analyzed immediately following the ICB, after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The acceptable recovery range for the CCV is 90-110% and the RSD of replicate readings must be <5%.

11.8. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

11.9. Contract Required Detection Limit (CRDL) Standard: A CRDL standard must be analyzed with each analytical run, at a minimum, after calibration. Acceptance limits for all target elements is 50-150%

recovery. If required by client or program, another CRDL standard must be analyzed after samples – not to exceed 8 hours between CRDL analyses.

11.10. CRDL Corrective Action: Samples associated with a failed CRDL must be re-analyzed unless the concentration of the target has failed high, then the associated samples determined to be <RL are reportable.

11.11. Interference Check Standard A (ICSA): An ICSA must be analyzed at the beginning of each analytical run. ICSA must be 80-120% of the true value for the elements in the mix. Non-ICSA elements must be within +/-2x the reporting limit.

11.12. ICSA Corrective Action: If the ICSA fails the acceptance criteria, another ICSA may be analyzed. If the second ICSA fails, then a new calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICSA must be reanalyzed. **Exception:** If the ICSA is >120% for any element in the mix or if any non-ICSA element is >2x the reporting limit, indicating high bias, associated samples determined to be <RL are reportable.

11.13. Interference Check Standard AB (ICSAB): If required by client or program an ICSAB must be analyzed at the beginning of each analytical run. ICSAB must be 80-120% of the true value for the elements in the mix.

11.14. ICSAB Corrective Action: If an ICSAB is required by client or program and the ICSAB fails the acceptance criteria, another ICSAB may be analyzed. If the second ICSAB fails, then a new calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICSAB must be reanalyzed. **Exception:** If the ICSAB is >120% for any element in the mix, indicating high bias, associated samples determined to be <RL are reportable.

11.15. Continuing Calibration Blank (CCB): The CCB consists of a reagent water solution that is 5% HNO_3 and 2% HCl. A CCB must be analyzed after every 10 samples following the CCV. If the CCB result is above the reporting limit, another CCB may be analyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable. **If required by client or program, the CCB must be evaluated as follows: If the CCB result exceeds ½ the RL, the CCB is considered to be unacceptable. Only samples determined to be <RL are reportable. If the absolute value of a negative concentration exceeds twice the established MDL, the CCB is considered to be unacceptable. Samples associated with a failed CCB must be re-analyzed unless the concentration of the target analyte is greater than 10 times the absolute value of the CCB result.**

12. Procedure

12.1. Before using this procedure to analyze samples, there must be data available documenting initial demonstration of performance. The required data document the selection criteria of background correction points; analytical dynamic ranges; the applicable equations, and the upper limits of those ranges; the method and instrument detection limits; and the determination and verification of interelement correction equations or other routines for correcting spectral interferences. This data must be generated using the same instrument, operating conditions and calibration routine to be used for sample analysis.

12.2. Configure the ICP per manufacturer's instructions and allow it to become thermally stable.

12.3. Approximately 10mL portions of each standard, Method Blank, LCS, sample and MS/MSD are poured into autosampler tubes for analysis.

12.4. Establish initial calibration as described in Section 10.

12.5. Once initial calibration is established, analyze each sample, Method Blank, LCS and MS/MSD. An example sequence may be as follows:

Initial calibration blank
Mix 1
Mix 2
Mix 3
Mix 4
Mix 5
Mix 6
Mix 7
ICV
ICB
CRDL
ICSA
ICSAB (if required)
Method blank
LCS
Client samples
CCV
CCB
Client samples
CRDL (if required)
ICSA (if required)
ICSAB (if required)
CCV
CCB

12.6. The instrument performs two replicate readings for each analysis and the average of the two readings is used to derive the concentration. For samples, the difference between the two readings must be $\leq$20% RSD for values that are >4x the reporting limit. If the RSD is >20% for values that are >4x the reporting limit, the sample must be reanalyzed.

12.7. Samples with analyte concentrations above the upper linear range must be diluted and reanalyzed or the over range results must be qualified as estimated.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or boiling chips	One per preparation batch of up to 20 samples, per matrix.	Target analyte must be less than reporting limits	Re-digest and reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required. 3) If sample concentration is >10x blank level, sample and method blank may be reported, but sample must be qualified. (Not for VAP)
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples, per matrix.	80-120% Recovery	Re-digest and reanalyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	75-125% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Internal Standard	Yttrium	Automatically added to each sample, blank, and standard as part of the analysis.	No acceptance criteria – used to monitor interferences. 60-140%	No corrective action required. Sample may be analyzed at a dilution if interference is indicated.

13.2. Post-Digestion Spike Addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrument detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

13.3. Dilution test: If the analyte concentration is sufficiently high, minimally, a factor of 10 above the instrument detection limit after dilution, an analysis of a 1:5 dilution should agree within +/-10% of the original determination. If not, a chemical or physical interference effect should be suspected.

14. Data Analysis and Calculations

14.1. Calculations for water samples are performed directly by the instrument software since initial sample aliquot and final digestate volumes are the same. If dilutions were performed, the appropriate factors must be applied to sample values.

14.2. The data system calculates the soil samples using the following equation:

$$C_s = \frac{C * V * D}{W}$$

Where: Cs = sample concentration (mg/kg, dry-weight basis)
C = concentration in extract (average of two readings taken)
V = volume of extract
D = dilution factor
W = dry weight of solid sample extracted (kg)

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$$

14.3. LCS equation:

$R = (C/S) * 100$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.4. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.5. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. **Method Detection Limit (MDL) Study**: An MDL study must be conducted every 12 months for each matrix per instrument.

18.2. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

18.3. **Linear Dynamic Range Study**: A linear dynamic range study must be conducted for each element by analyzing increasing concentrations of at least three, preferably five different concentration standards across the range. One of these should be near the upper limit of the range. The upper range limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Samples determined to be above the upper range limit must be diluted and reanalyzed. New dynamic ranges should be determined whenever there is a significant change in instrument response. For those analytes that periodically approach the upper limit, the range should be checked every six months. Refer to Section 7.2.5.4 of Method 6010B for more information.

18.4. **Interelement Correction Factors** must be verified and updated every 6 months or when an instrumentation change occurs. Refer to Section 3.1 of Method 6010B for more information.

19. Method Modifications

19.1. Mixed standard solutions are purchased as certified standards.

19.2. Instrument conditions may vary from those stated in the method.

19.3. Calibration blanks are evaluated to the reporting limit and not to three times the IDL.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock metals standards are toxic and must be handled with

extreme care. Also handle concentrated acids with care, making sure to wear appropriate personal protective equipment.

22.2. **Samples**: Take precautions when handling samples. Samples must always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment**: Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

24. Pollution Prevention

24.1. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24.2. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, latest revision, Method 6010B.

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; latest revision.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Table 1: Target Metals and Reporting Limits

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-019-rev.09	1. Section 2: streamlined summary of method 2. Section 3: removed SOP references and added a statement that weights, volumes, limits, etc… are subject to change. 3. Section 7: updated storage requirements, clarified hold time requirements and added requirements for dissolved metals. 4. Section 8: changed to a tabular format and updated information. Removed supplies relating to sample digestion. 5. Section 9: changed to a tabular format 6. Section 10: completely revised to clarify each QC sample and its corrective action, added calibration calculations and added Arsenic profiling criteria. 7. Section 11: removed table of ICP settings and added final concentration calculations. 8. Table 12.1: removed CCB, CRDL, ICSA, ICSAB and As profiling. Added RPD criteria for MS/MSD 9. Section 13: removed SOP references, added IDL information and expanded LDR information 10. Section 15: changed NELAC to TNI 11. Section 16: updated title of Attachment 1 12. Attachment 1: removed wavelengths and 200.7 information and update RLs.	23Jun2011
S-IN-I-019-rev.10	1. Section 3.1: added reference to MDLs. 2. Table 9.2: removed reference to Profiling Standard 3. Table 9.3: removed reference to Profiling Standard 4. Section 9.2.3: removed reference to Profiling Standard 5. Section 9.2.3.2: changed to a tabular format 6. Section 10: removed reference to Profiling Standard. 7. Section 11.9: added language that over range results can be reported if qualified as estimated. 8. Table 12.1: revised method blank corrective action. 9. Inserted new Method Modifications section.	19Sep2012
S-IN-M-019-rev.11	1. Converted SOP to Corporate 27-section format. 2. Cover page: changed phone number, changed effective date format and revised document control format. 3. Table 7.1: added requirement to record date/time of preservation. 4. Table 10.3: updated standard sources and added Li and P. 5. Section 10.2.3: updated standard preparation. 6. Section 11: removed linear regression equation, made CRDL a requirement for each analytical batch, and removed BP requirements and replaced with “If required by client or program.” 7. Table 1: updated RLs and added Li, Sr, and P.	18Dec2015

Table 1: Target Metals and Reporting Limits

Metals	Aqueous (µg/L)	Solid (mg/kg)
Aluminum - Al	200	50
Antimony - Sb	6	1
Arsenic - As	10	1
Barium - Ba	10	1
Beryllium - Be	4	0.5
Boron - B	100	5
Cadmium - Cd	2	0.5
Calcium - Ca	1000	50
Chromium - Cr	10	1
Cobalt - Co	10	1
Copper - Cu	10	1
Iron – Fe	100	50
Lead – Pb	10	1
Lithium – Li	20	5
Magnesium – Mg	1000	50
Manganese – Mn	10	1
Molybdenum - Mo	10	1
Nickel – Ni	10	1
Phosphorus – P	N/A	50
Potassium - K	1000	50
Selenium - Se	10	1
Silver – Ag	10	0.5
Sodium – Na	1000	50
Strontium – Sr	10	1
Thallium - Tl	10	1
Tin – Sn	10	5
Titanium - Ti	10	1
Vanadium - V	10	1
Zinc – Zn	20	1

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF MERCURY BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY

REFERENCE METHODS: EPA SW-846 METHODS 7470A AND 7471A

SOP NUMBER:	S-IN-M-040-rev.15
EFFECTIVE DATE:	July 5, 2017
SUPERSEDES:	S-IN-M-040-rev.14

APPROVAL

General Manager — June 22, 2017 Date

Quality Manager — June 22, 2017 Date

Department Manager — June 22, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining total mercury concentration while meeting the requirements specified in EPA method 7470A for aqueous samples and method 7471A for solid samples.

2. Summary of Method

2.1. Prior to analysis, all samples are digested by heating with appropriate acids and oxidizing agents to dissolve and oxidize mercury contents.

2.2. This cold-vapor method is based on the absorption of radiation at 253.7nm 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.

3. Scope and Application

3.1. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.2. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of mercury analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable for the measurement of mercury in groundwater, surface and saline waters, domestic and industrial wastes, TCLP extracts, soil, sediment, bottom deposits and sludge-type materials.

5. Limits of Detection and Quantitation

5.1. The default reporting limits for mercury are 0.002 mg/L for aqueous samples and 0.20 mg/kg for solid samples. Refer to the LIMS for method detection limits.

6. Interferences

6.1. High concentrations of sulfide may interfere in some water or solid samples. Potassium permanganate is added during digestion to eliminate sulfide interference. Concentrations as high as 20mg/L in water or 20mg/kg in soils have been demonstrated to cause no interference in spiked samples.

6.2. High concentrations of copper have been reported to interfere with mercury determinations. Concentrations as high as 10mg/L in water or 10mg/kg in soil have been demonstrated to cause no interference in spiked samples.

6.3. High concentrations of chloride, present in samples require additional potassium permanganate. The free chlorine produced during digestion should be removed with excess hydroxylamine hydrochloride solution.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous - Total	500 mL in plastic container.	HNO_3 to pH<2 Samples received at pH>2 must be preserved to pH<2 with HNO_3 and be allowed to equilibrate for 24 hours before being prepared for analysis. Record date/time of preservation in preservation logbook.	Ambient	Analysis must be completed within 28 days of collection date.
Aqueous - Dissolved	500 mL in plastic container	Filter; HNO_3 to pH<2	Ambient	Analysis must be completed within 28 days of collection date.
Solid	100g in a 4oz glass container	None	Cool to ≤6°C	Analysis must be completed within 28 days of collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Automated Mercury Analyzer	CETAC/Teledyne Leeman	M-6100, M-7600 or equivalent	To include an atomic absorption spectrophotometer, mercury lamp, absorption cell, air pump, flow meter, drying tube, autosampler and data system.
Hot Block	Environmental Express	56-well or equivalent	Adjustable and capable of maintaining a temperature of 90°C to 95°C.
Balance	OHaus	GT400 or equivalent	Readability to 0.01g

9.2. General Supplies

Item	Vendor	Description
Auto-pipettes	Eppendorf or equivalent	Various sizes
Volumetric flasks	Class A	100mL
Graduated cylinder	Class A	25mL
Digestion cups	Environmental Express or equivalent	50mL capacity, volumetrically certified
Autosampler tubes	Moldpro, Inc or equivalent	17x100 mm
Plunger filters	Environmental Express or equivalent	For use with digestion cups

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Hydrochloric acid	Concentrated, trace metal grade or equivalent
Hydrochloric acid (3%)	Dilute 30 mLs of concentrated HCl to 1L with reagent water. Used for dilution preparation.
Nitric acid	Concentrated, trace metal grade or equivalent
Sulfuric acid	Concentrated, trace metal grade or equivalent
Aqua Regia	Carefully add one volume of nitric acid to three volumes of hydrochloric acid. Must be prepared in a hood and must be prepared immediately before use each day.
Stannous Chloride	Crystals, reagent grade
Stannous Chloride solution	Add 100g stannous chloride and 70 mL conc. HCl in reagent water and dilute to 1L. This solution is good for 3 days. Refrigerate when not in use.
Sodium Chloride	Crystals, reagent grade
Hydroxylamine hydrochloride	Crystals, reagent grade
Sodium Chloride/ Hydroxylamine Hydrochloride solution	Dissolve 120g sodium chloride and 120g hydroxylamine hydrochloride in reagent water and dilute to 1L. This solution is good for 6 months from preparation (hydroxylamine sulfate may be substituted for hydroxylamine hydrochloride).
Potassium permanganate solution (5%)	Commercially purchased Mercury-free, 5% solution (w/v)
Potassium persulfate	Crystals, reagent grade
Potassium persulfate solution	Dissolve 50g Potassium persulfate in reagent water and dilute to 1L. Expires 6 months from the date of preparation and can be stored at room temperature.
Rinse/Probe Wash Solution	Add 25mL HNO3 and 10mL HCl to 500mL reagent water and dilute to 1L.
Boiling chips	Or equivalent to be used as a simulated soil matrix.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Contract Required Detection Limit Standard	A standard prepared at a concentration equivalent to the reporting limit for verification at that level.	CRDL only if required by program or client
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains the target analyte and is used to spike MS/MSD sets.	Same solution can be used for both the LCS and MS/MSD.

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Mercury Calibration standard	Ricca; catalog # AHG1KN; 1000mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Mercury Calibration standard	Refer to Section 10.2.3.1	Solution expires 6 months from date of preparation.	Same as for stock standard.
Daily Spike Mercury Calibration standard	Refer to Section 10.2.3.2	Must be prepared fresh daily.	Not Applicable
Working Mercury Calibration standards	Refer to Section 10.2.3.3	One-time use standards.	Not Applicable
Stock Mercury ICV/Spiking standard	SPEX; catalog # PLHG4-2Y; 1000mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Mercury ICV/Spiking standard	Refer to Section 10.2.3.4	Solution expires 6 months from date of preparation.	Same as for stock standard.
Daily Spike Mercury ICV/Spiking standard	Refer to Section 10.2.3.5	Must be prepared fresh daily.	Not Applicable
Working Mercury ICV standard	Refer to Section 10.2.3.6	One-time use standard.	Not Applicable
Working CRDL standard	Refer to Section 10.2.3.3	One-time use standard.	Not Applicable

10.2.3. Standard Preparation Procedures

Refer to the standard preparation logbook or database for specific instructions regarding preparation of standards for Mercury analysis

10.2.3.1 Intermediate Mercury Calibration Standard Preparation

Dilute 1mL of the Stock Mercury Calibration Standard (1000mg/L) to 100mLs with 2% HNO_3 for a final concentration of 10mg/L. This standard is good for 6 months from the date of preparation.

10.2.3.2 Daily Spike Mercury Calibration Standard Preparation

Dilute 1mL of the Intermediate Mercury Calibration Standard (10mg/L) to 100mLs with 2% HNO_3 for a final concentration of 100ug/L. This standard must be prepared fresh daily.

10.2.3.3 Working Mercury Calibration Standards Preparation

Working calibration standards are one-time use and are prepared by diluting the Daily Spike Mercury Calibration Standard (100ug/L) with reagent water. Examples of possible calibration standards are as follows:

Aqueous:

Standard ID	Amount of Daily Spike	Final Volume in reagent water	Final Concentration
Standard 1 (CRDL)	0.06mL	30mL	0.2ug/L
Standard 2	0.3mL	30mL	1.0ug/L
Standard 3	0.6mL	30mL	2.0ug/L
Standard 4 (CCV)	1.5mL	30mL	5.0ug/L
Standard 5	2.25mL	30mL	7.5ug/L
Standard 6	3.0mL	30mL	10.0ug/L

Solid:

Standard ID	Amount of Daily Spike	Final Volume in reagent water	Final Concentration
Standard 1 (CRDL)	0.1mL	50mL	0.2ug/L
Standard 2	0.5mL	50mL	1.0ug/L
Standard 3	1.0mL	50mL	2.0ug/L
Standard 4 (CCV)	2.5mL	50mL	5.0ug/L
Standard 5	3.75mL	50mL	7.5ug/L
Standard 6	5.0mL	50mL	10.0ug/L

10.2.3.4 Intermediate Mercury ICV/Spiking Standard Preparation

Intermediate Mercury ICV Standard: Dilute 1mL of the Stock Mercury ICV/Spiking Standard (1000mg/L) to 100mLs with 2% HNO_3 for a final concentration of 10mg/L. This standard is good for 6 months from the date of preparation.

10.2.3.5 Daily Spike Mercury ICV/Spiking Standard Preparation

Dilute 1mL of the Intermediate Mercury ICV/Spiking Standard (10mg/L) to 100mLs with 2% HNO_3 for a final concentration of 100ug/L. This standard must be prepared fresh daily and is also used to prepare the LCS and MS/MSD.

10.2.3.6 Working Mercury ICV Standard Preparation

Aqueous: Dilute 1.5mL of the Daily Spike Mercury ICV Standard (100ug/L) to 30mL with reagent water for a standard concentration of 5.0ug/L. This standard is a one-time use standard.

Solid: Dilute 2.5mL of the Daily Spike Mercury ICV Standard (100ug/L) to 50mL with reagent water for a standard concentration of 5.0ug/L. This standard is a one-time use standard.

11. Calibration

11.1. Initial Calibration: A minimum of a calibration blank and five calibration standards is required. The lowest calibration standard must be at or below the reporting limit. A new initial calibration curve with freshly prepared standard is analyzed on each working day. Refer to the Quality Manual for more information regarding calibration curves.

11.2. Linear Calibration: Using the instrumentation software, prepare a standard curve by plotting absorbance versus mercury concentration of each calibration standard. The analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995.

11.3. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be digested analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed. Refer to Section 11.12 for additional information.

11.4. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110%.

11.5. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. Exception: If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported. Refer to Section 11.12 for additional information.

11.6. Initial Calibration Blank (ICB): The ICB consists of reagent water that is prepared per Section 11. An ICB must be analyzed immediately following the ICV. If the ICB result is above the reporting limit, the ICB may be reanalyzed. If the second ICB fails, then a new initial calibration curve must be analyzed. Samples associated with a failed ICB must be reanalyzed. **Exception:** If the ICB is >RL, associated samples determined to be <RL are reportable. Refer to Section 11.12 for additional information.

11.7. Continuing Calibration Verification (CCV): A CCV must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable recovery range for the CCV is 90-110%.

11.8. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable. Refer to Section 11.12 for additional information.

11.9. Continuing Calibration Blank (CCB): The CCB consists of reagent water that is prepared per Section 11. A CCB must be analyzed after each CCV. If the CCB result is above the reporting limit, the CCB may be reanalyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable. Refer to Section 11.12 for additional information.

11.10. Contract Required Detection Limit Standard (CRDL): The CRDL is an optional check standard at or below the concentration of the reporting limit that is only analyzed if required by program or client. If required by client or program, the CRDL must be analyzed at the beginning of an analytical run, after every 20 samples, and at the end of the analytical run. The acceptable recovery range for the CRDL is 50-150%.

11.11. CRDL Corrective Action: If the CRDL is required by client or program and fails the acceptance criteria, another CRDL may be analyzed. If the second CRDL fails, associated samples must be qualified. **Exception:** If the CRDL is required and is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable without qualification.

11.12. Failure of the initial calibration, ICV, CCV, ICB or CCB that is due to improper or inadequate preparation requires the re-digestion and reanalysis of the associated preparation batch(es). Failure of the initial calibration, ICV, CCV, ICB or CCB due to instrument malfunction requires the instrument to be restored to proper working order and the reanalysis of samples associated with the failed QC.

12. Procedures

12.1. Aqueous Sample Preparation

12.1.1 Transfer a 30mL aliquot of well-mixed sample to a labeled 50mL graduated digestion cup.

12.1.2 Prepare a Method Blank by adding 30mL of reagent water to a labeled digestion cup.

12.1.3 Prepare an LCS by adding 1.5mL of the Daily Spike Mercury ICV/Spiking Standard (100ug/L) to a labeled digestion cup and diluting to 30mL with reagent water for a spike concentration of 5.0ug/L.

12.1.4 Prepare an MS and MSD set by transferring 30mL aliquots of well-mixed sample to separate labeled digestion cups and adding 1.5mL of the Daily Spike Mercury ICV/Spiking Standard (100ug/L) for a spike concentration of 5.0ug/L

12.1.5 Add 0.75mL concentrated HNO_3 to each digestion cup then add 1.5mL concentrated H_2SO_4 to each digestion cup, mixing after each addition.

12.1.6 Add 5mL of 5% potassium permanganate solution to each digestion cup. Ensure that equal amounts of permanganate solution are added to Method Blank and LCS. Swirl to mix. If the purple color does not persist after 15 minutes, then start over at Section 12.1.1 using a diluted aliquot of sample.

12.1.7 Add 2.5mL potassium persulfate solution to each digestion cup, cap loosely and heat samples for 2 hours in the Hot Block at 95°C.

12.1.8 Cool samples and add 1.8mL of sodium chloride/hydroxylamine hydrochloride solution to each sample to reduce the excess potassium permanganate. **CAUTION**: perform this addition in a fume hood, as chlorine gas could be produced. Proceed to Section 12.4.

12.2. Solid Sample Preparation

12.2.1 Weigh 0.3g of sample into a labeled 50mL digestion cup. To ensure the sample is representative of the entire container, the analyst should weigh out three 0.1g aliquots from different parts of the same container.

12.2.2 Prepare a Method Blank by placing several boiling chips into a labeled digestion cup.

12.2.3 Prepare an LCS by placing several boiling chips into a labeled digestion cup and adding 1.5mL of the Daily Spike Mercury ICV/Spiking Standard (100ug/L) for a final concentration of 0.5mg/Kg.

12.2.4 Prepare an MS and MSD by weighing 0.3g portions of a sample into separate labeled digestion cups and adding 1.5mL of the Daily Spike Mercury ICV/Spiking Standard (100ug/L) for a spike concentration of 0.5mg/Kg.

12.2.5 Add 5mL of reagent water to each digestion cup.

12.2.6 Add 2.5mL of aqua regia to each digestion cup.

12.2.7 Heat samples for 2 minutes in the Hot Block at 95°C.

12.2.8 Cool samples and add 25mL reagent water then add 7.5mL of 5% potassium permanganate

solution. Loosely cap each digestion cup.

12.2.9 Return the samples to the Hot Block and heat for 30 minutes at 95°C.

12.2.10 Cool samples again and add 3mL of the sodium chloride/hydroxylamine hydrochloride solution to each sample to reduce the excess potassium permanganate. **CAUTION**: perform this addition in a fume hood, as chlorine gas could be produced.

12.2.11 Adjust the digestate volumes to 50mL with reagent water and mix. Proceed to Section 12.4.

12.2.12 If needed, a plunger filter may be used to filter the digestate. The Method Blank and LCS must also be filtered if any client samples are filtered in the batch.

12.3. Calibration Standard Preparation

12.3.1 Prepare calibration standards in labeled 50mL digestion cups per the instructions in Section 10.2.3.3.

12.3.2 Follow steps in Section 12.1 to prepare calibration standards for aqueous matrix.

12.3.3 Follow steps in Section 12.2 to prepare calibration standards for solid matrix.

12.4. All sample volumes, reagent volumes, spiking standard volumes, standard/reagent ID numbers, hot block ID numbers, hot block temperature, thermometer ID number, and preparation date and time must be recorded in the electronic prep log.

12.5. Determination of Mercury

12.5.1 Configure the mercury analyzer according to manufacturer's instructions. Allow the colorimeter and recorder to warm up. Run a baseline with all reagents, using reagent water to flush the tubing. Whenever new tubing is used, allow ample time to flush the tubing.

12.5.2 Approximately 10mL portions of each standard, Method Blank, LCS, sample and MS/MSD are poured into autosampler tubes for analysis.

12.5.3 Establish initial calibration as described in Sections 11.1 through 11.6.

12.5.4 Once initial calibration is established, analyze each sample, Method Blank, LCS and MS/MSD. An example sequence may be as follows:

Initial calibration standards
ICV
ICB
CRDL (only if required)
Method blank
LCS
Client samples
CCV
CCB
Client samples
CCV
CCB
CRDL (only if required)

12.6. Any sample digestate with a mercury concentration that exceeds the linear range of the calibration curve must be diluted with 3% HCl solution and re-analyzed or over range results must be qualified as estimated. Alternatively, the sample may be re-digested at a dilution and re-analyzed.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or boiling chips	One per preparation batch of up to 20 samples, per matrix.	Target analyte must be less than reporting limits	Re-digest and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples, per matrix.	80-120% Recovery	If original LCS is outside acceptance limits, re-analyze the LCS. If LCS is still outside acceptance limits, re-digest and re-analyze associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	75-125% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

14. Data Analysis and Calculations

14.1. Calculations are performed directly by the instrument software. If dilutions were performed, the appropriate factors must be applied.

14.2. The instrument software calculates the amount of Mercury in the sample aliquot as follows:

$$X_s = (y - b)/a$$

Where: X_s = Concentration of the analyte
y = Total area or response of the analyte
a = slope of the line (the coefficient of x)
b = intercept of the line

14.3. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_f)(D)}{(V_i)} \qquad \text{Solid Sample (mg/kg)} = \frac{(X_s)(V_f)(D)}{(W_s)} \times 1000$$

Where: X_s = Mercury concentration, ug/L
V_f = Final sample volume of digestate, L
D = Dilution factor of the sample digestate
V_i = Initial sample volume digested, L
W_s = Weight of solid sample digested, mg

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100- \%Moisture)}} \times 100$$

14.4. LCS equation:

$$\mathbf{R = (C/S) * 100}$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.5. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.6. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. Method Detection Limit (MDL) Study: An MDL study must be conducted annually for each matrix per instrument.

18.2. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Digestion procedure modified to use digestion cups in a hot block instead of BOD bottles in a water bath.

19.2. Standards and some reagents purchased as certified solutions.

19.3. Stannous Chloride solution not stirred continually because it is a solution and not a suspension.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. Equipment: Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 7470A and 7471A .

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not Applicable

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-040-rev.12	1. Section 3.2: added reference to MDLs and revised RL for solids. 2. Section 9.1: added potassium persulfate, wash solution and boiling chips. Revised or expanded other reagents. 3. Table 9.2: added RLVS 4. Table 9.3: added RLVS 5. Section 9.2.3.2: added RLVS 6. Section 10: added RLVS 7. Section 11.1.5: added that digestion cups be capped loosely for digestion. 8. Section 11.4.4: added RLVS 9. Section 11: added that calculations are performed by instrument software. 10. Section 11.8: added that over range results must be qualified. 11. Table 12.1: revised method blank corrective action. 12. Inserted new Method Modifications section.	27Sep2012
S-IN-I-040-rev.13	1. Table 9.1: revised details of Stannous Chloride reagent use and handling. 2. Section 14: added a modification for no continuous stirring of Stannous Chloride.	29Oct2012
S-IN-M-040-rev.14	1. Converted SOP to Corporate 27-section format. 2. Cover page: changed SOP name to reflect "M" for metals department, changed phone number, changed effective date format and changed document control format. 3. Section 9.2: added plunger filters. 4. Section 10.1: revised wash solution recipe. 5. Section 10: updated standard information and changed Intermediate #2 to Daily Spike. 6. Section 11: removed linear regression equation, made RLVS optional unless required by client or program and updated RLVS corrective action. 7. Section 12: changed Intermediate #2 to Daily Spike, updated procedure when permanganate color does not persist for 15 minutes, added optional use of plunger filters, and added requirement to document all information in the prep log.	15Dec2015
S-IN-M-040-rev.15	1. Section 5.1: updated default RL for solids. 2. Table 7.1: revised storage conditions for solids. 3. Section 9.1: updated instrument information. 4. Section 10.1: updated reagent information. 5. Tables 10.2 and 10.3: changed RLVS to CRDL. 6. Section 10.2.3.3: changed RLVS to CRDL. 7. Section 11.1: added requirement for calibration blank to be analyzed. 8. Sections 11.10 and 11.11: changed RLVS to CRDL. 9. Section 12.5.4: changed RLVS to CRDL. 10. Section 12.6: clarified that digestate can be diluted or sample can be re-digested at a dilution. 11. Table 13.1: updated LCS corrective action. 12. Section 14.3: updated units in equations and added x1000 to equation for solids. 13. Section 25: added years 2003 and 2009 to TNI reference.	21Jun2017

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF VOLATILE ORGANICS BY GC/MS
REFERENCE METHOD: EPA SW-846 METHODS 8260C, 5030A, 5030B AND 5035A

LOCAL SOP NUMBER:	S-IN-O-029-rev.19
EFFECTIVE DATE:	December 19, 2016
SUPERSEDES:	S-IN-O-029-rev.18

LOCAL APPROVAL

General Manager — December 8, 2016 Date

Quality Manager — December 2, 2016 Date

Department Manager — December 2, 2016 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. This Standard Operating Procedure (SOP) documents the procedures used by Pace Analytical Services – Indianapolis to determine the concentration of Volatile Organic Compounds (VOCs) in environmental samples. The laboratory utilizes purge-and-trap GC/MS and bases these documented procedures on those listed in SW-846 Method 8260C, 5030A, 5030B, and 5035A.

2. Summary of Method

2.1. Volatile organic compounds are introduced into the gas chromatograph by a purge-and trap method. The analytes are purged from a sample aliquot or extract using an inert gas. The purged analytes are collected on an absorbent trap. At the completion of the purge time, the trap is rapidly heated and back flushed to drive out the trapped analytes. The analytes are transferred into the inlet of a capillary gas chromatography column. The carrier gas flow through the column is controlled and the temperature is increased according to a set program to achieve optimum separation of purged analytes. The mass spectrometer is operated in a repetitive scan mode. Analytes are identified by the GC/MS retention times and by a comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected primary ion relative to an internal standard against a calibration curve.

3. Scope and Application

3.1. This method is applicable to most organic compounds that have boiling points below 200 ºC and are insoluble or slightly soluble in water. Volatile water-soluble compounds may also be determined although quantitation limits are typically higher due to their hydrophilic properties (e.g. ketones, oxygenates).

3.2. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable to most water and solid samples, regardless of moisture content. Matrices are groundwater, surface water, soil, sediment and waste. Procedures may need to be adapted to address limitations in the method or equipment that might hinder or interfere with sample analysis.

5. Limits of Detection and Quantitation

5.1. The list of target compounds and reporting limits is found in Table 1. Refer to the LIMS for method detection limits.

6. Interferences

6.1. Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the absorbent trap. Many common solvents, most notably acetone and methylene chloride, are frequently found in laboratory air at low levels. The use of polytetrafluoroethylene (PTFE, Teflon) as thread sealants, tubing, or in flow controllers is highly recommended since other materials can be sources of contamination which may concentrate in the trap during the purging.

6.2. A common source of interfering contamination is carryover. This may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive action to this condition is rinsing the purging apparatus and sample syringes with organic free water between samples. Analyze one or more blanks to check for contamination prior to sample analysis. If the sample immediately following the high concentration sample does not contain the compounds present in the high level sample, freedom from carryover contamination has been established.

6.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

6.4. Since methylene chloride and acetone are common laboratory solvents, special precautions must be taken. The volatiles analysis and sample storage area must be located as far as possible from areas where these solvents are used or stored. Where possible, the volatiles analysis and sample storage area should be served by a separate HVAC system and maintained under positive pressure to prevent intrusion of contaminants. Laboratory clothing previously exposed to methylene chloride fumes during extraction procedures can contribute to sample contamination.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
5030B Aqueous	Minimum (3) VOA vials Additional sample is required if MS/MSD is requested	Acidified w/ 1:1 HCl to pH<2, no headspace	Cool to ≤6°C	pH>2: Analysis must be completed within 7 days of collection date. pH <2: Analysis must be completed within 14 days of collection date. (pH determined post analysis)
5035A Solid Terra Core Kits (Preferred)	One (1) 2-4 oz. wide mouth jar for % moisture **AND** Two (2) 5-g portions in vials with magnetic stir bar and 5.0mL reagent water plus one (1) 5 g portion in a vial with 5.0mL methanol. Additional sample is required if MS/MSD is requested.	Either no preservative or Methanol as a preservative.	Cool to ≤6°C for no more than 48 hours from collection then freeze at -7°C to -20°C. Methanol vials may be stored at 0° to 6°C.	Analysis must be completed within 48 hours if samples are not frozen or preserved with methanol prior to the expiration of the 48 hour period. The holding time may be extended to 14 days if the sample is frozen or preserved with methanol prior to the expiration of the 48 hour period.
5035A Solid Coring Devices (Alternate)	One (1) 2-4 oz. wide mouth jar for % moisture **AND** Two (2) EnCore, TerraCore or similar sampling tubes. Additional sample is required if MS/MSD is requested.	No preservative Sample is extruded into a vial with a magnetic stir bar and 5.0mL reagent water.	Freeze at -7°C to -20°C within 48 hours of collection.	Analysis must be completed within 14 days of collection date.
5030A Solid Bulk Jars	One (1) 2-4 oz. wide mouth jar for % moisture **AND** One bulk sample jar, usually 4 oz. or 8oz.	No preservative Sample is weighed into a vial with a magnetic stir bar and 5.0mL reagent water.	Cool to ≤6°C	Analysis must be completed within 14 days of collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatographs	Agilent	Lab uses models 6850 and 6890	Or equivalent system
P&T Concentrators	EST Analytical, Tekmar, OI	Tekmar 3000 series, Encon, Encon Evolution, and 4660 Eclipse	Or equivalent system
Data Systems	Agilent	Chemstation	Or equivalent system
Autosamplers	EST Analytical, OI	EST 8100, Centurion, Centurion WS, 4551	Or equivalent system
Mass Spectrometers	Agilent	5973 and 5975	Or equivalent system

9.2. Chromatography Supplies

Item	Vendor	Model / ID	Description
Analytical Columns	Agilent	J&W Scientific DB-624	20m x 0.18mm x 1um or equivalent
Trap	Supelco	Trap K and OI #10	Or equivalent

9.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Syringe valves	2-way with Luer ends	Supelco or equivalent
Standard vials	stop/go vials, various sizes	Supelco or equivalent
Balance, Analytical/Top Load	Able to measure to nearest 0.001g/0.01g	Mettler, OHaus or equivalent
Sample vials	40mL vials; pre-cleaned	Eagle Picher or equivalent

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
Methanol	Purge-and trap grade or equivalent
Sand	Or equivalent material to be used as a simulated soil matrix.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.1 Standard Definitions

Standard	Description	Comments
Tune Standard	4-Bromofluorobenzene (BFB) solution used to verify ion response ratios prior to analysis	Must inject between 5 and 50ng
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains required spiking compounds, at a minimum, and is used to prepare MS/MSD sets.	Same solution can be used for the LCS, MS/MSD and CCV.

10.2.2. Storage Conditions

Table 10.2 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock VOA calibration standards	o2si; catalog #122961-01; 250-1250ug/mL and #121106-02; 250-5000ug/mL; and #121093-04; 1000-2000ug/mL; and #020249-03; 2500ug/mL, or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Stock Gas calibration standards	o2si; catalog #120016-06; 250ug/mL and catalog #020229-09, 10,000ug/mL and #121093-04, 500/2000ug/mL, or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Intermediate VOA calibration standard	Refer to Section 10.2.3.1.	Solution good for 1 month from preparation	Same as stock standard.
Intermediate Gas calibration standard	Refer to Section 10.2.3.2.	Solution good for 1 week from preparation	Same as stock standard
Working VOA calibration standards	Refer to Section 10.2.3.3.	One-time use	Not applicable
Stock VOA ICV/Spiking standards	o2si; catalog #121092-02-SS; 250-5000ug/mL and #121091-06-SS, 250-5000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Intermediate VOA ICV/Spiking standard	Refer to Section 10.2.3.4.	Solution good for 1 month from preparation	Same as stock standard
Working ICV/Spiking standard	Refer to Section 10.2.3.5.	One-time use	Not applicable
Stock VOATune/ Surrogate standard	Restek; catalog #30240, 2500ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Stock VOA Internal standards	Restek; catalog #30241, 2500ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Working Tune/Surrogate/Internal standard mix	Refer to Section 10.2.3.7.	Solution good for 1 month from preparation	Stored on autosampler under pressure in a 5mL vial.

10.2.3. Preparation Procedures

10.2.3.1. Intermediate VOA Calibration Standard Preparation (Example)

Dilute 1mL of o2si #122961-01 plus 1mL of o2si #121106-02 plus 100uL of o2si #020249-03 plus 1mL of o2si #121093-04 to 5.0mL with Methanol for a nominal concentration of 50mg/L.

10.2.3.2. Intermediate Gas Calibration Standard Preparation (Example)

Dilute 200uL of o2si #120016-06 plus 100uL of o2si #020229-09 plus 100uL of o2si #121093-04 to 1.0mL with Methanol for a nominal concentration of 50mg/L.

10.2.3.3. Working VOA Calibration Standards Preparation

Refer to Table 10.3 for examples of possible one-time use calibration standards.

Table 10.3 – Working Calibration Standards (examples only)

Standard	Int. VOA Cal. Standard amount	Int. Gas Cal. Standard amount	Final Total Volume	Final Concentration
Calibration Std 1	1uL	1uL	50mL	1ppb
Calibration Std 2	2uL	2uL	50mL	2ppb
Calibration Std 3	5uL	5uL	50mL	5ppb
Calibration Std 4	10uL	10uL	50mL	10ppb
Calibration Std 5	2uL	2uL	5mL	20ppb
Calibration Std 6 (CCV)	5uL	5uL	5mL	50ppb
Calibration Std 7	15uL	15uL	5mL	150ppb
Calibration Std 8	30uL	30uL	5mL	300ppb

10.2.3.4. Intermediate VOA ICV/Spiking Standard Preparation (Example)

Dilute 800uL of o2si #121092-02-SS plus 800uL of o2si #121091-06-SS to 4.0mL with Methanol for a final nominal concentration of 50mg/L.

10.2.3.5. Working ICV/Spiking Standard Preparation (Example)

Add 5uL of the Intermediate ICV standard per 5mL water for a final ICV concentration of 50ug/L.

10.2.3.6. Laboratory Control Sample (LCS) and Matrix Spike (MS/MSD) Preparation

10.2.3.6.1. Aqueous LCS: add 5uL of the Intermediate ICV/Spiking standard per 5mL reagent water for a nominal LCS concentration of 50ug/L.

10.2.3.6.2. Aqueous MS: add 5uL of the Intermediate ICV/Spiking standard per 5mL sample for a nominal MS concentration of 50ug/L.

10.2.3.6.3. Low-level Soil LCS: place 5 +/-0.5g of simulated soil matrix, 5mL reagent water and a stir bar into a vial. Add 5uL of the Intermediate ICV/Spiking standard for a nominal LCS concentration of 50ug/Kg.

10.2.3.6.4. Low-level Soil MS: place 5 +/-0.5g of sample, 5mL reagent water and a stir bar into a vial. Add 5uL of the Intermediate ICV/Spiking standard for a nominal MS concentration of 50ug/Kg.

10.2.3.6.5. Medium-level Soil LCS: place 4.8mL reagent water and 200uL methanol into a vial. Add 5uL of the Intermediate ICV/Spiking standard for an LCS concentration of 50ug/Kg.

10.2.3.6.6. Medium-level Soil MS: place a maximum of 200uL methanol sample extract into a vial and add enough reagent water to bring the final volume to 5mL. Add 5uL of the Intermediate ICV/Spiking standard for an LCS concentration of 50ug/Kg.

10.2.3.7. Working Tune/Surrogate/Internal Standard Preparation (Examples only, may vary)

Centurion/Centurion WS Autosamplers: Dilute 100uL of Restek #30240 plus 100uL of Restek #30241 to 5mL with Methanol for a final concentration of 50mg/L.

8100 Soil Autosamplers: Dilute 500uL of Restek #30240 plus 500uL of Restek #30241 to 5mL with Methanol for a final concentration of 250mg/L.

11. Calibration and Standardization

11.1. Tune Verification: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer must be hardware tuned by injecting 5-50ng BFB. This is done by analyzing a standard containing BFB. The BFB and calibration verification standard may be combined as long as both tuning and calibration verification acceptance criteria are met without interferences. Use the BFB mass intensity criteria in the table below as tuning acceptance criteria. Alternate tuning criteria may be used provided that method performance is not adversely affected.

Mass (m/z)	Ion Abundance criteria
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	<2% of m/z 174
174	>50% of m/z 95
175	5 to 9% of m/z 174
176	95 to 101% of m/z 174
177	5 to 9% of m/z 176

The mass spectrum of BFB may be obtained by averaging three scans, the peak apex scan and the scans immediately preceding and following the apex. Background subtraction is required using this approach and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak. Alternatively, the analyst may use other approaches suggested below:

1. A single scan within the BFB peak with background subtraction of a single scan no more than 20 scans prior to the elution of BFB.
2. An average of multiple scans within the BFB peak with background subtraction of a single scan no more than 20 scans prior to the elution of BFB.

If the ratios do not meet the criteria above, reanalyze the BFB tune. If the BFB still fails the criteria, instrument maintenance and/or preparation of new standards must be considered.

11.2. Initial Calibration: Initial Calibration standards are introduced into the GC/MS from the lowest to highest concentration of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate

linearity. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
A_{IS} = Area of the quantitation ion for the internal standard.
C_{IS} = Concentration of the internal standard
C_x = Concentration of the compound being measured.

11.3. The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

11.4. The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of average RF for a compound
RF_{avg} = Mean of RFs for a compound

11.5. The %RSD should be $\leq$20% for each target analyte.

11.6. For each calibration standard, all reported compounds that appear in Table 3 must meet the minimum response factor criteria shown.

11.7. If the percent relative standard deviation (%RSD) of the RFs for a compound is $\leq$20% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

11.7.1. If the % RSD for any compound is >20%, the analyst may employ a linear regression equation, non-weighted or weighted, that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.8. When calculating the calibration curve using the linear regression model, a minimum quantitation check on the viability of the calibration standard that corresponds to the reporting limit should be performed by re-fitting the response from the calibration standard that corresponds to the reporting limit back into the curve. The recalculated concentration of the reporting limit standard should be within +/-30% of the standard's true concentration. Reported compounds that fail this criteria must be qualified as estimated if the reported concentration is <2x the reporting limit. Alternatively, the reporting limit can be raised to the level of a calibration standard that meets the criteria when re-fitted against the curve.

11.9. Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be used and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of

contaminants below the response of the lowest calibration point may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be $\geq$0.99. Refer to Method 8000C for additional information regarding calibration.

11.10. If compounds fail to meet the criteria in Sections 11.5-11.9, the calibration fit must be set to average response factor and associated samples concentrations may be determined but they must be reported as estimated. In order to report non-detects, the compound must have been detected in the initial calibration standard that corresponds to the reporting limit.

11.11. Initial Calibration Corrective Action: If more than 10% of the compounds included with the initial calibration exceed the $\leq$20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternative curve fits, then the chromatographic system is considered too imprecise for analysis to begin. Instrument maintenance and/or preparation of new calibration standards must be considered prior to repeating the initial calibration procedure.

11.12. Each day that analysis is performed, the calibration standards and/or check standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably. Additionally, examination of signal-to-noise ratio of analytes in low-level standards may be a useful diagnostic tool to monitor instrument performance. Signal-to-noise ratio is defined as the ratio of the analyte signal to the noise measured on a blank. It is recommended but not required that the signal-to-noise ratio be at least three to confirm detection.

11.13. Initial Calibration Verification (ICV): The initial calibration curve should be verified immediately after performing the standard analysis using a second source standard (ICV) that is prepared using standards different from the calibration standards, with a concentration near the midpoint of the calibration range. The acceptance limits for the ICV are 70-130% recovery for all reported compounds, with the following exceptions:

Acetone	50-150%
Acrolein	50-150%
Bromomethane	50-150%
Iodomethane	50-150%
Methyl acetate	50-150%

11.14. ICV Corrective Action: If the ICV fails the criteria, another ICV may be analyzed. If the second ICV fails, a new initial calibration curve may be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Quantitative sample analysis should not proceed for those analytes that fail in the ICV or associated results must be qualified as estimated if analysis continues. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

11.15. Continuing Calibration Verification: The initial calibration is verified every 12 hours by analyzing a BFB tune that must meet the criteria in Section 10.1, followed by a Continuing Calibration Verification (CCV) standard. The CCV is normally prepared using the same standard solution used for the initial calibration but an ICV/LCS can be used as a CCV if it passes the required criteria for a CCV.

11.16. All target compounds in the CCV must be evaluated using a +/-20% variability criterion. Use percent difference when performing the average RF model calibration. Use percent drift when calibrating using a regression fit model.

$$\% \text{ Difference (\%D)} = \frac{\text{Calculated amount of standard} - \text{Expected amount of standard}}{\text{Expected amount of standard}} \times 100$$

$$\% \text{ Drift} = \frac{\text{Calculated concentration} - \text{Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

11.17. If the percent difference or percent drift for a compound is $\leq$20%, then the initial calibration for that compound is considered to be valid and sample analysis can continue. If the criterion is not met for more than 20% of the compounds included in the calibration, then corrective action must be taken prior to sample analysis.

In cases where compounds fail, they may still be reported as non-detects if the compound was detected in the initial calibration standard that corresponds to the reporting limit. For situations when the failed compound is present in samples, reported concentrations must be qualified as estimated values. Alternatively, the sample may be reanalyzed and reported for the compounds in question with a CCV that meets the criteria.

11.18. All reported compounds that appear in Table 3 must meet the minimum response factor criteria shown. If the minimum response factors are not met, the system should be evaluated and corrective action should be taken before sample analysis begins.

11.19. The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 10 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

11.20. CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. Alternatively, an ICV/LCS may be used as a CCV if it passes the CCV acceptance criteria. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. Exception: If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

12. Procedures

12.1. Configure the purge & trap system and GC/MS system per manufacturer's instructions. All samples must be analyzed at room temperature and the system must be calibrated and free of contamination before samples are analyzed.

12.2. Sample Preparation and Handling

12.2.1. Aqueous Samples

Water samples to be analyzed using the Centurion/OI autosampler require no sample preparation and are loaded as full 40mL VOA vials, unless they require a dilution. Refer to Section 7 for additional information regarding sample handling.

Water samples to be purged on the Archon/8100 autosampler are prepared by quickly measuring a 5mL aliquot of the sample using a 5mL gastight syringe and transferring it to a 40mL VOA vial.

This is done as quickly as possible to minimize analyte loss. The syringe is thoroughly rinsed inside and out with reagent water before measuring each sample.

Dilutions on aqueous samples must be prepared in a volumetric fashion. Sample aliquots are measured in either a volumetric pipet or gas-tight syringe and brought to volume in either a volumetric flask or gas-tight syringe.

After analysis, check the residue in the vial using pH paper. The pH should be <2 if HCl-preserved vials were used. Holding time for water samples with pH >2 is 7 days. Appropriately qualify on the sequence log and in LIMS any sample not meeting the pH requirement and/or holding time requirement. A stamp may be used to document on sequence logs that all water samples are pH<2 unless otherwise noted.

12.2.2. Soil Samples

12.2.2.1. Low-Level soils

Preferably, samples received for low level analysis should be contained in pre-weighed Terra Core vials with reagent water. Prior to analysis the sample weight must be determined and recorded by weighing the vial and recording the weight. Subtract the tare weight indicated on the vial and correct for the label weight to determine the sample weight. The sample is ready for analysis. Refer to Section 7 for additional information regarding sample handling.

Alternatively, samples received in coring devices, such as Encore, must be extruded into a pre-weighed VOA vial either with or without 5mL reagent water and a stir bar. Record the weight of the vial after the sample has been placed into it. Subtract the tare weight determined initially to determine the sample weight. The sample is ready for analysis.

Samples received in bulk soil jars are sub-sampled into a VOA vial. Place an empty VOA vial on the balance pan and tare the balance. Quickly add 5 +/-0.5g of sample to the vial. Record the sample weight. Add 5mL of reagent water and a stir bar and cap the vial. The sample is ready for analysis.

12.2.2.2. Medium-Level soils

Preferably, samples received for medium-level analysis should be received in pre-weighed Terra Core vials with methanol as a preservative. Prior to analysis the sample weight must be determined and recorded by weighing the vial and recording the weight. Subtract the tare weight indicated on the vial and correct for the label weight to determine the sample weight. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL reagent water is used for analysis. Refer to Section 7 for additional information regarding sample handling.

Alternatively, samples received in coring devices, such as Encore, must be extruded into a pre-weighed VOA vial with 5mL methanol. Record the weight of the vial after the sample has been placed into it. Subtract the tare weight determined initially to determine the sample weight. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL reagent water is used for analysis.

Samples received in bulk soil jars are sub-sampled into a VOA vial. Place an empty VOA vial on the balance pan and tare the balance. Quickly add 5 +/-0.5g of sample to the vial. Record the sample weight. Add 5mL methanol and cap the vial. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL reagent water is used for analysis.

12.3. Qualitative Analysis: Compounds are identified as present when the following criteria are met:

12.3.1. The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.

12.3.2. The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Refer to Table 2 for the characteristic ions.

12.3.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.4. Quantitative Analysis: Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique.

12.5. Refer to the Manual Integrations SOP for guidance on performing and documenting manual integrations.

12.6. If the sample concentration exceeds the linear range of the analysis, the sample must be diluted and reanalyzed or reported as an estimated concentration.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required. 3) If contaminant is present in the sample at a concentration >10x the method blank, sample may be reported with qualification.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	Reanalyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits RPD ≤20% Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Sample Duplicate (Dup)	Sample	One sample duplicate per batch of up to 20 samples if no MS/MSD.	RPD ≤20%	No corrective actions necessary. Qualify duplicate appropriately if RPD is out-of-control.
Surrogates	Applicable surrogate compounds	Added to each standard, sample, and method blank.	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.
As required by client only: **Internal Standards**	Applicable Internal Standard compounds	Added to each standard, sample, and method blank.	Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV.	Samples with internal standard failures must be reanalyzed at the same dilution or more concentrated. *Exception:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified.

13.2. Batch QC consisting of a Method Blank and LCS, at a minimum, is required for each batch of 20 or fewer samples for each matrix and preparation method combination (aqueous, low-level soil, and medium-level soil.).

13.3. Method Blank Preparation

13.3.1. Waters on Archon autosamplers: The Method Blank consists of a 40mL VOA vial containing 5mL reagent water.

13.3.2. Waters on Centurion autosamplers: The Method Blank consists of an HCl-preserved 40mL VOA vial filled completely with reagent water.

13.3.3. Low-level soils: The Method Blank consists of a 40mL VOA vial containing 5 +/-0.5g simulated soil matrix and 5mL reagent water and a stir bar.

13.3.4. Medium-level soils: The Method Blank consists of a 40mL VOA vial containing 4.8mL reagent water and 200uL methanol.

13.4. Laboratory Control Sample (LCS) and Matrix Spike (MS/MSD) Preparation: Refer to Section 10.2.3.6.

13.5. Allowable Marginal Exceedances: If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. A marginal exceedance (ME) is defined as being beyond the LCS control limit of +/-3 standard deviations, but within the ME limits of +/-4 standard deviations around the mean. The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and correction action is necessary. If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken.

The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

NOTE: As allowed by client or program, the LCS may be outside the control limits but ≥10% recovery for up to four additional volatile compounds with the exception of benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, total xylenes and any requested oxygenate without corrective action.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = (X_s)(D) \qquad \text{Solid Sample (ug/kg)} = \frac{(X_s)(V_f)(D)}{(W_s)}$$

Where: X_s = On-column concentration of the analyte, ug/L
V_f = Final volume, L
D = Dilution factor
W_s = Weight of solid sample, kg

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100- \%Moisture)}} \times 100$$

14.2. LCS equation:

$$\mathbf{R = (C/S) * 100}$$

Where: R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where: R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where: RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control of Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. An MDL and/or LOD/LOQ verification study must be conducted annually for each matrix per instrument.

18.2. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. GC columns and chromatographic conditions may differ from those recommended.

19.2. Calibration solutions are purchased as certified standards.

20. Instrument/Equipment Maintenance

20.1. Refer to maintenance log and/or instrument manufacturer's instructions.

21. Troubleshooting

21.1. Refer to maintenance log and/or instrument manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed Waste Handling, or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

24. Pollution Prevention

24.1. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24.2. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000C, 8260C, 5030A, 5030B, and 5035A.

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Table 1: Method 8260C Target Compounds and Reporting Limits
26.2. Table 2: Characteristic Ions and Internal Standard Association of Target Compounds
26.3. Table 3: Minimum Response Factor Criteria

27. Revisions

Document Number	Reason for Change	Date
S-IN-O-029-rev.17	1. Section 3.1: added reference to MDLs 2. Section 9.1: added simulated soil matrix. 3. Section 9.2.3.6: added detail for preparation of LCS and MS to include simulated soil matrix. 4. Table 12.1: revised method blank corrective action. 5. Section 12.2.3: revised low-level soil method blank prep to include simulated soil matrix. 6. Inserted new Method Modifications section.	24Sep2012
S-IN-O-029-rev.18	1. Cover: changed 8260B to 8260C and added actual effective date. 2. Section 1.1: changed 8260B to 8260C 3. Section 4: revised to reflect 8260C 4. Table 8.3: revised balance specifications to match practice 5. Table 9.2: revised for standard mixes currently in use. 6. Section 9.2.3: revised recipes for standard mixes currently in use, changed approx. 5g to 5 +/- 0.5g, added batch QC details for medium-level soils, and detailed surrogate prep for both types of autosampler. 7. Section 10.1: added BFB acquisition guidance. 8. Section 10.13: added as guidance for evaluation of ICAL standards. 9. 10.5 - 10.21: revised to comply with Method 8260C, clarified use of LCS as CCV, and clarified requirement to correct vial TC vial weight for the label weight. 10. Section 11.2.2: added that tared soil vials need to be corrected for label weight and changed approx. 5g to 5 +/- 0.5g. 11. Section 11.5: added as a reference to the Manual Integrations SOP. 12. Section 11.6: added to require over range samples be diluted and reanalyzed or qualified as estimated. 13. Section 12.2: added to clarify the requirement for batch QC. 14. Section 12.3.2: clarified that blank is to be prepared using an HCl preserved vial. 15. Section 12.3.3: changed approx. 5g to 5 +/- 0.5g and added stir bar. 16. Section 13.1: added optional LOD/OQ verification. 17. Section 16.1: removed reference to 8000B and 8260A and changed 8260B to 8260C. 18. Section 17: changed 8260B to 8260C and added Table 3 attachment	31Oct2013

S-IN-O-029-rev.19	1. Converted to Corporate 27-section format. 2. Cover page: changed phone number and revised document control format. 3. Table 7.1: updated temperature format and preservation for 5035A. 4. Section 9.1: updated to include OI instrumentation. 5. Section 9.2: updated to include OI instrumentation. 6. Table 10.2: updated to current standards in use. 7. Section 10.2.3: updated to current standard preparation procedures. 8. Section 11: removed equations for different curve fits. 9. Section 11.7: added option of weighted linear. 10. Section 11.10: added requirement to set failing compounds to average fit. 11. Section 12.2.1: added OI and indicated that sample pH should be <2 if HCl vials were used. 12. Table 13.1: added an exception to MB when sample concentration is>10x MB concentration. Added sample duplicate to table and added RPD criteria. 13. Section 14: equations for water and solid final concentration fixed. 14. Table 1: added compounds for consistency between tables. 15. Table 2: added internal standard association and updated 1,2,3-TCP ions. 16. Table 3: revised minimum RF for TCE and PCE.	23Nov2016

Table 1: Method 8260C Target Compounds and Reporting Limits[1]

Analyte	RL water (ug/L)	RL soil Low-level (ug/kg)	RL soil Medium-level (ug/kg)
Dichlorodifluoromethane	5	5	125
Chloromethane	5	5	125
Vinyl Chloride	2	5	125
Bromomethane	5	5	125
Chloroethane	5	5	125
Trichlorofluoromethane	5	5	125
Acrolein	50	100	2500
1,1,2-Trichlorotrifluoroethane	5	5	125
1,1-Dichloroethene	5	5	125
Acetone	100	100	2500
Iodomethane	10	100	2500
Carbon Disulfide	10	10	250
Methylene Chloride	5	20	500
Acrylonitrile	100	100	2500
Methyl tert-butyl ether	4	5	125
trans-1,2-Dichloroethene	5	5	125
Vinyl Acetate	10	100	2500
1,1-Dichloroethane	5	5	125
2-Butanone (MEK)	25	25	625
cis-1,2-Dichloroethene	5	5	125
2,2-Dichloropropane	5	5	125
Bromochloromethane	5	5	125
Chloroform	5	5	125
Cyclohexane	100	100	2500
1,1,1-Trichloroethane	5	5	125
Carbon Tetrachloride	5	5	125
1,1-Dichloropropene	5	5	125
Benzene	5	5	125
1,2-Dichloroethane	5	5	125
Trichloroethene	5	5	125
Methylcyclohexane	50	50	1250
1,2-Dichloropropane	5	5	125
Dibromomethane	5	5	125
Bromodichloromethane	5	5	125
cis-1,3-Dichloropropene	5	5	125
4-Methyl-2-pentanone (MIBK)	25	25	625
Toluene	5	5	125
trans-1,3-Dichloropropene	5	5	125
Ethyl Methacrylate	100	100	2500
1,1,2-Trichloroethane	5	5	125
Tetrachloroethene	5	5	125
1,3-Dichloropropane	5	5	125
2-Hexanone	25	100	2500
Dibromochloromethane (Chlorodibromomethane)	5	5	125
1,2-Dibromoethane (EDB)	5	5	125
Chlorobenzene	5	5	125
1,1,1,2-Tetrachloroethane	5	5	125
Ethylbenzene	5	5	125
m&p-Xylene	5	5	125
o-Xylene	5	5	125

Analyte	RL water (ug/L)	RL soil Low-level (ug/kg)	RL soil Medium-level (ug/kg)
Styrene	5	5	125
Isopropylbenzene	5	5	125
Bromobenzene	5	5	125
trans-1,4-Dichloro-2-butene	100	100	2500
Bromoform	5	5	125
1,1,2,2-Tetrachloroethane	5	5	125
1,2,3-Trichloropropane	5	5	125
n-Propylbenzene	5	5	125
2-Chlorotoluene	5	5	125
1,3,5-Trimethylbenzene	5	5	125
4-Chlorotoluene	5	5	125
tert-Butylbenzene	5	5	125
1,2,4-Trimethylbenzene	5	5	125
sec-Butylbenzene	5	5	125
1,3-Dichlorobenzene	5	5	125
p-Isopropyltoluene	5	5	125
1,4-Dichlorobenzene	5	5	125
n-Butylbenzene	5	5	125
1,2-Dichlorobenzene	5	5	125
1,2-Dibromo-3-chloropropane	10	10	250
1,2,4-Trichlorobenzene	5	5	125
Hexachlorobutadiene	5	5	125
Naphthalene	5	5	125
1,2,3-Trichlorobenzene	5	5	125
2-Methylnaphthalene	10	10	250

[1]Target Compounds and Reporting Limits are subject to change.

Table 2: Characteristic Ions and Internal Standard Association of Target Compounds[2]

Analyte	Primary Ion	Secondary Ion(s)
Group 1 – Fluorobenzene (IS)	**96**	**-**
Dichlorodifluoromethane	85	87
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
Trichlorofluoromethane	101	103
Acrolein	56	55
1,1,2-Trichlorotrifluoroethane	101	151
1,1-Dichloroethene	96	61, 63
Acetone	43	58
Iodomethane	142	127
Carbon Disulfide	76	78
Methylene Chloride	84	86, 49
Acrylonitrile	53	52, 51
Methyl tert-butyl ether	73	57
trans-1,2-Dichloroethene	96	61, 98
Vinyl Acetate	43	86
1,1-Dichloroethane	63	65, 83
2-Butanone (MEK)	43	57, 72
cis-1,2-Dichloroethene	96	61, 98
2,2-Dichloropropane	77	97
Bromochloromethane	49	128
Chloroform	83	85
Dibromofluoromethane (Surr)	113	111
Cyclohexane	56	84, 41
1,1,1-Trichloroethane	97	99, 61
Carbon Tetrachloride	117	119, 121
1,1-Dichloropropene	75	110, 77
Benzene	78	52, 77
1,2-Dichloroethane	62	98, 64
Trichloroethene	95	97, 130, 132
Methylcyclohexane	55	69, 83
1,2-Dichloropropane	63	62, 112
Dibromomethane	93	95, 174
Bromodichloromethane	83	85, 127
Group 2 – Chlorobenzene-d5 (IS)	**117**	**82, 119**
cis-1,3-Dichloropropene	75	77
4-Methyl-2-pentanone (MIBK)	43	58, 85
Toluene-d8	98	99, 100
Toluene	91	92
trans-1,3-Dichloropropene	75	77
Ethyl Methacrylate	69	99, 114
1,1,2-Trichloroethane	83	97, 85
Tetrachloroethene	166	129, 168
1,3-Dichloropropane	76	78
2-Hexanone	43	58, 100
Dibromochloromethane (Chlorodibromomethane)	129	127
1,2-Dibromoethane (EDB)	107	109
Chlorobenzene	112	77, 114

Analyte	Primary Ion	Secondary Ion(s)
Group 2 – Chlorobenzene-d5 (IS) Continued	**117**	**82, 119**
1,1,1,2-Tetrachloroethane	131	133, 119
Ethylbenzene	106	91
m&p-Xylene	106	91
o-Xylene	106	91
Styrene	104	78
Isopropylbenzene	105	120
4-Bromofluorobenzene (Surr)	95	174, 176
Bromobenzene	77	156, 158
trans-1,4-Dichloro-2-butene	53	88, 75
Group 3 – 1,4-Dichlorobenzene-d4 (IS)	**152**	**115, 150**
Bromoform	173	175, 254
1,1,2,2-Tetrachloroethane	83	131, 85
1,2,3-Trichloropropane	110	75, 77
n-Propylbenzene	91	120
2-Chlorotoluene	91	126
1,3,5-Trimethylbenzene	105	120
4-Chlorotoluene	126	91
tert-Butylbenzene	119	91, 134
1,2,4-Trimethylbenzene	105	120
sec-Butylbenzene	105	134
1,3-Dichlorobenzene	146	111, 148
p-Isopropyltoluene	119	134, 91
1,4-Dichlorobenzene	146	111, 148
n-Butylbenzene	91	92, 134
1,2-Dichlorobenzene	146	111, 148
1,2-Dibromo-3-chloropropane	155	75, 157
1,2,4-Trichlorobenzene	180	182, 145
Hexachlorobutadiene	225	223, 227
Naphthalene	128	127
1,2,3-Trichlorobenzene	180	182, 145
2-Methylnaphthalene	142	141, 115

[2]Subject to change.

Table 3: Minimum Response Factor Criteria

Analyte	Minimum Response Factor (RF)
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
Methylene Chloride	0.100
1,1-Dichloroethene	0.100
trans-1,2-Dichloroethene	0.100
1,1-Dichloroethane	0.200
cis-1,2-Dichloroethene	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon Tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
*Trichloroethene	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
Toluene	0.400
1,1,2-Trichloroethane	0.100
*Tetrachloroethene	0.100
Dibromochloromethane (Chlorodibromomethane)	0.100
1,2-Dibromoethane (EDB)	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
m&p-Xylene	0.100
o-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2,4-Trichlorobenzene	0.200
trans-1,3-Dichloropropene	0.100
cis-1,3-Dichloropropene	0.200
*Acetone	0.010
*2-Butanone (MEK)	0.010
*4-Methyl-2-pentanone (MIBK)	0.050
*2-Hexanone	0.050
Methyl tert-butyl ether	0.100
Carbon Disulfide	0.100
1,2-Dibromo-3-chloropropane	0.050
Methylcyclohexane	0.100
1,1,2-Trichlorotrifluoroethane	0.100
Methyl acetate	0.100

*Alternate minimum RF criteria based on compound performance.

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STANDARD OPERATING PROCEDURE

DETERMINATION OF DIESEL RANGE ORGANICS BY GC/FID
REFERENCE METHOD: EPA SW-846 METHOD 8015D

SOP NUMBER:	S-IN-O-020-rev.16
EFFECTIVE DATE:	August 14, 2017
SUPERSEDES:	S-IN-O-020-rev.15

APPROVAL

General Manager	August 7, 2017 Date
Quality Manager	August 7, 2017 Date
Department Manager	August 7, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
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1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of diesel range organics in aqueous and solid samples while meeting the requirements specified in EPA method 8015D and various state petroleum hydrocarbon methods.

2. Summary of Method

2.1. Extracts from an appropriate solvent extraction method are introduced into the gas chromatograph (GC) by direct injection and are detected by a flame-ionization detector (FID). The non-halogenated and semi-volatile compounds are separated using a capillary GC column. See other SOPs for sample preparation techniques for aqueous and solid samples.

3. Scope and Application

3.1. Method 8015D defines diesel range organics (DRO) as the range of hydrocarbons from C_{10} to C_{28} covering a boiling range of about 170 ^{o}C -430^{o}C. Other alkane ranges, such as C_8 to C_{28}, C_{10} to C_{20}, or as determined by client request or regulatory requirement may also be reported.

3.2. Reporting limits, control limits, alkane ranges, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC systems and interpretation of DRO data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable to most groundwater, surface water, wastewater, soil, sediment and waste samples.

5. Limits of Detection and Quantitations

5.1. The reporting limits ranges are: 0.1-2 mg/L for aqueous samples and 10-20 mg/kg for solid samples, depending on the range and procedure requested. Refer to the LIMS for method detection limits.

6. Interferences

6.1. Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory blanks. The use of high purity reagents and solvents helps to minimize interference problems.

6.2. Extraction glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by detergent washing with hot water, and rinses with tap water and distilled water.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid, preferably 1L or 125mL widemouth, or equivalent.	None required	Cool to $\leq 6^{o}C$	Must be extracted within 7 days of collection date and analyzed within 40 days of extraction date.
Solid	200 grams in 4oz glass jar, or equivalent	None required	Cool to $\leq 6^{o}C$	Must be extracted within 14 days of collection date and analyzed within 40 days of extraction date.
Non-aqueous waste	200 grams in 4oz glass jar, or equivalent	None required	Cool to $\leq 6^{o}C$	Must be extracted within 14 days of collection date and analyzed within 40 days of extraction date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Vendor	Description/Comments
Gas Chromatograph	Agilent	7890A FID or equivalent
Autosampler	Agilent	HP7693 or equivalent
Data System	Hewlett Packard	Chemstation or equivalent
Mach unit (fast GC)		GD-C1017, 10m x 0.32mm; DB-5 column or equivalent

9.2. General Supplies

Item	Vendor	Description
GC Column	Agilent	DB-5, 10m x 32mm x 0.50um or equivalent
Micro syringes	Hamilton or equivalent	Various sizes
Autosampler Vials		2mL with crimp-top caps or equivalent

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Methylene Chloride	JT Baker Ultra Resi-Analyzed 9266-8P, or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains all target analytes and may or may not be prepared from the same standards as the calibration standards.	Same solution can be used for the LCS and MS/MSD

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock DRO Calibration standard	Restek Diesel Fuel #2; catalog # 31259; 50,000ug/mL in methylene chloride or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock DRO Surrogate standard	Restek pentacosane; catalog # 31487; 10,000ug/mL in methylene chloride or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working DRO Calibration standards	Refer to Section 9.2.3.1.	Good for 6 months from preparation	Refrigerate
Stock DRO ICV standard	Ultra Diesel Fuel #2; catalog # RGO-616; 50,000ug/mL in methylene chloride or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working DRO ICV standard	Refer to Section 9.2.3.2.	Good for 6 months from preparation	Refrigerate
Stock DRO Marker standard	Restek Florida TRPH; catalog #31266; 500ug/mL in hexane or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working DRO Marker standard	Refer to Section 9.2.3.3.	Good for 6 months from preparation	Refrigerate

10.2.3. Standard Preparation Procedures

10.2.3.1. Working DRO Calibration Standards

Working DRO Calibration Standards are prepared from the Stock DRO Calibration Standard (50,000ug/mL) and the Stock DRO Surrogate Standard (10,000ug/mL) to a final volume of 1mL in methylene chloride. Table 9.4 represents examples of possible calibration concentrations but actual concentrations may vary.

Table 10.4 – Working Calibration Standards

Standard	Stock DRO Standard amount	Stock Surrogate Standard amount	Final Total Volume	Final DRO Concentration	Final Surrogate Concentration
Calibration Std 1	1.5uL	1.5uL	1mL	75ug/mL	15ug/mL
Calibration Std 2	2uL	2uL	1mL	100ug/mL	20ug/mL
Calibration Std 3	6uL	6uL	1mL	300ug/mL	60ug/mL
Calibration Std 4	10uL	10uL	1mL	500ug/mL	100ug/mL
Calibration Std 5	20uL	20uL	1mL	1000ug/mL	200ug/mL
Calibration Std 6 (CCV)	50uL	50uL	1mL	2500ug/mL	500ug/mL
Calibration Std 7	100uL	100uL	1mL	5000ug/mL	1000ug/mL
Calibration Std 8	150uL	150uL	1mL	7500ug/mL	1500ug/mL
Calibration Std 9	200uL	200uL	1mL	10,000ug/mL	2000ug/mL

10.2.3.2. Working DRO ICV Standard

Dilute 50uL of the Stock DRO ICV Standard (10,000ug/mL) and 50uL of the Stock DRO Surrogate Standard (10,000ug/mL) to 1mL with methylene chloride for a final DRO concentration of 2500ug/mL and a final surrogate concentration of 500ug/mL.

10.2.3.3. Working DRO Marker Standards

Dilute 200uL of the Stock DRO Marker Standard (500ug/mL) to 1mL with methylene chloride for a final concentration of 100ug/mL.

11. Calibration and Standardization

11.1. Initial Calibration

11.1.1. The retention time range for DRO is defined during initial calibration. Inject 1uL of the stock/working marker standard listed in Table 9.4 to establish retention time windows for the beginning and end of all carbon ranges. The marker standard is analyzed prior to initial calibration standards. The retention time range is then calculated based on the lower limit of the RT window for the first eluting component and the upper limit of the RT window for the last eluting component. The total area within the RT range is used for quantitation of sample concentrations.

11.1.2. For initial calibration, analyze a minimum of five concentrations of calibration standard. The lowest calibration standard must be at or below the required reporting limit. Refer to the Quality Manual for more information regarding calibration curves. Determine the calibration factor (CF) of each standard using the calculation below. Determine the relative standard deviation (RSD) of the calibration factors using the calculation below. If the RSD is $\leq$20% over the working range, linearity through the origin is assumed, and the average calibration factor can be used in place of a calibration curve to calculate sample concentrations.

CF = Peak Area/ug of Standard RSD = (Std. Dev./CF) x 100

11.1.3. If the RSD is >20% over the working range, then linearity through the origin cannot be assumed. A regression equation that does not pass through the origin can be employed. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.1.4. Initial Calibration Corrective Action: If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.1.5. Each day that analysis is performed, the calibration standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably. Additionally, examination of signal-to-noise ratio of analytes in low-level standards may be a useful diagnostic tool to monitor instrument performance. Signal-to-noise ratio is defined as the ratio of the analyte signal to the noise measured on a blank. It is recommended but not required that the signal-to-noise ratio be at least three to confirm detection.

11.1.6. Initial Calibration Verification (ICV): The initial calibration must be verified through the analysis of an Initial Calibration Verification (ICV) standard. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The ICV is analyzed immediately following the initial calibration curve. The ICV recovery is evaluated against a default acceptance range of 70-130% recovery using the calculation below.

$$\% \text{ Recovery} = \frac{\text{Observed concentration}}{\text{Theoretical concentration}} \times 100$$

11.1.7. ICV Corrective Action: If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee. **Exception:** If the ICV exceeds the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.1.8. Retention Time Windows: The retention time range for DROs is defined during initial calibration. The range is established from the retention times of the C_8 or C_{10} and C_{28} alkanes. To establish retention time windows, make three or more injections of a retention time standard over the course of a 72-hour period, at a minimum. Record the retention time in minutes for the C_8 or C_{10} and C_{28} peaks and surrogate to three decimal places. Calculate the mean and standard deviation of the absolute retention times of the peaks. The retention time window is defined as +/-3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.

Establish the center of the retention time window daily by using the absolute retention time from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

11.2. Daily and Continuing Calibration

11.2.1. The working calibration curve and retention times must be verified at the beginning of each analytical sequence or every 12-hours as a minimum requirement. Daily calibration begins with the analysis of the marker standard and a Continuing Calibration Verification Standard (CCV). The CCV is analyzed daily following the marker standard and every 12 hours thereafter, at a minimum.

11.2.2. For each CCV, if the response or calculated concentration is within +/-20% Difference of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF values from the initial calibration to calculate sample results.

$$\% \text{ Difference} = [CF - CF_{avg}] / CF_{avg} * 100$$

11.2.3. **CCV Corrective Action*:*** If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

12. Procedures

12.1. Configure the GC per manufacturer's instructions and inject standards and extracts for analysis.

12.2. The total area within the RT range is the sample response. Sample concentrations are calculated by comparing sample response data with the initial calibration. Results are based on retention time windows for specific carbon ranges quantitated against a diesel standard. Pattern matching for quantitative purposes is not performed on a routine basis. Refer to the Manual Integration SOP for manual integration guidelines.

12.3. If sample response exceeds the limits of the initial calibration range, a dilution of the sample must be analyzed or the result must be reported as estimated.

12.4. It may be necessary to analyze a solvent blank after a highly concentrated sample to eliminate carryover.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or simulated soil matrix.	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogate	Applicable surrogate compound	Added to each sample, standard and method blank	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_f)(D)}{(V_s)} \qquad \text{Solid Sample (mg/kg)} = \frac{(X_s)(V_f)(D)}{(W_s)}$$

Where: X_s = On-column concentration of the analyte in mg/L
V_f = Final volume of extract in Liters
D = Dilution factor of extract
V_s = Volume of aqueous sample extracted in Liters
W_s = Weight of solid sample purged or extracted in kilograms

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$$

14.2. LCS equation:

$$\mathbf{R = (C/S) * 100}$$

Where R = percent recovery
C = observed LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = observed spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. An MDL study and/or LOD/LOQ verification must be conducted every 12 months for each matrix per instrument.

18.2. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Instrument conditions and setup may differ from those described in the method due to improvements in columns and instrumentation since the method was written.

19.2. Fuel standards are purchased as certified materials and not prepared from neat due to the need for consistency in the quality of stock standards.

19.3. Ranges other than C_{10}-C_{28} are analyzed due to specific client or program requirements.

19.4. Subtraction of column bleed area is not performed due to improvements in columns and instrumentation since the method was written.

20. Instrument/Equipment Maintenance

20.1. Refer to maintenance log and/or instrument manufacturer's instructions.

21. Troubleshooting

21.1. Refer to maintenance log and/or instrument manufacturer's instructions.

22. Safety

22.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions must be prepared in a hood whenever possible.

22.2. Samples

Take precautions when handling samples. Samples must always be treated as potentially hazardous "unknowns". Gloves**,** lab coats and safety glasses are required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

22.3. Equipment

Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment must be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention.

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, Methods 8015D and 8000C.

25.2. Pace Manual Integration SOP, current revision.

25.3. Pace Analytical Quality Manual; latest revision.

25.4. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable

27. Revisions

Document Number	Reason for Change	Date
S-IN-O-020-rev.14	1. Cover page: added actual effective date. 2. Tables 8.1 and 8.2: updated to reflect current supplies used. 3. Table 9.3: updated for current standards used. 4. Section 10.1.3: removed equation and made reference to 8000C. 5. Section 10.1.5: added as guidance for evaluation of low ICAL points 6. Section 10.1.8: added retention time window guidance. 7. Section 11.5 and 11.6: converted equations back to SW-846 equations 8. Section 13.1: added LOD/LOQ verification as option to annual MDL requirement. 9. Section 16.1: updated reference to 8000C	01Nov2013
S-IN-O-020-rev.15	1. Cover page: changed method reference to 8015D and changed phone number of lab. 2. Section 1.1: changed method reference to 8015D. 3. Section 3.1: changed method reference to 8015D and added TPH range C10-C20. 4. Table 7.1: added non-aqueous waste as a matrix. 5. Table 9.1: updated methylene chloride currently in use. 6. Table 9.3: updated storage of working solutions to refrigerate. 7. Table 9.4: removed Calibration Standard 10. 8. Section 10.1.1: updated last sentence to indicate that sample quantitation is based on the total area in the RT range – not the RT window. 9. Section 10.2.2: changed CCV acceptance criteria to +/-20%. 10. Section 11: removed calculations for average and linear curve fits. 11. Section 11.1: removed 1uL injection volume. 12. Section 11.2: updated first sentence to indicate that the sample response is the total area within the RT range – not the RT window. 13. Table 12.1: updated method blank components for soil to “simulated soil matrix.” 14. Section 14: added reasoning to explain some method modifications. 15. Section 16.1: changed method reference to 8015D.	10Aug2015
S-IN-O-020-rev.16	1. Converted to 27 section format. 2. Table 7.1: updated storage temperature format. 3. Section 14.1: updated calculations to be in like unit. 4. Section 25.4: added years 2003 and 2009 to TNI reference.	03Aug2017

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STANDARD OPERATING PROCEDURE

DETERMINATION OF GASOLINE RANGE ORGANICS BY GC/FID
REFERENCE METHOD: EPA SW-846 METHOD 8015D

SOP NUMBER:	S-IN-O-109-rev.13
EFFECTIVE DATE:	September 25, 2017
SUPERSEDES:	S-IN-O-109-rev.12

APPROVAL

General Manager — September 18, 2017 Date

Quality Manager — September 13, 2017 Date

Department Manager — September 14, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
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Table of Contents

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory procedure for determining the concentration of Gasoline Range Organics (GRO) in aqueous and solid environmental samples while meeting the requirements specified in SW-846 Method 8015D in conjunction with 5030A, 5030B and 5035A.

2. Summary of Method

2.1. The volatile organic compounds are introduced into the gas chromatograph by a purge-and trap method. The analytes are purged from a sample aliquot or extract by purging with an inert gas. The purged analytes are trapped in a sorbent tube. At the completion of the purge time, the sorbent tube is rapidly heated and back flushed to desorb trapped analytes directly into the inlet of a capillary gas chromatography column. The carrier gas flow through the column is controlled and the temperature is increased according to a set program to achieve optimum elution of GRO.

2.2. GRO concentrations are quantified by comparison of the total FID response for all peaks within the defined elution range with a calibration curve constructed from the responses of authentic gasoline standards.

3. Scope and Application

3.1. This method is applicable to gasoline components and other purgeable organics that elute within the same range. Reporting of GRO, for the state of Indiana, corresponds to an alkane range of C_5-C_{12}. Alkane ranges of C_6-C_{10} and C_6-C_{12} are frequently reported. Other alkane ranges may be reported.

3.2. Reporting limits, control limits, alkane ranges, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC systems and interpretation of GRO data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable to most groundwater, surface water, soil and sediment samples, regardless of moisture content.

5. Limits of Detection and Quantitation

5.1. The default reporting limits for GRO are: 0.2 mg/L for aqueous samples and 1mg/kg for solid samples. Refer to the LIMS for method detection limits.

6. Interferences

6.1. Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the absorbent trap. The use of polytetrafluoroethylene as thread sealants, tubing, or in flow controllers is highly recommended since other materials can be sources of contamination which may concentrate in the trap during the purging.

6.2. A common source of interfering contamination is carryover. This may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive action for this condition is rinsing the purging apparatus and sample syringes with organic free water between samples. Analyze blanks to check for cross contamination prior to sample analysis.

6.3. Samples containing high concentrations of heavier petroleum products such as diesel fuel may contain volatile components that respond within the GRO elution range. Also, any purgeable organic that elutes within the GRO range and produces an FID response will be quantitated.

6.4. The volatiles analysis and sample storage area should be located as far as possible from areas where solvents are used or stored. Where possible, the volatiles analysis and sample storage area should be served by a separate HVAC system and maintained under positive pressure to prevent intrusion of contaminants. Laboratory clothing previously exposed to methylene chloride fumes during extraction procedures can contribute to sample contamination.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
5030B Aqueous	Minimum (3) VOA vials Additional sample is required if MS/MSD is requested	Acidified with HCl to pH<2, no headspace	Cool to ≤6°C	pH>2: Analysis must be completed within 7 days of collection date. pH <2: Analysis must be completed within 14 days of collection date. (pH may be determined post analysis)
5035A Solid Terra Core Kits (Preferred)	One (1) 2-4 oz. wide mouth jar for % moisture **AND** Two (2) 5-g portions in vials with magnetic stir bar and 5.0mL reagent water plus one (1) 5 g portion in a vial with 5.0mL methanol. Additional sample is required if MS/MSD is requested.	No preservative or Methanol	Cool to ≤6°C for no more than 48 hours from collection then freeze at -7°C to -20°C. Methanol vials may be stored cool to ≤6°C	Analysis must be completed within 48 hours if samples are not frozen or preserved with methanol prior to the expiration of the 48 hour period. The holding time may be extended to 14 days if the sample is frozen or preserved with methanol prior to the expiration of the 48 hour period.
5035A Solid Coring Devices (Alternate)	One (1) 2-4 oz. wide mouth jar for % moisture **AND** Two (2) EnCore, TerraCore or similar sampling tubes. Additional sample is required if MS/MSD is requested.	No preservative Sample is extruded into a vial with a magnetic stir bar and 5.0mL reagent water.	Freeze at -7°C to -20°C within 48 hours of collection.	Analysis must be completed within 14 days of collection date.
5030A Solid Bulk Jars	One (1) 2-4 oz. wide mouth jar for % moisture **AND** One bulk sample jar, usually 4 oz. or 8oz.	No preservative Sample is weighed into a vial with a magnetic stir bar and 5.0mL reagent water or 5.0mL methanol.	Cool to ≤6°C	Analysis must be completed within 14 days of collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatographs	Agilent	Models 5890 and 6890	Or equivalent equipment
P&T Concentrators	Tekmar/ EST Analytical	LSC 3000/ Encon	Or equivalent equipment
Data Systems	Hewlett Packard/Target		Or equivalent data system
Autosamplers	EST Analytical	EST 8100	Or equivalent equipment

9.2. Chromatography Supplies

Item	Vendor	Model / ID	Description
Analytical Columns	Fisher	J&W Scientific DB-624	20m x 0.18mm x 1 um, or equivalent column
Trap	Supelco	Trap-K	Or equivalent

9.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Syringe valves	2-way with Luer ends	Supelco or equivalent
Standard vials	2mL stop/go vials	Supelco or equivalent
Balance, Analytical/Top Load	Able to measure to nearest 0.001g/0.01g	Mettler, OHaus or equivalent
Vortex mixer		Fisher or equivalent
Sample vials	40mL vials; pre-cleaned	Eagle Picher or equivalent

10. Reagents and Standards

10.1. Reagents

Reagent	Requirements/ Vendor/ Item #
Reagent water	ASTM Type II water
Methanol	JT Baker P&T Grade/MG Scientific catalog # 9077-02 or equivalent
Sand	Or equivalent material to be used as a clean simulated soil matrix.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.1 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains the target analyte and is used to spike MS/MSD sets.	Same solution can be used for both the LCS and MS/MSD.

10.2.2. Storage Conditions

Table 10.2 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock GRO calibration standard	Restek; catalog # 30081; 2500ug/mL or equivalent.	Manufacturer's recommended expiration. Once opened, standard is good for one month.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working GRO calibration standards	Refer to Section 10.2.3.1	One-time use standard	Not applicable
Stock GRO ICV standard	O2si; catalog #020942-01, 5000ug/mL or equivalent	Manufacturer's recommended expiration date or 1 month from opening of ampule.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working GRO ICV standard	Refer to Section 10.2.3.2	One-time use standard.	Not applicable
Stock/Working Surrogate standard	O2si; catalog #020135-18, 5000ug/mL or equivalent	Manufacturer's recommended expiration date. Once opened, standard is good for one month.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock Retention Time Marker standard	O2Si; catalog #122421-01, 12,500ug/mL or equivalent	Manufacturer's recommended expiration date. Once opened, standard is good for one month.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working Retention Time Marker standard	Refer to Section 10.2.3.3	One-time use standard	Not applicable

10.2.3. Standard Preparation Procedures

Refer to the standard preparation logbook or database for more information regarding the preparation of standards for GRO analysis.

10.2.3.1. Working GRO Calibration Standards Preparation

Refer to Table 10.3 for examples of possible one-time use calibration standards.

Table 10.3 – Working Calibration Standards (examples only)

Standard	Stock GRO Standard amount	Final Total Volume	Final Concentration
Calibration Std 1	8uL	100mL	0.2 mg/L
Calibration Std 2	20uL	100mL	0.5 mg/L
Calibration Std 3	40uL	100mL	1 mg/L
Calibration Std 4	10uL	5mL	5 mg/L
Calibration Std 5	20uL	5mL	10 mg/L
Calibration Std 6	50uL	5mL	25 mg/L
Calibration Std 7	100uL	5mL	50 mg/L

10.2.3.2. Working GRO Initial Calibration Verification Standard (ICV) Preparation

Add 10uL of the Stock GRO ICV standard (5000ug/mL) per 5mL water for a final ICV concentration of 10mg/L.

10.2.3.3. Working Retention Time Marker Standard Preparation

Add 1-2uL of the Stock Retention Time Marker Standard per 5mL water. The Retention Time Marker Standard is for qualitative purposes only.

11. Calibration

11.1. Initial Calibration

11.1.1. The retention time range for GRO is defined during initial calibration. Specific gasoline components are used to establish the range, pentane (C_5), 2-methylpentane (C_6), 1,2,4-Trimethylbenzene (C_{10}) and n-dodecane (C_{12}). The marker standard is analyzed prior to initial calibration standards. The retention time range is then calculated based on the lower limit of the RT window for the first eluting component and the upper limit of the RT window for the last eluting component. The total area within the RT window is used for quantitation.

11.1.2. For initial calibration, analyze a minimum of five concentrations of calibration standard. The lowest calibration standard must be at or below the required reporting limit. Determine the calibration factor (CF) of each standard using the calculation below. Determine the relative standard deviation (RSD) of the calibration factors using the calculation below. If the RSD is $\leq$20% over the working range, linearity through the origin is assumed, and the average calibration factor can be used to calculate sample concentrations in place of a calibration curve. Refer to the Quality Manual for more information regarding calibration curves.

CF = Peak Area/ug of Standard RSD = (Std. Dev./CF) x 100

11.1.3. If the RSD is >20% over the working range, then linearity through the origin cannot be assumed. A regression equation that does not pass through the origin can be employed. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$0.99. Refer to Method 8000C for additional information regarding calibration.

11.1.3.1. If a linear regression calibration model is used, the lowest calibration standard should be quantitated against the curve to determine if bias exists at the low end of the linear range. If the recovery of the lowest calibration standard is within +/-30% of the true value, the linear calibration model may be used. Otherwise, a weighted linear curve fit or recalibration should be considered.

11.1.4. Initial Calibration Corrective Action: If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.1.5. Each day that analysis is performed, the calibration standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks/patterns look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably. Additionally, examination of signal-to-noise ratio of analytes in low-level

standards may be a useful diagnostic tool to monitor instrument performance. Signal-to-noise ratio is defined as the ratio of the analyte signal to the noise measured on a blank. It is recommended but not required that the signal-to-noise ratio be at least three to confirm detection.

11.1.6. Initial Calibration Verification (ICV): The initial calibration must be verified through the analysis of an Initial Calibration Verification (ICV) standard. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, a different lot from the same vendor may be used. The ICV is analyzed immediately following the initial calibration curve. The ICV recovery is evaluated against a default acceptance range of 70-130% recovery using the calculation below.

$$\% \text{ Recovery} = \frac{\text{Observed concentration}}{\text{Theoretical concentration}} \times 100$$

11.1.7. ICV Corrective Action: If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee. **Exception:** If the ICV exceeds the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.1.8. Retention Time Windows: The retention time range for GRO is defined during initial calibration. The range is established from the retention times of the C_5, C_6, C_{10} and C_{12} alkanes. To establish retention time windows, make three or more injections of a retention time marker standard over the course of a 72-hour period, at a minimum. Record the retention time in minutes for each alkane and surrogate peak to three decimal places. Calculate the mean and standard deviation of the absolute retention times of the peaks. The retention time window is defined as +/-3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.

Establish the center of the retention time window daily by using the absolute retention times from the retention time marker standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention times of retention time marker standard analyzed prior to the initial calibration.

11.2. Daily and Continuing Calibration

11.2.1. Retention times and the initial calibration must be verified daily as a minimum requirement. Daily analysis begins with the analysis of the retention time marker standard and a Continuing Calibration Verification Standard (CCV). The CCV must be analyzed daily following the marker standard, after every 10 injections, and at the end of the analytical sequence, at a minimum.

11.2.2. For each CCV, if the response or calculated concentration is within +/-20% Difference of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF values from the initial calibration to calculate sample results. An LCS may be used as a CCV provided that it passes the CCV acceptance criteria.

$$\% \text{ Difference} = [CF - CF_{avg}] / CF_{avg} * 100$$

11.2.3. CCV Corrective Action*:* If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. Alternatively, an LCS may be used as a CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

12. Procedures

12.1. Sample Preparation and Handling

12.1.1. Aqueous Samples

12.1.1.1. Quickly measure a 5mL aliquot of the sample using a 5mL gastight syringe and transfer it to a clean, labeled 40mL VOA vial. Perform this transfer as quickly as possible to minimize analyte loss. Thoroughly rinse the syringe inside and out with reagent water before measuring each sample.

12.1.1.2. Prepare a Method Blank: quickly measure a 5mL aliquot of reagent water using a 5mL gastight syringe and transfer it to a clean, labeled 40mL VOA vial.

12.1.1.3. Prepare an LCS: quickly measure a 5mL aliquot of reagent water using a 5mL gastight syringe and transfer it to a clean, labeled 40mL VOA vial and add 10uL of the Stock GRO ICV (5000ug/mL) for an LCS concentration of 10mg/L.

12.1.1.4. Prepare a Matrix Spike: quickly measure a 5mL aliquot of sample using a 5mL gastight syringe and transfer it to a clean, labeled 40mL VOA vial and add 10uL of the Stock GRO ICV (5000ug/mL) for a Matrix Spike concentration of 10mg/L.

12.1.1.5. Dilutions on aqueous samples must be prepared in a volumetric fashion. Sample aliquots are measured in either a volumetric pipet or gas-tight syringe and brought to volume in either a volumetric flask or gas-tight syringe.

12.1.1.6. The pH of all aqueous samples must be determined using pH paper. The pH should be <2. Holding time for water samples with pH >2 is 7 days. Qualify on the sequence log and in LIMS any sample not meeting the pH requirement and/or holding time requirement. A default comment may be used to document on sequence logs that all water samples are pH<2 unless otherwise noted.

12.1.2. Low-Level Soil Samples

12.1.2.1. Terra Core Vials: Prior to analysis the sample weight must be determined and recorded by weighing the vial and recording the weight. Subtract the tare weight indicated on the vial and correct for the label weight to determine the sample weight. Mix the sample well on a vortex mixer and allow it to settle. The sample is ready for analysis. Refer to Section 7 for additional information regarding handling of Terra Core samples.

12.1.2.2. Coring Devices: Samples received in coring devices, such as Encore, must be extruded into a pre-weighed VOA vial either with or without 5mL reagent water and a stir bar. Record the weight of the vial after the sample has been placed into it. Subtract the tare weight determined initially to determine the sample weight. Mix the sample well on a vortex mixer and allow it to settle. The sample is ready for analysis.

12.1.2.3. Bulk Soil Jars: Samples received in bulk soil jars are are sub-sampled into a VOA vial. Place an empty, labeled VOA vial on the balance pan and tare the balance. Quickly add 5 +/-0.5g of the sample to the vial. Record the sample weight. Add 5mL of reagent water and a magnetic stir bar and cap the vial. Mix the sample well on a vortex mixer and allow it to settle. The sample is ready for analysis.

12.1.2.4. Prepare a Method Blank: Place an empty, labeled VOA vial on the balance pan and tare the balance. Quickly add 5 +/-0.5g of simulated soil matrix to the vial. Record the weight. Add 5mL of reagent water and a magnetic stir bar and cap the vial.

12.1.2.5. Prepare an LCS: Place an empty, labeled VOA vial on the balance pan and tare the balance. Quickly add 5 +/-0.5g of simulated soil matrix to the vial. Record the weight. Add 5mL of reagent water, a magnetic stir bar, and 10uL of the Stock GRO ICV (5000ug/mL) and cap the vial. Final LCS concentration is 10mg/Kg.

12.1.2.6. Prepare a Matrix Spike: Place an empty, labeled VOA vial on the balance pan and tare the balance. Quickly add 5 +/-0.5g of soil sample to the vial. Record the weight. Add 5mL of

reagent water, a magnetic stir bar, and 10uL of the Stock GRO ICV (5000ug/mL) and cap the vial. Final Matrix Spike concentration is 10mg/Kg.

12.1.3. Medium-Level soils

12.1.3.1. Terra Core Vials: Prior to analysis the sample weight must be determined and recorded by weighing the vial and recording the weight. Subtract the tare weight indicated on the vial and correct for the label weight to determine the sample weight. Mix the sample well on a vortex mixer and allow it to settle. A maximum of 200uL of the methanol extract per 5mL reagent water is used for analysis. Refer to Section 7 for additional information regarding sample handling.

12.1.3.2. Coring Devices: Samples received in coring devices, such as Encore, must be extruded into a pre-weighed VOA vial with 5mL methanol. Record the weight of the vial after the sample has been placed into it. Subtract the tare weight determined initially to determine the sample weight. Mix the sample well on a vortex mixer and allow it to settle. A maximum of 200uL of the methanol extract per 5mL reagent water is used for analysis.

12.1.3.3. Bulk Soil Jars: Samples received in bulk soil jars are sub-sampled into a VOA vial. Place an empty VOA vial on the balance pan and tare the balance. Quickly add 5 +/-0.5g of the sample to the vial. Record the sample weight. Add 5mL methanol and cap the vial. Mix the sample well on a vortex mixer and allow it to settle. A maximum of 200uL of the methanol extract per 5mL reagent water is used for analysis.

12.1.3.4. Prepare a Method Blank: Place 4.8mL of reagent water and 200uL of methanol in a clean, labeled 40mL VOA vial.

12.1.3.5. Prepare an LCS: Place 4.8mL of reagent water and 200uL of methanol in a clean, labeled 40mL VOA vial and add 10uL of the Stock GRO ICV (5000ug/mL) for an LCS concentration of 10mg/Kg.

12.1.3.6. Prepare a Matrix Spike: Place 4.8mL of reagent water and 200uL of sample methanol extract in a clean, labeled 40mL VOA vial and add 10uL of the Stock GRO ICV (5000ug/mL) for a Matrix Spike concentration of 10mg/Kg.

12.2. Sample Analysis

12.2.1. Configure the purge & trap system and GC/MS system per manufacturer's instructions. All samples must be analyzed at room temperature and the system must be calibrated and free of contamination before samples are analyzed.

12.2.2. The total area within the RT range is the sample response. Sample concentrations are calculated by comparing sample response data with the initial calibration. Results are based on retention time windows for specific carbon ranges quantitated against a gasoline standard. Pattern matching for quantitative purposes is not performed on a routine basis. Refer to the Manual Integration SOP for manual integration guidelines.

12.2.3. If sample response exceeds the limits of the initial calibration range, a dilution of the sample must be analyzed or over range concentrations must be qualified as estimated.

12.2.4. It may be necessary to analyze a solvent blank after a highly concentrated sample to eliminate carryover.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or simulated soil matrix	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Reanalyze associated samples if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required. 3) If sample concentration is >10x blank level, sample and method blank may be reported, sample must be qualified.
Laboratory Control Sample (LCS)	Target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits for recovery. Refer to the LIMS for acceptance limits.	Reanalyze LCS. If LCS is still outside acceptance limits, re-prepare and reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits for recovery. ≤20% RPD Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogate	Applicable surrogate compound	Added to each standard, sample, and method blank.	Lab-generated limits for recovery. Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample results are non-detect, the sample results may be reported without qualifiers. The surrogate must be qualified. 3) If surrogate results are above the QC limits due to obvious sample matrix interference, results may be reported but must be qualified. 4) MS/MSD surrogate recovery failures do not constitute the reanalysis of samples but the surrogate data must be qualified.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

Aqueous Sample (ug/L) = $(X_s)(D)$ Solid Sample (mg/kg) = $\frac{(X_s)(V_f)(D)}{(W_s)(1000)}$

Where: X_s = On-column concentration of the analyte, ug/L
V_f = Volume purged or total volume of methanol extract, L
D = Dilution factor of aqueous sample or methanol extract
W_s = Weight of solid sample purged or extracted, Kg

Moisture corrected concentration = $\frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$

14.2. LCS equation:

$$\mathbf{R = (C/S) * 100}$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Action for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. An MDL study and/or LOD/LOQ verification must be conducted annually for each matrix per instrument.

18.2. Each analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. GC columns and chromatographic conditions may differ from those recommended in 8015D.

20. Instrument/Equipment Maintenance

20.1. Refer to maintenance log and/or instrument manufacturer's instructions.

21. Troubleshooting

21.1. Refer to maintenance log and/or instrument manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods, 8000C, 8015D, 5030A, 5030B, and 5035A.

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-O-109-rev.11	1. Cover page: added actual effective date. 2. Table 8.3: revised balance specifications to match practice 3. Section 9.2.3.4: changed approx. 5g to 5 +/- 0.5g, added batch QC details for medium-level soils 4. Section 10.1.3: removed 8000C equations and made reference to 8000C. 5. Section 10.1.6: added as guidance for evaluation of ICAL standards. 6. Section 10.2.3: clarified that LCS can be used as a CCV. 7. Section 11.2.2: added that tared soil vials need to be corrected for label weight and changed approx. 5g to 5 +/- 0.5g. 8. Section 11.3: added reference to the Manual Integrations SOP, 9. Section 12.2: added to clarify the requirement for batch QC 10. Section 12.3: added procedure for MB for both autosampler types. Changed approx. 5g to 5 +/- 0.5g 11. Section 13.1: added optional LOD/OQ verification. 12. Section 16.1: removed reference to 8000B.	31Oct2013
S-IN-O-109-rev.12	1. Cover page: changed 8015B to 8015D and updated phone number. 2. Section 1.1: changed 8015B to 8015D. 3. Section 9.2: updated storage conditions of opened stock standards. 4. Section 9.3: updated low curve point concentration. 5. Section 1.01.1: added pentane as part of GRO range and added C_{10} as the alkane identifier of 1,2,4-Trimethylbenzene. 6. Section 10.1.3.1: added for evaluation of linear calibration models. 7. Section 10.1.8: added more detailed information on determination of retention time windows. 8. Section 10.2.2: changed CCV criteria from +/-15% to +/-20%. 9. Section 11.3: In the first sentence, changed “window” to “range.” 10. Section 14.1: changed 8015B to 8015D. 11. Section 16.1: changed 8015B to 8015D.	02Sep2015
S-IN-O-109-rev.13	1. Converted to 27 section format. 2. Table 7.1: updated storage temperature format, added storage option for methanol vials, and added methanol preservative option for bulk soil jar prep. 3. Section 9.3: added vortex mixer. 4. Section 12: completely reorganized to include prep for all types of sample containers plus batch QC. 5. Table 13.1: updated components of batch QC and LCS corrective action and updated method blank corrective action. 6. Section 14: corrected equations to be in like terms with instrument output. 7. Section 25.3: added years 2003 and 2009 to TNI reference.	06Sep2017

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STANDARD OPERATING PROCEDURE

SEPARATORY FUNNEL EXTRACTION
REFERENCE METHOD: EPA SW-846 METHOD 3510C

SOP NUMBER:	S-IN-O-054-rev.16
EFFECTIVE DATE:	September 18, 2017
SUPERSEDES:	S-IN-O-054-rev.15

APPROVAL

General Manager — September 7, 2017 Date

Quality Manager — September 6, 2017 Date

Department Manager — September 6, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for extracting non-volatile and semi-volatile organic compounds from groundwater and surface water samples in a separatory funnel while meeting the requirements specified in SW-846 Method 3510C.

2. Summary of Method

2.1. A measured volume of sample, normally about 1 liter, is serially extracted with solvent in a separatory funnel. Reduced sample volumes may be used providing that the ratio of sample to solvent remains consistent with the ratio indicated in Method 3510C. Reduced sample volume extraction may also be referred to as Low Volume Extraction (LVE). Some extractions also require the monitoring and adjusting of the pH of the sample. The extract is separated from the sample and is concentrated, followed by cleanup, if necessary, or analysis.

3. Scope and Application

3.1. Applicable compounds, volumes/weights utilized, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.2. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of separatory funnel equipment and reagents. Each analyst work cell must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This procedure is for extracting water insoluble or slightly water soluble organic compounds from groundwater, surface water and other aqueous samples using methylene chloride as the extraction solvent.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Solvents, reagents and glassware can all contribute to compound artifacts or raised baselines; both conditions that can affect chromatography. Analyzing method blanks is therefore crucial in determining the presence of contaminants.

6.2. Phthalate esters are common contaminant products in many products in the lab. All plastic products should be avoided when performing this method.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid, preferably 1L, 125mL widemouth, or equivalent.	None	Cool to ≤6°C	Samples must be extracted within 7 days of collection date and extracts must be analyzed within 40 days of extraction date. Samples for **PCB** analysis must be extracted within 6 months of collection date and extract must be analyzed within 40 days of extraction date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Vendor	Description / Comments
N-EVAP concentrator	Organomation	Or equivalent equipment
Zymark concentrator (2) and glassware	Zymark	Or equivalent equipment
Shaker Tables	Glass-Col	Or equivalent equipment

9.2. General Supplies

Item	Description
Separatory Funnels	2L or 125mL, Teflon, with PTFE stopcocks and Teflon lids or equivalent
Glass beakers	400mL Pyrex or equivalent
Autosampler vials	~2mL, clear glass with aluminum crimp-top seals
Micro-syringes	Various sizes
Glass funnels	
Glass wool	
Graduated cylinders	Glass, Class A
Kuderna-Danish Concentrator Sets	500mL flash with 10mL concentrator tube and 3-ball Snyder column
Heated water bath	Temperature controlled
Boiling Chips	Teflon or equivalent
Pasteur pipettes	For testing sample pH
pH paper	pH range 1-12
Glass stirring rods	For breaking up emulsions
Glass tubes	Disposable, 20x150mm or equivalent
Filter paper	For filtration of extract
Pipettes	Volumetric, Class A, various sizes

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
Sodium Sulfate	Anhydrous, granular 10-60 mesh, meets ACS specs or equivalent. Rinse thoroughly with methylene chloride and allow it to dry prior to use.
Methylene Chloride	Extraction solvent, pesticide grade or equivalent
Acetone	Extraction solvent, pesticide grade or equivalent
Hexane	Exchange solvent, pesticide grade or equivalent
Sulfuric acid solution (1:1)	Reagent grade
Sodium Hydroxide solution (10N)	Dissolve 400g sodium hydroxide pellets into 1L of reagent water or purchase premade

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions and vendors

Standard	Description	Comments
Surrogate standard	Surrogates are added to each sample and QC sample to monitor extraction efficiency.	
Spiking Standard	This solution contains all target analytes.	Same solution can be used for the LCS and MS/MSD

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock BNA & BNA LVE spike standard	1. Restek; catalog # 31004, B/N spike; 1000ug/mL, or equivalent 2. Restek; catalog # 31014, Acid spike; 2000ug/mL, or equivalent 3. Restek; catalog # 561763, Custom PAH spike; 5000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working BNA & BNA LVE spike standards	Refer to Section 10.2.3.1	Good for 6 months from preparation date	Refrigerate
Stock/Working BNA & BNA LVE surrogate standard	O2si; catalog # 11004-83-1L; 100ug/mL, or equivalent. Use 1mL for each BNA. Use 100uL for each BNA LVE.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock Full-list BNA & Full list BNA LVE spike standard	1. SVOA MegaMix; Restek; cat#31850; 1000ug/mL 2. 8270 Mix 1; Restek; cat#572178, 2000ug/mL 3. 8270 Mix 2: Restek; cat#572448, 2000ug/mL or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working Full-list BNA & Full list BNA LVE spike standard	Refer to Section 10.2.3.2	Good for 6 months from preparation date	Refrigerate

Standard Type	Description	Expiration	Storage
Stock PCB spike standard	Restek; catalog #32039, Aroclors 1016/1260; 1000ug/mL, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Intermediate PCB spike standard	Refer to Section 10.2.3.3	Good for 6 months from preparation date	Refrigerate
Working PCB spike standard	Refer to Section 10.2.3.4	Good for 6 months from preparation date	Refrigerate
Stock PCB/8081 surrogate standard	Restek; catalog#32457, TCMX/DCB mix; 200ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PCB/8081 surrogate standard	Refer to Section 10.2.3.5	Good for 6 months from preparation date	Refrigerate
Stock 8081 spike standard	Restek; catalog #32292, 8-80ug/mL of each compound, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8081 spike standard	Refer to Section 10.2.3.6	Good for 6 months from preparation date	Refrigerate
Stock DRO spike standard	Restek; catalog # 31258, Diesel #2 standard; 50,000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working DRO spike standard	Refer to Section 10.2.3.7	Good for 6 months from preparation date	Refrigerate
Stock DRO surrogate standard	Restek; catalog # 31487, Pentacosane; 10,000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working DRO surrogate standard	Refer to Section 10.2.3.8	Good for 6 months from preparation date	Refrigerate
Stock PAH-SIM & PAH-SIM LVE spike standard	Restek; catalog # 31622, Cal. Mix 5; 2000ug/mL of each compound, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PAH-SIM & PAH-SIM LVE spike standard	Refer to Section 10.2.3.9	Good for 6 months from preparation date	Refrigerate
Stock PAH-SIM & PAH-SIM LVE surrogate standard	Restek; catalog # 31062, B/N surrogate; 5000ug/mL of each compound, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PAH-SIM & PAH-SIM LVE surrogate standard	Refer to Section 10.2.3.10	Good for 6 months from preparation date	Refrigerate
Stock/Working Scan/SIM Combo & Scan/SIM Combo LVE spike standard	O2si, catalog #114072-06, 10-100ug/mL or equivalent. Use 1ml for each LCS, MS and MSD. Use 100uL for the LVE LCS, MS, and MSD.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Freeze after opening.
Stock/Working Scan/SIM Combo & Scan/SIM Combo LVE surrogate spike standard	O2si, catalog #114071-06; 10-100ug/mL or equivalent. Use 1mL for each sample, Method Blank, LCS, MS and MSD. Use 100uL for the LVE LCS, MS, and MSD.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Freeze after opening.

Standard Type	Description	Expiration	Storage
Stock TCLP BNA spike standard	1. Restek; catalog # 31028, TCLP B/N spike; 2000ug/mL of each compound, or equivalent 2. Restek; catalog # 31027, TCLP Acid spike; 2000ug/mL of each compound, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working TCLP BNA spike standard	Refer to Section 10.2.3.11	Good for 6 months from preparation date	Refrigerate
Stock 8141 spike standard	Ultra; catalog #CUS-12835, 100ug/mL of each compound or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8141 spike standard	Refer to Section 10.2.3.12	Good for 2 months from preparation date	Refrigerate
Stock 8141 surrogate standard	Accustandard; catalog #M-507-1S-10X, Triphenylphosphate 5000ug/mL or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8141 surrogate standard	Refer to Section 10.2.3.13	Good for 2 months from preparation date	Refrigerate

10.2.3. Standard Preparation Procedures

10.2.3.1. Working BNA & BNA LVE Spike Standard Preparation

Dilute 2.5mL of the stock Acid spike standard (2000ug/mL), 5mL of the stock B/N spike standard (1000ug/mL) and 1mL of the stock Custom PAH spike (5000ug/mL) to 50mL with acetone for a final concentration of 100ug/mL. Add 1mL of this working standard to each BNA LCS, MS and MSD. Add 100uL of this working standard to each BNA LVE LCS, MS and MSD.

10.2.3.2. Working Full-list BNA & Full-list BNA LVE Spike Standard Preparation

Dilute 2mL of Stock Mix A (1000ug/mL), 1mL of Stock Mix B2 (2000ug/mL) and 1mL of Stock Mix C (2000ug/mL) to 20mL in acetone for a final concentration of 100ug/mL. Add 1mL of this working standard to each Full-list BNA LCS, MS, and MSD. Add 100uL of this working full-list spike standard to each BNA LVE LCS, MS, and MSD.

10.2.3.3. Intermediate PCB Spike Standard Preparation

Dilute 1mL of the Stock PCB spike standard (1000ug/mL) to 200mL in acetone for a final concentration of 5ug/mL.

10.2.3.4. Working PCB Spike Standard Preparation

Dilute 1mL of the Intermediate PCB standard (5ug/mL) to 500mL with acetone for a final concentration of 1ug/mL. Add 1mL of this working spike to each PCB LCS, MS and MSD.

10.2.3.5. Working PCB/8081 Surrogate Standard Preparation

Dilute 5mL of Stock 8081/PCB Surrogate Standard (200ug/mL) to 400mL in acetone for a final concentration of 2.5ug/mL. Add 200uL of this working standard to each 8081 and PCB sample, Method Blank, LCS, MS and MSD.

10.2.3.6. Working 8081 Spike Standard Preparation

Dilute 5mL of 8081 Stock Spike Standard (8-80ug/mL) to 20mL in acetone for a final concentration of 2-20ug/mL. Add 100uL of this working spike standard to each 8081 LCS, MS and MSD.

10.2.3.7. Working DRO Spike Standard Preparation

Dilute 10mL of the stock DRO spike standard (50,000ug/mL) to 250mL with acetone for a final concentration of 2500ug/mL. Add 1mL of this working standard to each DRO/ERO/Ohio mod LCS, MS and MSD.

10.2.3.8. Working DRO Surrogate Standard Preparation

Dilute 7.5mL of the stock DRO/DRO LVE surrogate standard (10,000ug/mL) to 500mL with acetone for a final concentration of 150ug/mL. Add 1mL of this working surrogate to each DRO/ERO/Ohio mod sample, Method Blank, LCS, MS and MSD.

10.2.3.9. Working PAH-SIM & PAH-SIM LVE Spike Standard Preparation

Dilute 5mL of the stock PAH-SIM/PAH-SIM LVE spike standard (2000ug/mL) to 200mL with acetone for a final concentration of 50ug/mL. Add 200uL of this working spike to each PAH-SIM LCS, MS and MSD. Add 20uL of this working spike to each PAH-SIM LVE LCS, MS and MSD.

10.2.3.10. Working PAH-SIM & PAH-SIM LVE Surrogate Standard Preparation

Dilute 5.0mL of the stock PAH-SIM/PAH-SIM LVE surrogate standard (5000ug/mL) to 500mL with acetone for a final concentration of 50ug/mL. Add 200uL of this working surrogate to each PAH-SIM sample, Method Blank, LCS, MS and MSD. Add 20uL of this working surrogate to each PAH-SIM LVE sample, Method Blank, LCS, MS and MSD.

10.2.3.11. Working TCLP BNA Spike Standard Preparation

Dilute 1mL of the stock TCLP acid spike standard (2000ug/mL) and 1mL of the stock TCLP B/N spike standard (2000ug/mL) to 20mL with acetone for a final concentration of 100ug/mL. Add 1mL of this working spike to each TCLP BNA LCS, MS and MSD.

10.2.3.12. Working 8141 Spike Standard Preparation

Dilute 1mL of the Stock 8141 spike standard (100ug/mL) to 5mL in acetone for a final concentration of 20ug/mL. Add 100uL of this working spike to each 8141 LCS, MS and MSD.

10.2.3.13. Working 8141 Surrogate Standard Preparation

Dilute 200uL of the Stock 8141 Surrogate Standard (5000ug/mL) to 10mL in acetone for a final concentration of 100ug/mL. Add 25uL of this working surrogate standard to each 8141 sample, Method Blank, LCS, MS and MSD.

11. Calibration

11.1. Not applicable to this SOP.

12. Procedures

12.1. Make sure that all glassware and Teflon separatory funnels used for this procedure have been properly washed. All washed glassware must be rinsed prior to use with acetone to remove residual water and rinsed with methylene chloride to remove any residual contaminants.

12.2. Measure the initial pH of each sample using wide range pH paper by dipping a clean disposable Pasteur pipette into each sample and touching the pipette to a piece of pH paper. Record the initial pH in the extraction log.

12.3. A nominal volume of 1L of aqueous sample is routinely extracted. Low Volume Extractions (LVE) use a nominal volume of 100mL of aqueous sample. For samples expected to contain high concentrations of analytes, use a smaller aliquot of sample diluted to 1L or 100mL with reagent water.

12.4. Prepare a method blank by placing 1L or 100mL of reagent water in to a labeled separatory funnel in a ring stand on a secured rack. The method blank will be used to check for contamination in the system.

12.5. Prepare an LCS by placing 1L or 100mL of reagent water in to a labeled separatory funnel in a ring stand on a secured rack. Spike the reagent water with the appropriate amount of spike solution. The LCS will be used to determine the efficiency of the extraction method in extracting target compounds.

12.6. Using a Class A graduated cylinder, measure the desired volume of sample to be used for the extraction and record the volume in mLs. Transfer the sample into a labeled separatory funnel in a ring stand on a secured rack.

12.7. If available sample volume allows, prepare a matrix spike (MS) and matrix spike duplicate (MSD) in separate, labeled separatory funnels.

12.8. Add the appropriate **surrogate** solution to each method blank, sample, LCS, MS and MSD. Add the appropriate **spiking** solution to the LCS, MS and MSD. Refer to the standard preparation log and the sample preparation log for details regarding the appropriate surrogate and spiking solutions and volumes to be used.

12.9. Adjust the sample pH, if necessary, to the pH indicated in Table 1 using 1:1 Sulfuric Acid or 10N Sodium Hydroxide. The pH is checked by dipping the tip of a disposable glass pipet into each well-mixed sample and placing the tip onto the pH paper to obtain a pH measurement.

12.10. Rinse the graduated cylinder (or sample bottle) with the first 60mL portion of extraction solvent for a 1L sample or the first 6mL portion of extraction solvent for a 100mL sample and transfer the rinsate to the separatory funnel.

12.11. Seal the separatory funnels with Teflon lids and shake for two minutes with periodic venting. This can be done manually or on an automatic shaker. **NOTE**: Methylene chloride may cause excessive pressure in the separatory funnel. It is recommended to shake slightly and vent before placing funnels on an automatic shaker.

12.12. Return the 2L separatory funnels to their ring stands and allow the solvent layer to separate from the aqueous phase. The 125mL separatory funnels remain in the automatic shaker for draining. If an excessive emulsion is present in the solvent layer, it can be broken up manually by using a clean glass stirring rod. If this is not successful, the sample can be drained into a clean secondary container (i.e. VOA vial) and transferred to a centrifuge tube. The extract can be centrifuged and then decanted into the drying funnel.

12.13. Drain the solvent layer through a drying funnel consisting of a clean funnel containing a plug of clean glass wool topped with a portion of clean sodium sulfate. The solvent should be collected in labeled

beakers, labeled KD glassware, or labeled glass tubes.

12.14. Repeat the extraction two additional times with 60mL or 6mL aliquots of solvent. Drain the solvent through the drying funnel after each extraction. After the final solvent extraction has been collected, rinse the funnel with methylene chloride and remove the drying funnel.

12.15. If extraction at a secondary pH is required, add acid or base as necessary and serially extract the sample, as described in Sections 12.11-12.13, at the adjusted pH. Collect all sample extract fractions together for concentration. Refer to Table 1 for extraction conditions.

12.16. **Concentration procedure for 8270 BNA, 8270 Scan/SIM Combo, and TCLP samples:** Pour the extract into a labeled Kuderna-Danish concentrator with a concentrator tube securely attached. Add one or two clean boiling chips to the KD flask and attach a 3-ball Snyder column. Place the KD apparatus on a hot water bath so that the flask is partially immersed in the water. At the proper rate of distillation, the balls of the Snyder column should actively chatter but the chambers should not flood with solvent. Adjustment of the angle of the apparatus and the water temperature may be necessary to make the boiling more efficient. When the apparent volume of the extract reaches 4-6mL, remove the apparatus from the water bath and allow it to cool. Once cooled, carefully disassemble the KD apparatus rinsing each joint into the concentrator tube with a small amount of extraction solvent. Place the concentrator tube into the N-Evap and further concentrate the extract until the apparent volume is slightly below 1mL. Continue to Section 12.20.

12.17. **Concentration of PAH-SIM LVE samples:** Pour the extract into a labeled 20x150mm glass tube and place the tube on the N-Evap concentrator in a warm water bath (about 40^{o}C) and evaporate the solvent volume using a gentle stream of nitrogen. During the process, the tube should be rinsed several times with the appropriate solvent. The tube should be positioned so that water will not condense into the sample and the sample should not be allowed to go below 0.5mL, this could lead to losses of semi-volatile compounds.

12.18. **Concentration procedure for all other samples:** Pour the entire sample extract into a labeled Zymark extractor tube and place in the Zymark concentrator apparatus. Adjust the settings per manufacturer's instructions. When the apparent volume is slightly below the intended final volume, remove the concentrator from the apparatus and allow it to cool.

12.19. If a solvent exchange is required, see Table 1, add 50mL of the exchange solvent to the Zymark tube. Concentrate the extract to slightly below the intended final volume, remove from the water bath and allow it to cool.

12.20. If further concentration is necessary for any sample extract, nitrogen blowdown can be performed. For this procedure, place the concentrator tube on the N-Evap concentrator in a warm water bath (about 40^{o}C) and evaporate the solvent volume using a gentle stream of nitrogen. During the process, the tube should be rinsed several times with the appropriate solvent. The tube should be positioned so that water will not condense into the sample and the sample should not be allowed to go below 0.5mL, this could lead to the loss of semi-volatile compounds.

12.21. Prepare a calibrated vial by volumetrically dispensing the required volume of the solvent being used into a vial and securely capping the vial to eliminate evaporation. The calibrated vial must be prepared daily using a Class A pipet. Quantitatively transfer the sample extract from the Zymark tube or concentrator tube to a vial. Bring the sample extract in the vial to the required final volume listed in Table 1 by visually comparing the sample extract vial volume to the calibrated vial volume. Securely cap the sample extract vial. Store all extracts in the appropriate storage cooler. For extracts that will not concentrate to the usual final volume, use the procedure described above to bring the extract to the next higher practical volume for which a calibrated vial can be prepared.

12.22. Refer to appropriate cleanup SOPs if extract cleanup is required.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples	Target analytes must be less than reporting limits	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, affected data must be qualified. 3) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples	Lab-generated limits Refer to LIMS for control limits.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples	Lab-generated limits Refer to LIMS for control limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogate	Applicable surrogate compound	Added to each sample, standard and method blank	Lab-generated limits Refer to LIMS for control limits.	Samples with surrogate failures must be re-extracted and reanalyzed. QC samples with surrogate failures require the re-extraction and reanalysis of the QC samples and the associated client samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) If only one surrogate for BNA or Combo fails and it is >10% recovery, re-extraction is not required but affected data must be qualified. 4) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. Analysts performing this method must document acceptable accuracy and precision by passing a demonstration of capability study (DOC) on an annual basis.

19. Method Modifications

19.1. Spikes not added to graduated cylinder or sample bottle but instead added to the separatory funnel.

20. Instrument/Equipment Maintenance

20.1. Refer to manufacturer's instructions.

21. Troubleshooting

21.1. Refer to manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3510 "Separatory Funnel Extraction".

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Table 1: Extraction Conditions

27. Revisions

Document Number	Reason for Change	Date
S-IN-O-054-rev.13	1. Effective date added to cover page and body header. 2. Table 9.1: added preparation of sodium sulfate for use by solvent rinsing. 3. Section 11: revised to add new process of determining sample volume prior to extraction using Class A graduated cylinder. Removed language referring to the previous method of sample volume determination. Reordered sections to reflect the addition of surrogates and spike prior to the addition of solvent. Added a reference to extract cleanup SOPs. 4. Section 13.1: added optional LOD/LOQ verification. 5. Section 14: removed previous method modification of determining sample volume post extraction.	03Nov2013
S-IN-O-054-rev.14	1. Section 3.2: added Pesticides. 2. Section 9.2.2: added detail for 8081 and 8141 standards 3. Section 9.2.3: added detail for 8081 and 8141 standards 4. Table 1: added detail for 8081 and 8141 extractions.	28Feb2015
S-IN-O-054-rev.15	1. Table 7.1: revised holding time for extraction of samples for PCB analysis. 2. Table 9.3: updated storage conditions for standards and added Combo LVE spike. 3. Section 12: removed equations for LCS, RSD, MS. 4. Section 13: removed MDL study requirement.	02Sep2015
S-IN-O-054-rev.16	1. Converted to 27 section format. 2. Table 7.1: updated storage temperature format. 3. Table 10.3: updated standard descriptions. 4. Section 10.2.3: updated standard preparation where needed. 5. Section 12: separated instructions for method blank, LCS and MS/MSD. Specified "first portion" of solvent used to rinse cylinder or bottle. 6. Section 19: removed modification for some samples getting two extractions at each pH. 7. Section 25.3: added years 2003 and 2009 to TNI reference. 8. Table 1: removed columns for # of extractions.	05Sep2017

Table 1 – Extraction Conditions

Determinative Method	Initial Extraction pH	Secondary Extraction pH	Extraction Solvent	Exchange Solvent	Final Extract Volume (mL)
8015 DRO & DRO LVE (includes ERO and Ohio mod)	<2	N/A	Methylene Chloride	N/A	1
8081 OC PEST	5-9	N/A	Methylene Chloride	Hexane	10
8082 PCB	5-9	N/A	Methylene Chloride	Hexane	10
8141 OP PEST	As received	N/A	Methylene Chloride	Hexane	10
8270 PAH-SIM & PAH-SIM LVE	>11	N/A	Methylene Chloride	N/A	1
8270 BNA LVE	<2	>11	Methylene Chloride	N/A	1
8270 BNA Scan/SIM Combo LVE	<2	>11	Methylene Chloride	N/A	1
8270 TCLP BNA	<2	>11	Methylene Chloride	N/A	1

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STANDARD OPERATING PROCEDURE

MICROWAVE EXTRACTION
REFERENCE METHOD: EPA SW-846 METHOD 3546

SOP NUMBER:	S-IN-O-130-rev.06
EFFECTIVE DATE:	September 18, 2017
SUPERSEDES:	S-IN-O-130-rev.05

APPROVAL

General Manager — September 5, 2017 Date

Quality Manager — September 5, 2017 Date

Department Manager — September 5, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for extracting non-volatile and semi-volatile organic compounds from solid samples, such as soil and sediment, using a microwave extraction system while meeting the requirements specified in EPA method 3546.

2. Summary of Method

2.1. An aliquot of sample is weighed and loaded into an extraction vessel and the appropriate solvent mixture is added and the vessel is sealed. The vessel is heated to the extraction temperature and extracted for a pre-determined length of time. The mixture is allowed to cool. The vessel is opened and the contents are filtered. The solid material is rinsed and the various solvent fractions are combined. The extract is concentrated and exchanged as necessary.

3. Scope and Application

3.1. Methylene chloride or a mixture of methylene chloride and acetone are routinely used as extraction solvents. Other solvent systems may be used provided adequate performance can be demonstrated for the target analytes.

3.2. Applicable compounds, volumes/weights, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of microwave extraction equipment and reagents. Each analyst work cell must demonstrate their capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This procedure is for extracting water insoluble or slightly water soluble organic compounds from soils, sediments or other solid waste materials.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Solvents, reagents and glassware can all contribute to compound artifacts or elevated baselines; both conditions that can affect chromatography. Method blanks must be analyzed to determine the presence of contaminants.

6.2. Phthalate esters are common contaminant products in many products in the lab. All plastic products should be avoided when performing this method.

6.3. Extracts that exhibit interferences can be run through a cleanup procedure-see EPA method 3600. Before using a cleanup method, the analyst must run a series of calibration standards through the procedure to ensure that the elution order of compounds remains the same and that no new interferent has been introduced by the cleanup method.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Solid	Glass container with Teflon-lined lid (4oz jar).	None	Cool to ≤6°C	Samples must be extracted within 14 days of the collection date and extract must be analyzed within 40 days of extraction date. Samples for **PCB** analysis must be extracted within 6 months of collection date and extract must be analyzed within 40 days of extraction date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment

Equipment	Vendor	Description / Comments
Microwave Extractor	CEM MARS Express	or equivalent microwave extractor
Mechanical Capper	CEM	or equivalent
Analytical Balance	AND or equivalent	Capable of measuring to 0.1g
Concentrator apparatus	Zymark Turbo VAP II	or equivalent concentrator

9.2. Supplies

Item	Description
Weigh boats/Aluminum pans	For weighing soil samples
Teflon tubes	For use in the microwave extraction system
Concentrator tubes	Graduated for concentration of sample to 1 mL
Autosampler vials	~2mL, clear glass with aluminum crimp-top seals
Micro-syringes	Various sizes
Glass funnels	
Glass wool	
Filter paper	For filtering extracts
Spatulas	
Pipettes	Volumetric, Class A, various sizes

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
Sodium Sulfate	Anhydrous, granular 10-60 mesh, meets ACS specs or equivalent. Rinse thoroughly with methylene chloride and allow it to dry prior to use.
Methylene Chloride	Extraction solvent, pesticide grade or equivalent
Acetone	Extraction solvent, pesticide grade or equivalent
Hexane	Exchange solvent, pesticide grade or equivalent
Solvent Mixture	3:1 Methylene Chloride:Acetone
Ottawa sand	Mesh size 20-30 or other equivalent simulated soil matrix.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, initial calibration verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions and vendors

Standard	Description	Comments
Surrogate standard	Surrogate compounds must be added to each sample and QC sample to monitor extraction efficiency.	
Spiking Standard	This solution contains method-specific spike compounds.	Same solution can be used for LCS and MS/MSD

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock BNA spike standard	1. Restek; catalog # 31004, B/N spike; 1000ug/mL, or equivalent 2. Restek; catalog # 31014, Acid spike; 2000ug/mL, or equivalent 3. Restek; catalog # 561763, Custom PAH spike; 5000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working BNA spike standards	Refer to Section 10.2.3.1	Good for 6 months from preparation date	Refrigerate
Stock/Working BNA surrogate standard	O2si; catalog # 11004-83-1L; 100mg/L, or equivalent. Use 1mL for each BNA.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock Full-list BNA spike standard	1. SVOA MegaMix; Restek; cat#31850; 1000ug/mL 2. 8270 Mix 1; Restek; cat#572178, 2000ug/mL 3. 8270 Mix 2: Restek; cat#572448, 2000ug/mL or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.

Standard Type	Description	Expiration	Storage
Working Full-list BNA spike standard	Refer to Section 10.2.3.2	Good for 6 months from preparation date	Refrigerate
Stock PCB spike standard	Restek; catalog # 32039, Aroclor 1016/1260; 1000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PCB spike standard	Refer to Section 10.2.3.3	Good for 6 months from preparation date	Refrigerate
Stock PCB/8081 surrogate standards	Restek; catalog#32457, TCMX/DCB mix; 200ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PCB/8081 surrogate standard	Refer to Section 10.2.3.4	Good for 6 months from preparation date	Refrigerate
Stock 8081 spike standard	Restek; catalog #32292, 8-80ug/mL of each compound, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8081 spike standard	Refer to Section 10.2.3.5	Good for 6 months from preparation date	Refrigerate
Stock DRO spike standard	Restek; catalog # 31258, Diesel #2 standard; 50,000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working DRO spike standard	Refer to Section 10.2.3.6	Good for 6 months from preparation date	Refrigerate
Stock DRO surrogate standard	Restek; catalog # 31487, Pentacosane; 10,000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working DRO surrogate standard	Refer to Section 10.2.3.7	Good for 6 months from preparation date	Refrigerate
Stock PAH-SIM spike standard	Restek; catalog # 31622, Cal. Mix 5; 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PAH-SIM spike standard	Refer to Section 10.2.3.8	Good for 6 months from preparation date	Refrigerate
Stock PAH-SIM surrogate standard	Restek; catalog # 31062, B/N surrogate; 5000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PAH-SIM surrogate standard	Refer to Section 10.2.3.9	Good for 6 months from preparation date	Refrigerate
Stock/Working Scan/SIM Combo spike standard	O2si; catalog#114072-06, 10-100ug/mL or equivalent. Use 1mL each for each Scan/SIM Combo LCS, MS, and MSD.	Manufacturer's recommended expiration date	Manufacturer's recommended expiration date. Freeze after opening.
Stock/Working Scan/SIM Combo surrogate standard	O2si; catalog#114071-06, 10-100ug/mL or equivalent. Use 1mL each for each Scan/SIM Combo	Manufacturer's recommended expiration date	Manufacturer's recommended expiration date. Freeze after opening.
Stock 8141 spike standard	Ultra; catalog #CUS-12835, 100ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8141 spike standard	Refer to Section 10.2.3.10	Good for 2 months from preparation date	Refrigerate

Standard Type	Description	Expiration	Storage
Stock 8141 surrogate standard	Accustandard; catalog #M-507-1S-10X, Triphenylphosphate 5000ug/mL or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8141 surrogate standard	Refer to Section 10.2.3.11	Good for 2 months from preparation date	Refrigerate

10.2.3. Standard Preparation Procedures

10.2.3.1. Working BNA Spike Standard Preparation

Dilute 2.5mL of the stock Acid spike standard (2000ug/mL), 5mL of the stock B/N spike standard (1000ug/mL) and 1mL of the stock Custom PAH spike (5000ug/mL) to 50mL with acetone for a final concentration of 100ug/mL. Add 1mL of this working standard to each BNA LCS, MS and MSD.

10.2.3.2. Working Full-list BNA Spike Standard Preparation

Dilute 5mL of SVOA MegaMix (1000ug/mL), 2.5mL of Stock 8270 Mix 1 (2000ug/mL), and 2.5 mL of Stock 8270 Mix 2 (2000ug/mL) to 50mL with acetone for a final concentration of 100ug/mL. Add 1mL of this working standard to each Full-list BNA LCS, MS, and MSD.

10.2.3.3. Working PCB Spike Standard Preparation

Dilute 1mL of the stock PCB spike standard, Aroclors 1016/1260, (1000ug/mL each) to 200mL with acetone for a final concentration of 5ug/mL. Add 1mL of this working spike to each PCB LCS, MS and MSD.

10.2.3.4. Working PCB/8081 Surrogate Standard Preparation

Dilute 5mL of the Stock 8081/PCB surrogate standard (200ug/mL) to 400mL with acetone for a final concentration of 2.5ug/mL of each surrogate. Add 200uL of this working standard to each PCB and 8081sample, Method Blank, LCS, MS and MSD.

10.2.3.5. Working 8081 Spike Standard Preparation

Dilute 5mL of the Stock 8081 spike standard (8-80ug/mL) to 20mL with acetone for a final concentration of 2-20 ug/mL. Add 100uL of this working spike standard to each 8081 LCS, MS and MSD.

10.2.3.6. Working DRO Spike Standard Preparation

Dilute 10mL of the stock DRO spike standard (50,000ug/mL) to 200mL with acetone for a final concentration of 2500ug/mL. Add 1mL of this working standard to each DRO/ERO/Ohio mod LCS, MS and MSD.

10.2.3.7. Working DRO Surrogate Standard Preparation

Dilute 7.5mL of the stock DRO surrogate standard (10,000ug/mL) to 500mL with acetone for a final concentration of 150ug/mL. Add 1mL of this working surrogate to each DRO/ERO/Ohio mod sample, Method Blank, LCS, MS and MSD.

10.2.3.8. Working PAH-SIM Spike Standard Preparation

Dilute 5mL of the stock PAH-SIM spike standard (2000ug/mL) to 200mL with acetone for a final concentration of 50ug/mL. Add 200uL of this working spike to each PAH-SIM LCS, MS and MSD.

10.2.3.9. Working PAH-SIM Surrogate Standard Preparation

Dilute 5.0mL of the stock PAH-SIM surrogate standard (5000ug/mL) to 500mL with acetone for a final concentration of 50ug/mL. Add 200uL of this working surrogate to each PAH-SIM sample, Method Blank, LCS, MS and MSD.

10.2.3.10. Working 8141 Spike Standard Preparation

Dilute 1mL of the Stock 8141 Spike standard (100ug/mL) to 5mL with acetone for a final concentration of 20ug/mL. Add 100uL of this working spike to each 8141 LCS, MS and MSD.

10.2.3.11. Working 8141 Surrogate Standard Preparation

Dilute 200uL of the Stock 8141 Surrogate Standard (5000ug/mL) to 10mL with acetone for a final concentration of 100ug/mL. Add 25uL of this working surrogate standard to each 8141 sample, Method Blank, LCS, MS and MSD.

11. Calibration

11.1. Not applicable to this SOP.

12. Procedures

12.1. Configure the microwave extraction unit per the manufacturer's instructions.

12.2. Make sure that all glassware and Teflon tubes used for this procedure have been properly cleaned. All washed glassware and Teflon tubes must be rinsed prior to use with acetone to remove residual water and with methylene chloride to remove any residual contaminants.

12.3. Water and/or foreign objects should be excluded from sample portion weighed for analysis. If the soil sample contains a water layer or foreign objects such as sticks, leaves, rocks, etc., decant water or remove objects and place into an aluminum pan. Sample must be homogenized as much as possible prior to weighing for analysis. Once sample portion is weighed for analysis, add water or foreign objects back into the original sample container.

12.4. Weigh either 30g or 20g sample into a weigh boat or aluminum pan – see Table 1. Quantitatively transfer the sample into a labeled Teflon tube.

12.5. Prepare a method blank by weighing 30g or 20g of simulated soil matrix and transferring it to a labeled Teflon tube – see Table 1. The method blank will be used to detect potential laboratory contamination.

12.6. Prepare an LCS by weighing 30g or 20g of simulated soil matrix and transferring it to a labeled Teflon tube. Spike the matrix in the tube with the appropriate amount of spike solution. The LCS will be used to determine the efficiency of the extraction method in recovering target compounds

12.7. Choose one sample in the batch to use as a matrix spike (MS) and matrix spike duplicate (MSD). Weigh two additional 30g or 20g portions of the sample for the MS and MSD and transfer each portion to labeled Teflon tubes. Spike the soil in each tube with the appropriate amount of spike solution.

12.8. Spike each tube with the appropriate amount of surrogate solution.

12.9. Add 25mL of the appropriate solvent (see Table 1) to each Teflon tube and seal the vessels tightly using a mechanical capper.

12.10. Place all Teflon tubes into the extraction unit and proceed with microwave extraction per manufacturer's instructions. Once established, the same extraction conditions must be used for all method blanks, client samples, and QC samples in the same extraction batch.

12.11. Once the microwave cycle is complete, allow the extracts to cool to room temperature. After cooling, carefully open the Teflon tubes using the mechanical capper and filter each extract through a funnel fitted with filter paper or a glass wool plug and containing sodium sulfate. Collect the filtered extract in a clean, labeled concentrator tube. Rinse extraction vessel with methylene chloride and pour into funnel. Once the extract and rinse is filtered, rinse the sodium sulfate with methylene chloride and collect in the concentrator tube.

12.12. Concentration procedure: Refer to Table 1 for routine final volumes. Place each concentrator tube into the Zymark concentrator apparatus. Adjust the settings per manufacturer's instructions. When the volume of the extract reaches a level slightly below the required final volume, remove the concentrator tube from the apparatus and allow extract to cool.

12.13. If a solvent exchange is required, see Table 1, add 50mL of the exchange solvent to the Zymark tube. Concentrate the extract to slightly below the intended final volume, remove from the water bath and allow it to cool.

12.14. Prepare a calibrated vial by volumetrically dispensing the required volume of the solvent being used into a vial and securely capping the vial to eliminate evaporation. The calibrated vial must be prepared daily. Quantitatively transfer the extract from the Zymark tube or concentrator tube to a vial. Bring the sample extract in the vial to the required final volume listed in Table 1 by visually comparing the sample extract vial volume to the calibrated vial volume. Securely cap the sample extract vial. Store all extracts in the appropriate storage cooler. For extracts that will not concentrate to the usual final volume, use the procedure described above to bring the extract to the next higher practical volume for which a calibrated vial can be prepared.

12.15. Refer to appropriate cleanup SOPs if extract cleanup is required.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Simulated soil matrix	One per preparation batch of up to 20 samples.	Target analytes must be less than reporting limits	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, affected data must be qualified. 3) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples.	Lab-generated limits Refer to LIMS for control limits.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples.	Lab-generated limits Refer to LIMS for control limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogate	Applicable surrogate compound	Added to each sample, standard and method blank	Lab-generated limits Refer to LIMS for control limits.	Samples with surrogate failures must be re-extracted and reanalyzed. QC samples with surrogate failures require the re-extraction and reanalysis of the QC samples and the associated client samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) If only one surrogate for BNA or Combo fails and it is >10% recovery, re-extraction is not required but data must be qualified. 4) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. Demonstration of Capability (DOC): Analysts performing this method must document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

18.2. Microwave Power Test: The microwave should be power tested per manufacturer's instructions monthly, at a minimum.

19. Method Modifications

19.1. Samples not air dried and ground nor are they mixed with sodium sulfate prior to extraction.

19.2. Balance weighs to 0.1g instead of 0.01g because dry weight is not determined by this procedure.

19.3. Samples are not particle sized or sieved prior to extraction but are homogenized.

19.4. Soil samples for 8081 and 8141 are weighed at 20g, not 30g.

20. Instrument/Equipment Maintenance

20.1. Refer to manufacturer's instructions and/or maintenance log.

21. Troubleshooting

21.1. Refer to manufacturer's instructions and/or maintenance log.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. Equipment: Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention and Waste Management

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3546 "Microwave Extraction".

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Table 1: Extraction Conditions

27. Revisions

Document Number	Reason for Change	Date
S-IN-O-130-rev.04	1. Table of Contents: new Section 14, Method Modifications added. 2. Table 8.1: updated balance details 3. Table 8.2: added volumetric pipettes 4. Table 9.1: updated solvent manufacturers 5. Table 9.3: updated for current standards, vendors and concentrations 6. Section 9.2.3: updated for current standards and volumes 7. Section 11.2: added Teflon tubes require washing and rinsing 8. Section 11.8: changed specific solvent to "appropriate solvent" 9. Section 11.11: updated final volumes for methods where needed 10. Section 11.13: added instructions for determination of non-standard final volumes 11. Section 11.14: added reference to cleanup SOPs. 12. Table 12.1: updated corrective action for Method Blank. 13. New Section 14, Method Modifications added. 14. Section 15.1: revised SOP reference. 15. Section 16: added reference to cleanup SOPs.	16May2013
S-IN-O-130-rev.05	1. Cover page: updated phone number 2. Section 3.2: added pesticides. 3. Section 3.3: updated solvents used. 4. Table 7.1: updated holding time for extraction of samples for PCBs to 6 months. 5. Table 9.1: updated reagents and added sodium sulfate rinsing procedure. 6. Table 9.3: updated standards and storage conditions and added standards for pesticides analysis. 7. Section 9.2.3: updated Full-list BNA standard prep and added preparation of pesticide standards. 8. Section 11: added references to the newly created Table 1 showing extraction conditions. 9. Section 12: removed equations for LCS, RSD, and MS. 10. Section 13: removed reference to MDL because it is covered in each determinative procedure SOP. 11. Section 16: removed the specific reference to cleanup SOPs. 12. Section 17: added Table 1 for extraction conditions.	02Sep2015
S-IN-O-130-rev.06	1. Converted to 27 section format. 2. Table 7.1: updated storage temperature format. 3. Section 9.2: added weigh boats/aluminum pans. 4. Table 10.3: updated standard descriptions. 5. Section 10.2.3: updated standard preparation where needed. 6. Section 12: clarified sample amounts and weighing procedure. Separated instructions for method blank and LCS. 7. Section 19: added method modification for reduced 8081 and 8141 weights. 8. Section 25.3: added years 2003 and 2009 to TNI reference. 9. Table 1: added column for routine sample weight by method and added BNA Scan/SIM Combo details.	05Sep2017

Table 1 – Extraction Conditions

Determinative Method	Initial Sample Weight (g)	Clean Soil Matrix Used for QC	Extraction Solvent	Exchange Solvent	Final Extract Volume (mL)
8015 DRO (includes ERO and Ohio mod)	30	Ottawa Sand	Methylene Chloride	N/A	1
8081 OC PEST	20	Sodium Sulfate	3:1 Methylene Chloride to Acetone	Hexane	10
8082 PCB	30	Sodium Sulfate	3:1 Methylene Chloride to Acetone	Hexane	10
8141 OP PEST	20	Sodium Sulfate	3:1 Methylene Chloride to Acetone	Hexane	10
8270 PAH-SIM	30	Ottawa Sand	3:1 Methylene Chloride to Acetone	N/A	1
8270 BNA	30	Ottawa Sand	3:1 Methylene Chloride to Acetone	N/A	1
8270 BNA Scan/SIM Combo	30	Ottawa Sand	3:1 Methylene Chloride to Acetone	N/A	1

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF SEMI-VOLATILE COMPOUNDS BY GC/MS
REFERENCE METHOD: EPA SW-846 METHOD 8270C

SOP NUMBER:	S-IN-O-068-rev.14
EFFECTIVE DATE:	December 14, 2015
SUPERSEDES:	S-IN-O-068-rev.13

APPROVAL

General Manager — December 7, 2015 Date

Quality Manager — December 7, 2015 Date

Department Manager — December 7, 2015 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
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Table of Contents

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of semi-volatile organic compounds in sample extracts while meeting the requirements specified in EPA method 8270C.

2. Summary of Method

2.1. Semi-volatile compounds are introduced into a gas chromatograph by injection of a sample extract onto a narrow-bore capillary column for analysis. The column is temperature programmed to separate the analytes that are then detected with a mass spectrometer. Identification of the analytes is made by comparing their mass spectra with spectra of known standards. Quantitation is accomplished by comparing the response of a major ion relative to the internal standard response using a multi-point calibration curve.

2.2. Method 8270C provides chromatographic conditions for the detection of semi-volatile compounds in organic extracts. Aqueous samples are extracted using SW-846 method 3510 Separatory Funnel Extraction or other applicable method. Solid samples are extracted using SW-846 method 3546 Microwave Extraction or other applicable method.

3. Scope and Application

3.1. This method can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, phenols, and nitrophenols.

3.2. The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent extraction and exhibits poor chromatographic behavior. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosdiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3.3. The following compounds are analyzed using this procedure but are not 8270C listed compounds: Carbazole, Biphenyl (Diphenyl), Caprolactam, Atrazine, Benzaldehyde, Diethyl Aniline, 2,3-Dichloroaniline, 1-Methylnaphthalene and 4-Chlorobenzotrifluoride.

3.4. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.5. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC/MS equipment and the interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This procedure is applicable to extracts prepared from many types of solid waste, soils, air sampling

media, and water samples.

5. Limits of Detection and Quantitation

5.1. The list of compounds and reporting limits analyzed for method 8270C is found in Table 1. Other compounds may be reported upon completion of appropriate validation procedures. Refer to the LIMS for method detection limits.

6. Interferences

6.1. Glassware for the preparatory steps for this method should be thoroughly cleaned and rinsed. Soap products can leave phthalates on the glassware that may appear in the analytical data. Hits for phthalates should be closely scrutinized and continuous hits should warrant checking on the glassware cleaning procedure.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid, preferably 1L or 125mL widemouth, or equivalent.	None required	0°C to 6°C	Must be extracted within 7 days of collection date and analyzed within 40 days of extraction date
Solid	> 200 grams in 4oz or 8oz glass jar	None required	0°C to 6°C	Must be extracted within 14 days of collection date and analyzed within 40 days of extraction date

Samples and sample extracts must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Description / Comments
Gas Chromatographs	Hewlett Packard/Agilent 6890/7890 or equivalent system
Data Systems	Hewlett Packard/Agilent Chemstation or equivalent system
Autosamplers	Hewlett Packard/Agilent or equivalent system
Mass Spectrometers	Hewlett Packard/Agilent 5973/5975. Or equivalent system.

9.2. Chromatography Supplies

Item	Vendor	Model / ID	Description
Analytical Columns	Restek	Rxi-5 Sil MS	30m x 0.25mm or equivalent column

9.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Syringe valves	2-way with Luer ends	Supelco or equivalent
Standard vials	2mL stop/go vials (clear vials)	Supelco or equivalent
Autosampler vials	1.8mL clear vials	Or equivalent

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Methylene Chloride	Pesticide grade or equivalent
Acetone	Pesticide grade or equivalent
Methanol	Pesticide grade or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions and vendors

Standard	Description	Comments
Tuning Standard	Standard used to tune the mass spectrometer	DFTPP solution
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains method required spiking compounds, at a minimum, and is used for spiking MS/MSD sets.	Same solution can be used for the LCS and MS/MSD

10.2.2. Details and Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock 8270 calibration standard A	O2si; catalog #111236-01, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 calibration standard B2	O2si; catalog #111447-1, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 calibration standard C	O2si; catalog #010442-08, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.

Standard Type	Description	Expiration	Storage
Working 8270/Intermediate 8270 LVE calibration standards	Refer to Sections 10.2.3.1	Expires 6 months from date of preparation.	Refrigerate
Working 8270 LVE calibration standards	Refer to Sections 10.2.3.2	Expires 6 months from date of preparation.	Refrigerate
Stock 8270 ICV standards-Phenol	Supelco; catalog #48904, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-BN1	Supelco; catalog #48900-U, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-HS1	Supelco; catalog #48907, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-HS2	Supelco; catalog #48908, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-Lilly ICV	Cresent; catalog #CCS-2579, 1000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-AccuCust	AccuStandard; catalog #S-939R, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-PAH	Supelco; catalog #CRM48905, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-Benzidines	Supelco; catalog #48906, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-BN2	Supelco; catalog #48120-U, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Intermediate 8270 LVE ICV standard	Refer to Section 10.2.3.3	Expires 6 months from date of preparation.	Refrigerate
Working 8270 LVE ICV standard	Refer to Section 10.2.3.4	Same expiration date as Intermediate 8270 LVE ICV standard	Refrigerate
Working 8270/Stock 8270 LVE internal standards	Restek; catalog # 31006; 4000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8270 LVE internal standards	Refer to Section 10.2.3.5	Expires 6 months from date of preparation.	Refrigerate
Stock 8270 LVE Surrogate standards	Supelco; catalog #CRM47262, 5000ug/mL, or equivalent Supelco; catalog #CRM 47261, 10000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock/Working 8270 DFTPP Tuning Standard	Supelco; catalog #CRM47387, 50ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8270 DFTPP LVE Tuning Standard	Refer to Section 10.2.3.6	Expires 6 months from date of preparation.	Refrigerate

10.2.3. Standard Preparation Procedures

10.2.3.1 Working 8270/Intermediate 8270 LVE Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Standard	Stock Cal. Std. A Amount	Stock Cal. Std. B2 Amount	Stock Cal. Std. C Amount	Stock Surrogate Std Added	Final Volume in Methylene Chloride	Final Nominal Cal Std Concentration	Stock Internal Standard amount added to Cal. Std.	Internal Standard Concentration
8270 Cal Std 1	5uL	2.5uL	2.5uL	5uL	1000uL	5ug/mL	10uL	40ug/mL
8270 Cal Std 2	10uL	5uL	5uL	10uL	1000uL	10ug/mL	10uL	40ug/mL
8270 Cal Std 3	20uL	10uL	10uL	20uL	1000uL	20ug/mL	10uL	40ug/mL
8270 Cal Std 4	50uL	25uL	25uL	50uL	1000uL	50ug/mL	10uL	40ug/mL
8270 Cal Std 5	80uL	40uL	40uL	80uL	1000uL	80ug/mL	10uL	40ug/mL
8270 Cal Std 6	100uL	50uL	50uL	100uL	1000uL	100ug/mL	10uL	40ug/mL
8270 Cal Std 7	150uL	75uL	75uL	150uL	1000uL	150ug/mL	10uL	40ug/mL

10.2.3.2 Working 8270 LVE Calibration Standards

Prepare using the Working 8270/Intermediate 8270 LVE Calibration Standards from Section 10.2.3.1. The following are examples of calibration standards and could vary based on requirements:

Standard	Intermediate Cal. Std. ID from Section 9.2.3.1	Intermediate Cal. Std. Amount	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Internal Standard Concentration
8270 LVE Cal. Std 1	8270 Calibration Std 1	50uL	500uL	0.5ug/mL	4ug/mL
8270 LVE Cal. Std 2	8270 Calibration Std 2	50uL	500uL	1.0ug/mL	4ug/mL
8270 LVE Cal. Std 3	8270 Calibration Std 3	50uL	500uL	2.0ug/mL	4ug/mL
8270 LVE Cal. Std 4	8270 Calibration Std 4	50uL	500uL	5.0ug/mL	4ug/mL
8270 LVE Cal. Std 5	8270 Calibration Std 5	50uL	500uL	8.0ug/mL	4ug/mL
8270 LVE Cal. Std 6	8270 Calibration Std 6	50uL	500uL	10ug/mL	4ug/mL
8270 LVE Cal. Std 7	8270 Calibration Std 7	50uL	500uL	15ug/mL	4ug/mL

10.2.3.3 Intermediate 8270 LVE ICV Standard

Combine the following:

25uL of Stock 8270 LVE - Phenol (2000ug/mL),
25uL of Stock 8270 LVE - BN1 (2000ug/mL),
25uL of Stock 8270 LVE - HS1 (2000ug/mL),
25uL of Stock 8270 LVE - HS2 (2000ug/mL),
25uL of Stock 8270 LVE - Lilly ICV (1000ug/mL),
50uL Stock 8270 LVE - AccuCust (2000ug/mL),
25uL of Stock 8270 LVE - PAH (2000ug/mL),
25uL of Stock 8270 LVE - Benzidines (2000ug/mL), and
25uL Stock 8270 LVE - BN2 (2000ug/mL)

Bring to a final volume of 1mL in Methylene Chloride for a final concentration o 50ug/mL.

10.2.3.4 Working 8270 LVE ICV Standard

Dilute 100ul of the Intermediate 8270 LVE ICV Standard (50ug/mL) to 500uL with methylene chloride for a final concentration of 10ug/mL.

10.2.3.5 Working 8270 LVE Internal Standards

Dilute 500uL of the Stock 8270 LVE internal standards (4000ug/mL) to 5mL with methylene chloride for a final concentration of 400ug/mL.

10.2.3.6 Working DFTPP LVE Tuning Standard

Dilute 50uL of the Stock DFTPP Tuning Standard (50ug/mL) to 500uL with methylene chloride for a final DFTPP concentration of 5ng/uL.

11 Calibration

11.2 DFTPP Tune Verification: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer must be hardware tuned using a 50ng injection of DFTPP. Analysis must not begin until the tuning criteria are met. Use the DFTPP mass intensity criteria in the table below as tuning acceptance criteria. Alternate tuning criteria may be used provided that method performance is not adversely affected. The 12-hour window during which standards and samples may be analyzed begins with the injection of DFTPP. All subsequent standards, samples MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

Mass (m/z)	Ion Abundance criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

If the DFTPP ratios do not meet the criteria, reanalyze the DFTPP tune. If the DFTPP still fails the criteria, autotune adjustment, instrument maintenance, and/or preparation of new standards must be considered.

The mass spectrum of DFTPP may be obtained by averaging three scans, the peak apex scan and the scans immediately preceding and following the apex. Background subtraction is required using this approach and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Do not background subtract part of the DFTPP peak. Alternatively, the analyst may use other approaches suggested below:

1. A single scan within the DFTPP peak with background subtraction of a single scan no more than 20 scans prior to the elution of DFTPP.
2. An average of multiple scans within the DFTPP peak with background subtraction of a single scan no more than 20 scans prior to the elution of DFTPP.

11.3 Initial Calibration: Initial Calibration standards are introduced into the GC/MS from the lowest to highest concentration of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
A_{IS} = Area of the quantitation ion for the internal standard.
C_{IS} = Concentration of the internal standard
C_x = Concentration of the compound being measured.

11.4 The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

11.5 The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of average RF for a compound
RF_{avg} = Mean of RFs for a compound

11.6 The %RSD should be should be $\leq$15% for each target analyte. However, the %RSD for each individual Calibration Check Compound (CCC) must be $\leq$30%. If the RSD of any CCC is >30%, then the chromatographic system is too reactive and instrument maintenance or preparation of new standards may be necessary before attempting recalibration. The CCCs are:

Base/Neutral Fraction	**Acid Fraction**
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

11.7 System Performance Check Compounds (SPCCs) are checked for a minimum average response factor (RF_{avg}) to determine potential instability and/or degradation caused by deterioration of instrument conditions or standard material. The minimum RF_{avg} for the semivolatile SPCCs are as follows:

N-nitroso-di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Instrument maintenance or preparation of new standards may be necessary. The SPCC criteria must be met for sample analysis to begin.

11.8 If the percent relative standard deviation (%RSD) of the RFs for a compound is $\leq$15% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

11.9 If the % RSD for any compound is >15%, the analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.10 Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.11 **Initial Calibration Corrective Action:** If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.12 Each day that analysis is performed, the calibration standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably. Additionally, examination of signal-to-noise ratio of analytes in low-level standards may be a useful diagnostic tool to monitor instrument performance. Signal-to-noise ratio is defined as the ratio of the analyte signal to the noise measured on a blank. It is recommended but not required that the signal-to-noise ratio be at least three to confirm detection.

11.13 **Initial Calibration Verification (ICV):** In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent recovery (%Rec) of the observed ICV according to the following equation:

$$\% \text{ Recovery} = \frac{\text{Observed concentration}}{\text{Theoretical concentration}} \times 100$$

The ICV is analyzed immediately following the initial calibration curve. The ICV recoveries are evaluated against a default acceptance range of 70-130% recovery. Alternative acceptance limits may be appropriate for some compounds.

11.14 ICV Corrective Action: If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

11.15 Continuing Calibration Verification: The initial calibration is verified every 12 hours by analyzing a DFTPP tune verification as described in Section 10.1, followed by a Continuing Calibration Verification (CCV) standard.

11.16 If the % difference (%D) or % Drift for each CCC is $\leq$20%, then the initial calibration is assumed to be valid. The response factors for all SPCCs in the CCV standard must meet the criteria in Section 10.6. The % difference (%D) or % Drift for each non-CCC compound should be <40%. If non-CCC compounds fail to meet this criteria, the concentrations above the reporting limit in associated samples must be qualified as estimated.

11.17 The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

11.18 CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

12 Procedures

12.1 All sample extracts must be analyzed at room temperature and the system must be tuned and calibrated as per Section 10, and free of contamination before samples are analyzed.

12.2 Gas Chromatography conditions: Configure the GC/MS per manufacturer's instructions.

12.3 The 1mL extract obtained from sample preparation for 8270 should be fortified with 10uL of the Stock 8270 internal standard (4000ug/mL) just prior to analysis such that 40ng of internal standard is injected onto the column. The 1mL extract obtained from sample preparation for 8270 LVE should be fortified with 10uL of the Working 8270 LVE internal standard (400ug/mL) just prior to analysis such that 4ng of internal standard is injected onto the column. Analyze each 8270 extract by injecting 2uL onto the column. Analyze each 8270 LVE extract by injecting 10uL onto the column.

12.4 Qualitative Analysis: Compounds are identified as present when the following criteria are met:

12.4.1 The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.
12.4.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Refer to Table 2 for characteristic ions.
12.4.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.5 **Quantitative analysis:** Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique. Calculations are subject to change based on the data reduction software used. Extract concentrations that exceed the upper calibration range must be diluted and reanalyzed or qualified as estimated. Additional internal standard must be added to the diluted extract to maintain the same concentration as the calibration standards.

12.6 Refer to the Manual Integrations SOP for guidance on performing and documenting manual integrations.

12.7 If the sample concentration exceeds the linear range of the analysis, the sample must be diluted and reanalyzed or reported as an estimated concentration.

12.8 Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_t)(D)}{(V_i)(V_s)} \qquad \text{Solid Sample (ug/Kg)} = \frac{(X_s)(V_t)(D)}{(V_i)(W_s)}$$

Where:
X_s = On-column concentration of the analyte in the sample aliquot injected
V_t = Total volume of concentrated extract
D = Dilution factor
V_i = Volume of the extract injected in uL
V_s = Volume of aqueous sample extracted in milliliters
W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100- \%Moisture)}} \times 100$$

13 Quality Control

13.1 Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 3) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits. Refer to Sections 12.2 and 12.3 for additional information.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogates	Applicable surrogate compounds	Added to each standard, sample, and method blank.	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) If only one surrogate fails and it is >10% recovery, re-extraction is not required but data must be qualified. 4) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.
As required by client only: **Internal Standards**	Applicable Internal Standard compounds	Added to each standard, sample, and method blank.	Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV.	Samples with internal standard failures must be reanalyzed undiluted or more concentrated. The laboratory may only dilute a sample prior to reanalysis if matrix interference is present. *Exception:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified.

13.2 The matrix spike may be used in place of the LCS as long as the acceptance criteria are as stringent as for the LCS.

13.3 **Allowable Marginal Exceedances**: If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. A marginal exceedance (ME) is defined as being beyond the LCS control limit of +/-3 standard deviations, but within the ME limits of +/-4 standard deviations around the mean. The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and correction action is necessary. If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken. The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

NOTE: As allowed by client, the LCS shall be allowed to be outside the control limits but ≥10% for hexachlorocyclopentadiene, N-nitrosodimethylamine, pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, and 4-chloroaniline without corrective action. The LCS shall be allowed to be outside the control limits but >10% for up to four additional compounds, with the exception of any PAH, without corrective action.

13.4 LCS equation

$$R = (C/S) * 100$$

Where R = percent recovery
C = observed LCS concentration
S = concentration of analyte added to the clean matrix

13.5 MS/MSD equation

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = observed spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

13.6 RPD calculations:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

14 Data Analysis and Calculations

14.1 Refer to Section 12.

15 Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 Refer to Sections 11 and 13.

16 Corrective Actions for Out-of-Control Data

16.1 Refer to Sections 11 and 13.

17 Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 Refer to Sections 11 and 13.

18 Method Performance

18.1 An MDL study and/or LOD/LOQ verification must be conducted annually for each matrix per instrument.

18.2 Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19 Method Modifications

19.1 Standards are purchased as certified stock solutions and not prepared from neat materials.

19.2 Microwave Method 3546 is used for the preparation of solid samples for analysis by 8270C.

19.3 Phenol-d5 is used as a surrogate instead of Phenol-d6.

19.4 Extract final volumes, volume of internal standards added to extracts, and volume of extract injected into the instrument may vary from those identified in Method 8270C.

20 Instrument/Equipment Maintenance

20.1 Refer to instrument maintenance logs and/or manufacturer's instructions.

21 Troubleshooting

21.1 Refer to instrument maintenance logs and/or manufacturer's instructions.

22 Safety

22.1 **Standards and Reagents:** The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. The use of gloves, lab coats and safety glasses is required. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock semi-volatile standards are toxic and should be handled with extreme care.

22.2 **Samples:** Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves, lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is

recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

23 Waste Management

23.1 Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

23.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24 Pollution Prevention

24.1 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25 References

25.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846 Methods 8000C and 8270C.

25.2 Pace Analytical Quality Manual; latest revision.

25.3 TNI Standard; Quality Systems section; latest revision.

26 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1 Table 1: Target Compounds and Reporting Limits

26.2 Table 2: Characteristic Ions of Target Compounds

27 Revisions

Document Number	Reason for Change	Date
S-IN-O-068-rev.11	1. Cover page revised to reflect periodic review format. 2. Table of Contents fixed to include Sections 10 and 11. 3. Section 2: removed reference to Method 3550-Ultrasonic Extraction. 4. Section 3: added language that limits, volumes, etc… are subject to change. 5. Table 7.1: removed TCLP and revised storage conditions. 6. Section 8: revised equipment and supplies where necessary and added "or equivalent" where applicable. 7. Section 9: revised and added "or equivalent" where applicable. 8. Section 10: condensed tuning information and added reference to QAM for ICAL details. Added more detailed equations. 9. Section 11: removed all references to evaluation of the tune mixture for tailing and degradation. Removed instrument conditions tables. Added the calculation for moisture correction. Removed information regarding TICs. 10. Table 12.1: added 1/10 criteria for method blank and reference to Sections 12.1.1 and 12.1.2 for marginal exceedances. 11. Sections 12.1.1 and 12.1.2: added to clarify LCS criteria per NELAC and BP. 12. Section 16: removed references to Tables 3 and 4. 13. Table 2: added internal standard and surrogates to table.	20April2011

S-IN-O-068-rev.12	1. Table of Contents: New Section 14, Method Modifications added. 2. Section 3.1: added a reference to MDLs. 3. Table 7.1: clarified bottle types for both versions of the water extraction. 4. Table 9.3: added detail for the 8270 LVE standards 5. Section 9.2.3: added detail for the 8270 LVE standards. 6. Sections 10.12 and 10.13: revised ICV language to reflect actual practice. 7. Section 11.3: added detail for internal standard addition and analysis of 8270 LVE extracts. 8. Section 11.5: added language for diluting over range samples 9. Sections 11.6-11.8: replaced 8000C equations with Target equations. 10. Table 12.1: revised corrective action for Method Blank. 11. New Section 14 added for Method Modifications 12. Section 15.1: revised SOP reference. 13. Section 16.3: changed NELAC to TNI 14. Attachments Table 1: 1-Methylnaphthalene added. 15. Attachments Table 2: 1-Methylnaphthalene added, Phenol-d6 changed to Phenol-d5.	07May2013
S-IN-O-068-rev.13	1. Cover page: added actual effective date. 2. Sections 10.8 and 10.9: removed SW-846 equations and made reference to Method 8000C. 3. Section 10.11: added as guidance for evaluation of ICAL standards. 4. Section 10.12: added that alternative limits may be appropriate for some ICV compounds. 5. Section 10.15: added criteria of <40% for non-CCC compounds in CCV. 6. Section 11.6: added as a reference to the Manual Integrations SOP. 7. Section 11.7: added to require over range samples be diluted and reanalyzed or qualified as estimated. 8. Sections 11.8-11.10: replaced Target equations with SW-846 equations. 9. Table 12.1: corrected references in LCS acceptance criteria section and removed client-specific reference in internal standard section. 10. Section 12.3 Note: removed client-specific reference. 11. Section 13.1: added optional LOD/LOQ verification. 12. Table 1: updated some soil RLs.	01Nov2013
S-IN-O-068-rev.14	1. Converted to SOT format with 27 sections. 2. Cover page: changed phone number and revised document control format. 3. Section 3: added a list of compounds that are analyzed but not listed in the method. 4. Section 9.2: updated column details 5. Table 10.3: updated standard details and storage conditions. 6. Section 10.2.3: updated several standard preparation procedures. 7. Section 12: removed calculations for curve fit types. 8. Updated Tables 1 and 2.	07Dec2015

Table 1: Target Compounds and Reporting Limits[1]

Analyte	RL water (ug/L)	RL soil (ug/kg)
Phenol	10	330
Bis (2-chloroethyl) ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
Benzyl Alcohol	20	660
1,2-Dichlorobenzene	10	330
2-Methylphenol (o-Cresol)	10	330
Bis (2-chloroisopropyl)ether	5	330
3&4-Methylphenol (m&p-Cresol)	20	660
N-Nitroso-di-n-propylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
Benzoic Acid	50	1600
Bis(2-chloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	5	330
4-Chloroaniline	20	660
Hexachlorobutadiene	5	330
4-Chloro-3-methylphenol	20	660
1-Methylnaphthalene	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	10	330
2-Chloronaphthalene	10	330
2-Nitroaniline	50	1600
Dimethyl phthalate	10	330
Acenaphthene	10	330
Acenaphthylene	10	330
2,4-Dinitrophenol	50	1600
4-Nitrophenol	50	1600
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
2,6-Dinitrotoluene	10	330
3-Nitroaniline	50	1600
Diethyl phthalate	10	330
4-Chlorophenyl phenyl ether	10	330
Fluorene	10	330
4-Nitroaniline	50	1600
4,6-Dinitro-2-methylphenol	50	1600
N-Nitrosodiphenylamine	10	330
4-Bromophenyl phenyl ether	10	330

Analyte	RL water (ug/L)	RL soil (ug/kg)
Hexachlorobenzene	10	330
Pentachlorophenol	50	1600
Phenanthrene	10	330
Anthracene	10	330
Di-n-butyl phthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butyl benzyl phthalate	10	330
3,3'-Dichlorobenzidine	20	660
Benzo(a)anthracene	10	330
Chrysene	10	330
Bis(2-ethylhexyl)phthalate	5	330
Di-n-octyl phthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	170
Indeno(1,2,3-cd)pyrene	10	330
Dibenz(a,h)anthracene	10	170
Benzo(g,h,i)perylene	10	330
N-Nitrosodimethylamine	10	330
Pyridine	10	1600
Benzidine	20	330
Acetophenone	10	330
2,6-Dichlorophenol	10	330
1,2-Diphenylhydrazine	10	330
2-Picoline	50	1600
1,2,4,5-Tetrachlorobenzene	10	330
1,3-Dinitrobezene	50	1600
2,3,4,6-Tetrachlorophenol	10	330

[1] Target Compounds and Reporting Limits are subject to change.

Table 2: Characteristic Ions of Target Compounds[2]

Analyte	Primary Ion	Secondary Ion(s)
Group 1 - 1,4-Dichlorobenzene-d4 (IS)	152	150, 115
N-Nitrosodimethylamine	42	74,44
Pyridine	79	52
2-Picoline	93	66, 92
4-Chlorobenzotrifluoride[3]	180	161,182
2-Fluorophenol (S)	112	64
Phenol-d5 (S)	99	71, 42
Benzaldehyde	77	105, 106
Phenol	94	65.66
Aniline	93	66, 65
Bis(2-Chloroethyl) ether	93	63.95
2-Chlorophenol	128	64,130
n-Decane[3]	57	43, 142
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
Benzyl alcohol	108	79, 77
1,2-Dichlorobenzene	146	148, 111
Bis(2chloro1methylethyl) ether[3]	45	77, 121
Bis(2-chloroisopropyl) ether	45	77, 121
3&4-methylphenol (m&p cresol)	108	107,77
Acetophenone	105	77, 120
N-Nitroso-di-n-propylamine	70	130, 101
Hexachloroethane	117	201, 199
Group 2 - Naphthalene-d8 (IS)	136	68
Nitrobenzene-d5 (S)	82	128, 54
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	109, 65
2,4-Dimethylphenol	122	107, 121
Bis(2-chloroethoxy) methane	93	95, 123
Benzoic Acid	105	122, 77
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 102
Apha-Terpineol	59	93, 121
2-Chloroaniline	127	65, 92
Hexachlorobutadiene	225	223, 227
Caprolactam	113	55, 56
Diethyl Aniline[3]	134	106, 77
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141, 115
1-Methylnaphthalene[3]	142	141, 115

Analyte	Primary Ion	Secondary Ion(s)
Group 3 - Acenaphthene-d10 (IS)	164	160, 162
Hexachlorocyclopetadiene	237	235, 272
1,2,4,5-Tetrachlorobenzene	216	179, 108
2,3-Dichloroaniline[3]	161	163, 90
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Fluorobiphenyl (S)	172	171
2-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
1,3-Dinitrobenzene	168	76, 50
2,6-Dinitrotoluene	165	63, 89
Acenaphthylene	152	150, 153
3-Nitroaniline	138	108, 92
Biphenyl (Diphenyl)	154	153, 152
Acenaphthene	153	154, 152
2,4-Dinitrophenol	184	154, 63
4-Nitrophenol	109	139, 65
2,4-Dinitrotoluene	165	63, 89
Dibenzofuran	168	139, 169
2,3,4,6-Tetrachlorophenol	232	131, 230
Diethylphthalate	149	177, 150
4-Chloropheyl-phenylether	204	206, 141
Fluroene	166	165, 139
4-Nitroaniline	138	108, 65
Group 4 - Phenanthrene-d10 (IS)	188	80,94
4,6-Dinitro-2-methylphenol	198	51,105
N-Nitrosodiphenylamine	169	168, 167
Azobenzene[3]	77	182, 105
1,2-Diphenylhydrazine	77	105, 182
2,4,6-Tribromophenol (S)	330	332, 141
4-Bromophenyl-phenyl ether	248	250, 141
Hexachlorobenzene	284	142, 249
Atrazine	200	215, 202
Pentachlorophenol	266	264, 268
n-Octadecane[3]	57	43, 71
Phenanthrene	178	179, 176
Anthracene	178	176, 179
Carbazole[3]	167	166, 139
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 203
Benzidine	184	92, 185
Group 5 - Chysene-d12 (IS)	240	120, 236
Pyrene	202	101, 203
p-Terphenyl-d14 (S)	244	122, 212
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Bis(2-Ethylhexyl) phthalate	149	167, 279
Benzo(a)athracene	228	229, 226
Chrysene	228	226, 229

Analyte	Primary Ion	Secondary Ion(s)
Group 6 - Perylene-d12 (IS)	264	260, 265
Di-n-ocytlphthalate	149	279, 43
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluroanthene	252	253, 125
Benzo(a)pyrene	252	253,125
Indeno (1,2,3-cd)pyrene	276	138, 277
Dibenz (a,h) athracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277

[2] Target Compounds are subject to change.
[3] Compound is not listed in Method 8270C.

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF SEMI-VOLATILE ORGANICS BY GC/MS SELECTED ION MONITORING (SIM)

REFERENCE METHOD: EPA SW-846 METHOD 8270C

SOP NUMBER:	S-IN-O-133-rev.05
EFFECTIVE DATE:	November 2, 2015
SUPERSEDES:	S-IN-O-133-rev.04

APPROVAL

General Manager — October 28, 2015 Date

Quality Manager — October 28, 2015 Date

Department Manager — October 27, 2015 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of semi-volatile organic compounds in sample extracts while meeting the requirements specified in EPA method 8270C, through selected ion monitoring (SIM).

2. Summary of Method

2.1. Semi-volatile compounds are introduced into a gas chromatograph by injection of a sample extract onto a narrow-bore capillary column for analysis. The column is temperature programmed to separate the analytes that are then detected with a mass spectrometer. Identification of the analytes is made by comparing their mass spectra with spectra of known standards. Quantitation is accomplished by comparing the response of a major ion relative to the internal standard response using a multi-point calibration curve. Selected ion monitoring (SIM) is the mode of scanning for this SOP.

2.2. Method 8270C SIM provides chromatographic conditions for the detection of semi-volatile compounds in organic extracts of groundwater, surface water, soil and sediment. Aqueous samples are extracted by using SW-846 method 3510 Separatory Funnel Extraction or other applicable method. Solid samples are extracted using SW-846 method 3546 Microwave Extraction or other applicable method.

3. Scope and Application

3.1. The full list of compounds and reporting limits analyzed by this laboratory for method 8270C SIM, is found in Table 1. Other analytes may be analyzed by this method but must have quality control documentation to support the performance of this method for those analytes. Refer to LIMS for method detection limits.

3.2. Reporting limits, control limits, volumes used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC/MS equipment and the interpretation of GC/MS SIM data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. Glassware for the preparatory steps for this method should be thoroughly cleaned and rinsed. Soap products can leave phthalates on the glassware that may interfere chromatographically.

5. Safety

5.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. The use of gloves, lab coats and safety glasses is required. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock semi-volatile standards are toxic and should be handled with extreme care.

5.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

6. Definitions

6.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber glass container with Teflon-lined lid, preferably 1L or 100mL widemouth.	None required	0°C to 6°C	Sample must be extracted within 7 days of collection date and extract must be analyzed within 40 days of extraction date.
Solid	200 grams in 4oz glass jar	None required	0°C to 6°C	Must be extracted within 14 days of collection date and analyzed within 40 days of extraction date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples. Sample extracts must be stored in the refrigerator or freezer until analysis is complete.

8. Equipment and Supplies

8.1. Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatographs	Hewlett Packard	Lab uses models 6890 and 7890	Or equivalent system
Data Systems	Hewlett Packard		Or equivalent system
Autosamplers	Hewlett Packard		Or equivalent system
Mass Spectrometers	Hewlett Packard	Lab uses models 5973 and 5975	Must be capable of operating in **SIM** mode. Or equivalent system.

8.2. Chromatography Supplies

Item	Vendor	Model / ID	Description
Analytical Columns	Restek	Rtx-5MS	15m x 0.25mm, or equivalent column

8.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Syringe valves	2-way with Luer ends	Supelco or equivalent
Standard vials	2mL stop/go vials (clear vials)	Supelco or equivalent
Autosampler vials	1.8mL clear vials	Or equivalent

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Methylene Chloride	Pesticide grade or equivalent
Methanol	Pesticide grade or equivalent

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains all target analytes and is used to prepare MS/MSD sets.	Same solution can be used for the LCS and MS/MSD

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
PAH-SIM Standards			
Stock PAH-SIM calibration standard	Restek; catalog # 31622; 2000ug/mL or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Intermediate PAH-SIM calibration standards 1 and 2	Refer to Sections 9.2.3.1 and 9.2.3.2.	Solution good for 6 months from preparation	Refrigerate
Working PAH-SIM calibration standard	Refer to Section 9.2.3.3.	Solution good for 6 months from preparation	Refrigerate
Stock PAH-SIM ICV standard w/surrogates	O2Si ; catalog #20-2590; 100ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PAH-SIM ICV standard w/surrogates	Refer to Section 9.2.3.4.	Solution good for 6 months from preparation	Refrigerate
Stock PAH-SIM Surrogate standard	Restek; catalog #31062, 5000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock PAH-SIM Internal standard	Restek; catalog #31006, 4000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PAH-SIM Internal standard	Refer to Section 9.2.3.5.	Solution good for 6 months from preparation	Refrigerate

PAH-SIM Low Volume Extraction (LVE) Standards			
Working PAH-SIM LVE calibration standards	Refer to Section 9.2.3.6	Solution good for 6 months from preparation	Refrigerate
Working PAH-SIM LVE ICV standard w/surrogates	Refer to Section 9.2.3.7	Solution good for 6 months from preparation	Refrigerate
Working PAH-SIM LVE Internal Standard	Refer to Section 9.2.3.8	Solution good for 6 months from preparation	Refrigerate

9.2.3. Standard Preparation Procedures

9.2.3.1 Intermediate PAH-SIM Calibration Standard 1

Dilute 100uL of the Stock PAH-SIM Calibration Standard (2000ug/mL) plus 40uL of the Stock PAH-SIM Surrogate Standard (5000ug/mL) to 1mL with methylene chloride for a final concentration of 200ug/mL.

9.2.3.2 Intermediate PAH-SIM Calibration Standard 2

Dilute 25uL of the Intermediate PAH-SIM Calibration Standard 1 to 1mL with methylene chloride for a final concentration of 5ug/mL.

9.2.3.3 Working PAH-SIM Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Standard	Intermediate Standard amount (1 or 2)	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Working Internal Standard amount added to Cal. Std.	Internal Standard Concentration
Calibration Std 1	10uL (Int 2)	1mL	0.05ug/mL	10uL	5ug/mL
Calibration Std 2	20uL (Int 2)	1mL	0.1ug/mL	10uL	5ug/mL
Calibration Std 3	100uL (Int 2)	1mL	0.5ug/mL	10uL	5ug/mL
Calibration Std 4	200uL (Int 2)	1mL	1ug/mL	10uL	5ug/mL
Calibration Std 5	25uL (Int 1)	1mL	5ug/mL	10uL	5ug/mL
Calibration Std 6	50uL (Int 1)	1mL	10ug/mL	10uL	5ug/mL
Calibration Std 7	100uL (Int 1)	1mL	20ug/mL	10uL	5ug/mL
Calibration Std 8	250uL (Int 1)	1mL	50ug/mL	10uL	5ug/mL

9.2.3.4 Working PAH-SIM ICV Standard with Surrogates

Dilute 10uL of the Stock ICV standard (100ug/mL) to 1mL with methylene chloride for a final concentration of 10ug/mL.

9.2.3.5 Working PAH-SIM Internal Standard

Dilute 375uL of the Stock Internal Standard (4000ug/mL) to3mL with methylene chloride for a final concentration of 500ug/mL.

9.2.3.6 Working PAH-SIM LVE Calibration Standards

The Working PAH-SIM LVE Calibration Standards are prepared from the Working PAH-SIM Calibration Standards from Section 9.2.3.3. The following are examples of calibration standards and could vary based on requirements:

Standard	Amount of Working PAH-SIM Calibration Standard from Sec. 9.2.3.3	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Intermediate Internal Standard amount added to Cal. Std.	Internal Standard Concentration
LVE Cal. Std 1	100uL Cal Std. 1	1mL	0.005ug/mL	10uL	0.5ug/mL
LVE Cal. Std 2	100uL Cal Std. 2	1mL	0.01ug/mL	10uL	0.5ug/mL
LVE Cal. Std 3	100uL Cal Std. 3	1mL	0.05ug/mL	10uL	0.5ug/mL
LVE Cal. Std 4	100uL Cal Std. 4	1mL	0.1ug/mL	10uL	0.5ug/mL
LVE Cal. Std 5	100uL Cal Std. 5	1mL	0.5ug/mL	10uL	0.5ug/mL
LVE Cal. Std 6	100uL Cal Std. 6	1mL	1.0ug/mL	10uL	0.5ug/mL
LVE Cal. Std 7	100uL Cal Std. 7	1mL	2.0ug/mL	10uL	0.5ug/mL
LVE Cal. Std 8	100uL Cal Std. 8	1mL	5.0ug/mL	10uL	0.5ug/mL
LVE Cal. Std 9	100uL Cal Std. 9	1mL	10.0ug/mL	10uL	0.5ug/mL

9.2.3.7 Working PAH-SIM LVE ICV Standard with Surrogates

Dilute 100uL of the Working PAH-SIM ICV Standard with Surrogates (10ug/mL) to 1mL with methylene chloride for a final concentration of 1.0ug/mL.

9.2.3.8 Working PAH-SIM LVE Internal Standard

Dilute 37.5uL of the Stock Internal Standard (4000ug/mL) to3mL with methylene chloride for a final concentration of 50ug/mL.

10. Calibration

10.1. The instrument is auto-tuned periodically using PFTBA according to manufacturer's instructions to maximize detector sensitivity. DFTPP tuning is not performed for this method. Verification of hardware tuning is accomplished through the analysis of initial and continuing calibration standards in which the known ions are selected for data acquisition.

10.2. **Initial Calibration:** Initial Calibration standards are introduced into the GC from the lowest to highest concentration by direct injection of 1uL of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. The 12 hour clock during which standards and samples may be analyzed begins with the injection of the first calibration standard. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
A_{IS} = Area of the quantitation ion for the internal standard.
C_{IS} = Concentration of the internal standard
C_x = Concentration of the compound being measured.

10.3. The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

10.4. The percent relative standard deviation (%RSD) of the response factors is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of the RFs for a compound
RF_{avg} = Mean of RFs for a compound

10.5. All reported PAH compounds in the calibration must be evaluated as CCC compounds. The %RSD of the RFs for each compound must be $\leq$30%.

10.6. All reported PAH compounds in the calibration must be evaluated as SPCC compounds. Each compound must meet a minimum average response factor of 0.050.

10.7. If the %RSD of the RFs for a compound is $\leq$15% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

10.8. If the %RSD of the RFs for any compound is >15%, the analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

10.9. **Initial Calibration Corrective Action:** If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards should also be considered. Samples associated with a failed initial calibration must be reanalyzed.

10.10. Each day that analysis is performed, the calibration standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably. Additionally, examination of signal-to-noise ratio of analytes in low-level standards may be a useful diagnostic tool to monitor instrument performance. Signal-to-noise ratio is defined as the ratio of the analyte signal to the noise measured on a blank. It is recommended but not required that the signal-to-noise ratio be at least three to confirm detection.

10.11. **Initial Calibration Verification (ICV):** In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent recovery (%Rec) of the observed ICV according to the following equation:

$$\% \text{ Recovery} = \frac{\text{Observed concentration}}{\text{Theoretical concentration}} \times 100$$

The ICV is analyzed immediately following the initial calibration curve. The ICV recoveries are evaluated against a default acceptance range of 70-130% recovery. Alternative acceptance limits may be appropriate for some compounds.

10.12. ICV Corrective Action: If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

10.13. Continuing Calibration Verification: The initial calibration is verified every 12 hours by analyzing a Continuing Calibration Verification (CCV) standard. The CCV is prepared using the same standard solution used for the initial calibration. The 12 hour clock during which standards and samples may be analyzed begins with the injection of the CCV.

10.14. If the % Difference (%D) or % Drift for each reported PAH compound is $\leq$20%, then the initial calibration is assumed to be valid. Each reported PAH compound must meet the SPCC criteria in Section 10.6. All reported PAH compounds are considered CCCs and SPCCs for the evaluation of the CCV.

$$\% \text{ Difference} = [RF_v - RF_{avg}] / RF_{avg} * 100$$

10.15. The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

10.16. CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11. Procedures

11.1. All sample extracts must be analyzed at room temperature and the system must be calibrated and free of contamination before samples are analyzed.

11.2. **Gas Chromatography conditions**: Configure the GC/MS per manufacturer's instructions.

11.3. The 1mL extract obtained from the sample preparation laboratory should be fortified with 10uL of the Working PAH-SIM or PAH-SIM LVE Internal Standard solution just prior to analysis, resulting in an internal standard concentration of 5ug/mL for PAH-SIM or 0.5ug/mL for PAH-SIM LVE. Analyze each extract by injecting 1uL onto the column.

11.4. **Qualitative Analysis:** Compounds are identified as present when the following criteria are met:

11.4.1 The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.

11.4.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. See Table 2 for primary and secondary ions.

11.4.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

11.5. **Quantitative analysis:** Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique. See Sections 11.6, 11.7, and 11.8 for equations to calculate the amount or analyte or surrogate introduced into the instrument. Calculations are subject to change based on the data reduction software used.

11.6. When compound concentrations exceed the calibration range, the sample extract should be rerun at a dilution or reported as an estimated concentration.

11.7. Refer to the Manual Integrations SOP for guidance on performing and documenting manual integrations.

11.8. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_t)(D)}{(V_i)(V_s)} \qquad \text{Solid Sample (ug/Kg)} = \frac{(X_s)(V_t)(D)}{(V_i)(W_s)}$$

Where: X_s = On-column concentration of the analyte in the sample aliquot injected
V_t = Total volume of concentrated extract
D = Dilution factor
V_i = Volume of the extract injected in uL
V_s = Volume of aqueous sample extracted in milliliters
W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$$

12. Quality Control

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 3) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits. Refer to Sections 12.1.1 and 12.1.2 for additional information.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogates	Applicable surrogate compounds	Added to each standard, sample, and method blank.	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.
As required by client only: **Internal Standards**	Applicable Internal Standard compounds	Added to each standard, sample, and method blank.	Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV.	Samples with internal standard failures must be reanalyzed undiluted or more concentrated. The laboratory may only dilute a sample prior to reanalysis if matrix interference is present. *Exception:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified.

12.1.1 The matrix spike may be used in place of the LCS as long as the acceptance criteria are as stringent as for the LCS.

12.1.2 **Allowable Marginal Exceedances**: If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. A marginal exceedance (ME) is defined as being beyond the LCS control limit of +/-3 standard deviations, but within the ME limits of +/-4 standard deviations around the mean. The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and correction action is necessary. If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken.

The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

12.2 LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.3 MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4 RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13 Method Performance

13.1 An MDL study and/or LOD/LOQ verification must be conducted annually per matrix per instrument.

13.2 Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14 Method Modifications

14.1 Method adapted for the analysis of PAH compounds by selected ion monitoring (SIM).

14.2 Hardware tuning using DFTPP is not performed because sample analysis is conducted in SIM mode.

14.3 All reported PAH compounds are treated as CCCs and SPCCs for initial and continuing calibration.

15 Pollution Prevention and Waste Management

15.1 Procedures for handling waste generated during this analysis are addressed in the Waste Handling or other applicable SOP.

15.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

15.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000C and 8270C

16.2 Pace Analytical Quality Manual; latest revision.

16.3 TNI Standard; Quality Systems section; latest revision.

17 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1 Table 1: Method Compounds and Reporting Limits

17.2 Table 2: Primary and Secondary Ions

18 Revisions

Document Number	Reason for Change	Date
S-IN-O-133-rev.02	1. Title page converted to periodic review format 2. Table of Contents fixed to contain Sections 10 and 11 3. Section 2.2: reference to Method 3550 removed. 4. Section 3: added language that limits, volumes, etc… are subject to change. 5. Table 7.1: removed TCLP and revised storage conditions. 6. Section 8: revised equipment and supplies where necessary and added "or equivalent" where applicable. 7. Section 9: revised and added "or equivalent" where applicable. 8. Section 10: added more detailed equations and revised SPCC criteria to match 8270C. Clarified ICAL criteria. 9. Section 11: removed table with instrument conditions and added the calculation for moisture correction. 10. Table 12.1: added 1/10 criteria for method blank and reference to Sections 12.1.1 and 12.1.2. 11. Sections 12.1.1 and 12.1.2 added to clarify LCS criteria. 12. Section 15: revised references to include 8000C.	18Mar2011
S-IN-O-133-rev.03	1. Table 7.1: added sample container for reduced sample volume extraction or PAH-SIM LVE 2. Table 9.3: added standards for PAH-SIM LVE. 3. Section 9.2.3: added preparation instructions for PAH-SIM LVE standards. 4. Section 11.3: added internal standard information for PAH-SIM LVE. 5. Section 14.1: revised SOP reference. 6. Section 15.3: changed NELAC to TNI	20Mar2012
S-IN-O-133-rev.04	1. Cover page and body header: added effective date. 2. Table of Contents: added Section 14, Method Modifications. 3. Section 3.1: added reference to LIMS for MDLs. 4. Table 9.3: updated for standards currently being used. 5. Section 9.2.3.1: updated to current procedure. 6. Section 9.2.3: removed intermediate PAH-SIM LVE standard solutions and revised to reflect that LVE standards are mode from a 10x dilution of regular PAH-SIM standards. 7. Section 9.2.3.8: updated to reflect working LVS ISTD. 8. Section 10.12: added as guidance for evaluation of low ICAL points. 9. Sections 10.13 and 10.14: revised ICV criteria and corrective action. 10. Section 10.16: clarified that CCC and SPCC criteria apply to all reported PAHs. 11. Section 11.6: added to require rerun or estimation of sample concentrations that exceed the linear range. 12. Section 11.7: added as a reference to the Manual Integrations SOP. 13. Table 12.1: updated corrective action for method blank and removed specific BP reference for ISTDs. 14. Section 13.1: added LOD/LOQ verification as option to annual MDL study requirement. 15. Section 14: added new Method Modifications section. 16. Section 16.1: removed reference to 8000B. 17. Table 1: updated soil RLs.	01Nov2013

S-IN-O-133-rev.05	1. Cover page: changed phone number and revised document control format. 2. Table 9.3: updated ICV details and revised storage conditions for standards. 3. Section 9.2.3.4: updated ICV preparation details. 4. Section 10.1: added PFTBA detail for auto tune. Removed ambiguous sentences regarding scan mode versus SIM mode for better clarity. 5. Section 10: removed quadratic curve fit option. 6. Section 11: removed calculations for curve fit types. 7. Updated Tables	27Oct2015

Table 1
Method 8270C SIM Compounds and Reporting Limits[1]

Analyte	RL aqueous (ug/L) 8270C SIM	RL solid (ug/kg) 8270C SIM
Acenaphthene	1	5
Acenaphthylene	1	5
Anthracene	0.1	5
Benzo(a)anthracene	0.1	5
Benzo(a)pyrene	0.1	5
Benzo(b)fluoranthene	0.1	5
Benzo(g,h,i)perylene	0.1	5
Benzo(k)fluoranthene	0.1	5
Chrysene	0.5	5
Dibenz(a,h)anthracene	0.1	5
Fluoranthene	1	5
Fluorene	1	5
Indeno(1,2,3-cd)pyrene	0.1	5
1-Methylnaphthalene[2]	1	5
2-Methylnaphthalene	1	5
Naphthalene	1	5
Phenanthrene	1	5
Pyrene	1	5

[1] Target Compounds and Reporting Limits are subject to change.
[2] Compound not listed in Method 8270.

Table 2
8270C SIM Primary and Secondary Ions[3]

Analytes	Primary Ion	Secondary Ion
Group 1-Naphthalene-d8 (IS)	**136**	**68**
Naphthalene	128	129
2-Methylnaphthalene	142	141
1-Methylnaphthalene[2]	142	141
Group 2-Acenaphthalene-d10 (IS)	**164**	**162**
2-Fluorobiphenyl (S)	172	171
Acenaphthylene	152	151
Acenaphthene	154	153
Fluroene	166	165
Group 3-Phenanthrene-d10 (IS)	188	94
Phenanthrene	178	179
Anthracene	178	176
Fluoranthene	202	101
Group 4-Chysene-d12 (IS)	**240**	**120**
Pyrene	202	200
Benzo(a)anthracene	228	229
Terphenyl-d_{14} (S)	244	122
Chrysene	228	226
Group 5-Perylene-d12 (IS)	**264**	**260**
Benzo(b)fluoranthene	252	253
Benzo(k)fluoranthene	252	253
Benzo(a)pyrene	252	253
Indeno(1,2,3-cd)pyrene	276	138
Dibenz(a,h)anthracene	278	139
Benzo(g,h,i)perylene	276	138

[3] Target Compounds and primary ions are subject to change.

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF SEMI-VOLATILE COMPOUNDS BY COMBINATION GC/MS SCAN AND SIM

REFERENCE METHOD: EPA SW-846 METHOD 8270C

SOP NUMBER:	S-IN-O-163-rev.04
EFFECTIVE DATE:	March 21, 2016
SUPERSEDES:	S-IN-O-163-rev.03

APPROVAL

General Manager — March 17, 2016 Date

Quality Manager — March 16, 2016 Date

Department Manager — March 17, 2016 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of semi-volatile organic compounds in sample extracts while meeting the requirements specified in EPA method 8270C.

2. Summary of Method

2.1. Samples undergo a solvent extraction and the resulting extract is divided into two equal portions. Semi-volatile compounds are introduced into a gas chromatograph by injection of the sample extract onto a narrow-bore capillary column for analysis. The column is temperature programmed to separate the analytes that are then detected with a mass spectrometer. Identification of the analytes is made by comparing their mass spectra with spectra of known standards using both Scan mode and Selective Ion Monitoring (SIM) mode. Quantitation is accomplished by comparing the response of a major ion relative to the internal standard response using a multi-point calibration curve.

3. Scope and Application

3.1. This method using Scan mode can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, phenols, and nitrophenols. This method using SIM mode can be used to quantitate a subset of semi-volatile compounds such as polynuclear aromatic hydrocarbon (PAH) compounds to achieve lower limits of detection. Other compounds may also be analyzed in SIM mode.

3.2. The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent extraction and exhibits poor chromatographic behavior. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosdiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3.3. The following compounds are analyzed using this procedure but are not 8270C listed compounds: Carbazole, Biphenyl (Diphenyl), Caprolactam, Atrazine, Benzaldehyde, Diethyl Aniline, 2,3-Dichloroaniline, 1-Methylnaphthalene and 4-Chlorobenzotrifluoride.

3.4. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.5. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC/MS equipment and the interpretation of GC/MS data acquired using both Scan and SIM modes. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. Method 8270C provides chromatographic conditions for the detection of semi-volatile compounds in organic extracts of aqueous and solid samples. Aqueous samples are extracted using SW-846 method 3510 Separatory Funnel Extraction or other applicable method. Solid samples are extracted using SW-846 method 3546 Microwave Extraction or other applicable method.

5. Limits of Detection and Quantitation

5.1. The list of compounds and reporting limits analyzed using both Scan and SIM modes of Method 8270C is found in Table 1. Other compounds may be reported upon completion of appropriate validation procedures. Refer to the LIMS for method detection limits.

6. Interferences

6.1. Glassware for the preparatory steps for this method should be thoroughly cleaned and rinsed. Soap products can leave phthalates on the glassware that in turn show up in the analytical data. Hits for phthalates should be closely scrutinized and continuous hits should warrant checking on the glassware cleaning procedure.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid, preferably 1L or 125mL, widemouth, or equivalent	None required	Cool to 6°C	Must be extracted within 7 days of collection date and analyzed within 40 days of extraction date
Solid	200 g in a 4 oz. glass jar	None required	Cool to 6°C	Must be extracted within 14 days of collection date and analyzed within 40 days of extraction date

Samples and sample extracts must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples. Sample extracts must be stored in the refrigerator or freezer until analysis is complete.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Description / Comments
Gas Chromatographs	Hewlett Packard/Agilent 6890/7890 or equivalent system
Data Systems	Hewlett Packard/Agilent Chemstation or equivalent system
Autosamplers	Hewlett Packard/Agilent or equivalent system
Mass Spectrometers	Hewlett Packard/Agilent 5973/5975. Must be capable of operating in **SIM** mode. Or equivalent system.

9.2. Chromatography Supplies

Item	Model / ID	Description
Analytical Column - Scan	Restek Rxi-5 Sil MS or equivalent	30m x 0.25mm or equivalent column
Analytical Column – SIM	Restek Rtx-5MS or equivalent	15m x 0.25mm, or equivalent column

9.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Syringe valves	2-way with Luer ends	Supelco or equivalent
Standard vials	2mL stop/go vials (clear vials)	Supelco or equivalent
Autosampler vials	1.8mL clear vials	Or equivalent

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Methylene Chloride	Pesticide grade or equivalent
Acetone	Pesticide grade or equivalent
Methanol	Pesticide grade or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions and vendors

Standard	Description	Comments
Tuning Standard	Standard used to tune the mass spectrometer in Scan mode. DFTPP tuning is not performed for SIM analysis.	DFTPP solution
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains method required spiking compounds, at a minimum, and is used for spiking MS/MSD sets.	Same solution can be used for the LCS and MS/MSD

10.2.2. Details and Storage Conditions

Table 10.3a – Analytical Standards and Storage Conditions for 8270 Scan Analysis

Standard Type	Description	Expiration	Storage
Stock 8270 calibration standard A	O2si; catalog #111236-01, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 calibration standard B2	O2si; catalog #111447-1, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 calibration standard C	O2si; catalog #010442-08, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8270/Intermediate 8270 LVE calibration standards	Refer to Sections 9.2.3.1	Expires 6 months from date of preparation.	Refrigerate
Working 8270 LVE calibration standards	Refer to Sections 9.2.3.2	Expires 6 months from date of preparation.	Refrigerate
Stock 8270 ICV standards-Phenol	Supelco; catalog #48904, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-BN1	Supelco; catalog #48900-U, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-HS1	Supelco; catalog #48907, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-HS2	Supelco; catalog #48908, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-Lilly ICV	Cresent; catalog #CCS-2579, 1000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-AccuCust	AccuStandard; catalog #S-939R, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-PAH	Supelco; catalog #CRM48905, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-Benzidines	Supelco; catalog #48906, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-BN2	Supelco; catalog #48120-U, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Intermediate 8270 LVE ICV standard	Refer to Section 9.2.3.3	Expires 6 months from date of preparation.	Refrigerate
Working 8270 LVE ICV standard	Refer to Section 9.2.3.4	Same expiration date as Intermediate 8270 LVE ICV standard	Refrigerate

Standard Type	Description	Expiration	Storage
Stock 8270 LVE internal standards	Restek; catalog # 31006; 4000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8270 LVE internal standards	Refer to Section 9.2.3.5	Expires 6 months from date of preparation.	Refrigerate
Stock 8270 LVE Surrogate standards	Supelco; catalog #CRM47262, 5000ug/mL, or equivalent Supelco; catalog #CRM 47261, 10000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 DFTPP Tuning Standard	Supelco; catalog #CRM47387, 50ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8270 DFTPP LVE Tuning Standard	Refer to Section 9.2.3.6	Expires 6 months from date of preparation.	Refrigerate

Table 10.3b – Analytical Standards and Storage Conditions for 8270 SIM Analysis

Standard Type	Description	Expiration	Storage
Stock 8270-SIM calibration standard	Restek; catalog # 31622; 2000ug/mL or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Intermediate 8270-SIM calibration standards #1 and #2	Refer to Sections 9.2.3.7 and 9.2.3.8	Expires 6 months from date of preparation.	Refrigerate
Working 8270-SIM calibration standard	Refer to Section 9.2.3.9	Expires 6 months from date of preparation.	Refrigerate
Stock 8270-SIM ICV standard w/surrogates	O2Si; catalog #20-2590; 100mg/L or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8270-SIM ICV standard w/surrogates	Refer to Section 9.2.3.10	Expires 6 months from date of preparation.	Refrigerate
Stock 8270-SIM Surrogate standard	Restek; catalog #31024, 1000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270-SIM Internal standard	Restek; catalog #31006, 4000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8270-SIM LVE calibration standards	Refer to Section 9.2.3.11	Expires 6 months from date of preparation.	Refrigerate
Working 8270-SIM LVE ICV standard w/surrogates	Refer to Section 9.2.3.12	Expires 6 months from date of preparation.	Refrigerate
Working 8270-SIM LVE Internal Standard	Refer to Section 9.2.3.13	Expires 6 months from date of preparation.	Refrigerate

10.2.3 Standard Preparation Procedures

10.2.3.1 Working 8270/Intermediate 8270 LVE Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Standard	Stock Cal. Std. A Amount	Stock Cal. Std. B2 Amount	Stock Cal. Std. C Amount	Stock Surrogate Std Added	Final Volume in Methylene Chloride	Final Nominal Cal Std Concentration	Stock Internal Standard amount added to Cal. Std.	Internal Standard Concentration
8270 Cal Std 1	5uL	2.5uL	2.5uL	5uL	1000uL	5ug/mL	10uL	40ug/mL
8270 Cal Std 2	10uL	5uL	5uL	10uL	1000uL	10ug/mL	10uL	40ug/mL
8270 Cal Std 3	20uL	10uL	10uL	20uL	1000uL	20ug/mL	10uL	40ug/mL
8270 Cal Std 4	50uL	25uL	25uL	50uL	1000uL	50ug/mL	10uL	40ug/mL
8270 Cal Std 5	80uL	40uL	40uL	80uL	1000uL	80ug/mL	10uL	40ug/mL
8270 Cal Std 6	100uL	50uL	50uL	100uL	1000uL	100ug/mL	10uL	40ug/mL
8270 Cal Std 7	150uL	75uL	75uL	150uL	1000uL	150ug/mL	10uL	40ug/mL

10.2.3.2 Working 8270 LVE Calibration Standards

Prepare using the Working 8270/Intermediate 8270 LVE Calibration Standards from Section 9.2.3.1. The following are examples of calibration standards and could vary based on requirements:

Standard	Intermediate Cal. Std. ID from Section 9.2.3.1	Intermediate Cal. Std. Amount	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Internal Standard Concentration
8270 LVE Cal. Std 1	8270 Calibration Std 1	50uL	500uL	0.5ug/mL	4ug/mL
8270 LVE Cal. Std 2	8270 Calibration Std 2	50uL	500uL	1.0ug/mL	4ug/mL
8270 LVE Cal. Std 3	8270 Calibration Std 3	50uL	500uL	2.0ug/mL	4ug/mL
8270 LVE Cal. Std 4	8270 Calibration Std 4	50uL	500uL	5.0ug/mL	4ug/mL
8270 LVE Cal. Std 5	8270 Calibration Std 5	50uL	500uL	8.0ug/mL	4ug/mL
8270 LVE Cal. Std 6	8270 Calibration Std 6	50uL	500uL	10ug/mL	4ug/mL
8270 LVE Cal. Std 7	8270 Calibration Std 7	50uL	500uL	15ug/mL	4ug/mL

10.2.3.3 Intermediate 8270 LVE ICV Standard

Combine the following:

25uL of Stock 8270 LVE - Phenol (2000ug/mL),
25uL of Stock 8270 LVE - BN1 (2000ug/mL),
25uL of Stock 8270 LVE - HS1 (2000ug/mL),
25uL of Stock 8270 LVE - HS2 (2000ug/mL),
25uL of Stock 8270 LVE - Lilly ICV (1000ug/mL),
50uL Stock 8270 LVE - AccuCust (2000ug/mL),
25uL of Stock 8270 LVE - PAH (2000ug/mL),
25uL of Stock 8270 LVE - Benzidines (2000ug/mL), and
25uL Stock 8270 LVE - BN2 (2000ug/mL)

Bring to a final volume of 1mL in Methylene Chloride for a final concentration o 50ug/mL.

10.2.3.4 Working 8270 LVE ICV Standard

Dilute 100uL of the Intermediate 8270 LVE ICV Standard (50ug/mL) to 500uL with methylene chloride for a final concentration of 10ug/mL.

10.2.3.5 Working 8270 LVE Internal Standards

Dilute 500uL of the Stock 8270 LVE internal standards (4000ug/mL) to 5mL with methylene chloride for a final concentration of 400ug/mL.

10.2.3.6 Working 8270 DFTPP LVE Tuning Standard

Dilute 50uL of the Stock DTPP Tuning Standard (50ug/mL) to 500uL with methylene chloride for a final DFTPP concentration of 5ng/uL.

10.2.3.7 Intermediate 8270-SIM Calibration Standard #1

Dilute 100uL of the Stock 8270-SIM Calibration Standard (2000ug/mL) plus 200uL of the Stock 8270-SIM Surrogate Standard (1000ug/mL) to 1mL with methylene chloride for a final concentration of 200ug/mL.

10.2.3.8 Intermediate 8270-SIM Calibration Standard #2

Dilute 25uL of the Intermediate 8270-SIM Calibration Standard #1 (200ug/mL) to 1mL with methylene chloride for a final concentration of 5ug/mL.

10.2.3.9 Working 8270-SIM Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Standard	Int. Std amt (#1 or #2)	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Working Internal Standard amount added to Cal. Std.	Internal Standard Concentration
8270-SIM Cal Std 1	10uL (Int #2)	1mL	0.05ug/mL	10uL	5ug/mL
8270-SIM Cal Std 2	20uL (Int #2)	1mL	0.1ug/mL	10uL	5ug/mL
8270-SIM Cal Std 3	100uL (Int #2)	1mL	0.5ug/mL	10uL	5ug/mL
8270-SIM Cal Std 4	200uL (Int #2)	1mL	1ug/mL	10uL	5ug/mL
8270-SIM Cal Std 5 (CCV)	25uL (Int #1)	1mL	5ug/mL	10uL	5ug/mL
8270-SIM Cal Std 6	50uL (Int #1)	1mL	10ug/mL	10uL	5ug/mL
8270-SIM Cal Std 7	100uL (Int #1)	1mL	20ug/mL	10uL	5ug/mL
8270-SIM Cal Std 8	250uL (Int #1)	1mL	50ug/mL	10uL	5ug/mL

10.2.3.10 Working 8270-SIM ICV Standard with Surrogates

Dilute 100uL of the Stock 8270-SIM ICV standard (100ug/mL) to 1mL with methylene chloride for a final concentration of 10ug/mL.

10.2.3.11 Working 8270-SIM LVE Calibration Standards

Prepare using the Working 8270-SIM Calibration Standards from Section 9.2.3.9.
The following are examples of calibration standards and could vary based on requirements:

Standard	8270 SIM Cal. Std. from Section 9.2.3.9	8270 SIM Cal. Std. Amount	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Internal Standard Concentration
8270-SIM LVE Cal Std 1	8270-SIM Cal Std 1	50uL	500uL	0.005ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 2	8270-SIM Cal Std 2	50uL	500uL	0.01ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 3	8270-SIM Cal Std 3	50uL	500uL	0.05ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 4	8270-SIM Cal Std 4	50uL	500uL	0.1ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 5 (CCV)	8270-SIM Cal Std 5	50uL	500uL	0.5ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 6	8270-SIM Cal Std 6	50uL	500uL	1.0ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 7	8270-SIM Cal Std 7	50uL	500uL	2.0ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 8	8270-SIM Cal Std 8	50uL	500uL	5.0ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 9	8270-SIM Cal Std 9	50uL	500uL	10.0ug/mL	0.5ug/mL

10.2.3.12 Working 8270-SIM LVE ICV Standard with Surrogates

Dilute 50uL of the Working 8270-SIM ICV Standard with Surrogates (10ug/mL) to 500uL with methylene chloride for a final concentration of 1.0ug/mL.

10.2.3.13 Working 8270-SIM LVE Internal Standard

Dilute 37.5uL of the Stock Internal Standard (4000ug/mL) to3mL with methylene chloride for a final concentration of 50ug/mL.

11. Calibration

11.1. Calibration for 8270 Scan Analysis

11.1.1 8270 Scan DFTPP Tune Verification: At the beginning of each 8270 Scan analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer must be hardware tuned using a 50ng injection of DFTPP. Analysis must not begin until the tuning criteria are met. Use the DFTPP mass intensity criteria in the table below as tuning acceptance criteria. Alternate tuning criteria may be used provided that method performance is not adversely affected. The 12-hour window during which standards and samples may be analyzed begins with the injection of DFTPP. All subsequent standards, samples MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

Mass (m/z)	Ion Abundance criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

If the DFTPP ratios do not meet the criteria, reanalyze the DFTPP tune. If the DFTPP still fails the criteria, autotune adjustment, instrument maintenance, and/or preparation of new standards must be considered.

The mass spectrum of DFTPP may be acquired by averaging three scans, the peak apex scan and the scans immediately preceding and following the apex. Background subtraction is required using this approach and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Do not background subtract part of the DFTPP peak. Alternatively, the analyst may use other approaches suggested below:

1. A single scan within the DFTPP peak with background subtraction of a single scan no more than 20 scans prior to the elution of DFTPP.
2. An average of multiple scans within the DFTPP peak with background subtraction of a single scan no more than 20 scans prior to the elution of DFTPP.

11.1.2 Initial Calibration: Initial Calibration standards are introduced into the GC/MS from the lowest to highest concentration of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
A_{IS} = Area of the quantitation ion for the internal standard.
C_{IS} = Concentration of the internal standard
C_x = Concentration of the compound being measured.

11.1.3 The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

11.1.4 The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of average RF for a compound
RF_{avg} = Mean of RFs for a compound

11.1.5 The %RSD should be should be $\leq$15% for each target analyte. However, the %RSD for each individual Calibration Check Compound (CCC) must be $\leq$30%. If the RSD of any CCC is >30%, then the chromatographic system is too reactive and instrument maintenance or preparation of new standards may be necessary before attempting recalibration. The CCCs are:

Base/Neutral Fraction	**Acid Fraction**
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

11.1.6 System Performance Check Compounds (SPCCs) are checked for a minimum average response factor (RF_{avg}) to determine potential instability and/or degradation caused by deterioration of instrument conditions or standard material. The minimum RF_{avg} for the semivolatile SPCCs are as follows:

N-nitroso-di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Instrument maintenance or preparation of new standards may be necessary. The SPCC criteria must be met for sample analysis to begin.

11.1.7 If the percent relative standard deviation (%RSD) of the RFs for a compound is $\leq$15% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

11.1.8 If the % RSD for any compound is >15%, the analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.1.9 Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be ≥ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.1.10 **Initial Calibration Corrective Action:** If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.1.11 Each day that analysis is performed, the calibration standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably. Additionally, examination of signal-to-noise ratio of analytes in low-level standards may be a useful diagnostic tool to monitor instrument performance. Signal-to-noise ratio is defined as the ratio of the analyte signal to the noise measured on a blank. It is recommended but not required that the signal-to-noise ratio be at least three to confirm detection.

11.1.12 **Initial Calibration Verification (ICV):** In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent recovery (%Rec) of the observed ICV according to the following equation:

$$\% \text{ Recovery} = \frac{\text{Observed concentration}}{\text{Theoretical concentration}} \times 100$$

The ICV is analyzed immediately following the initial calibration curve. The ICV recoveries are evaluated against a default acceptance range of 70-130% recovery. Alternative acceptance limits may be appropriate for some compounds.

11.1.13 **ICV Corrective Action:** If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

11.1.14 **Continuing Calibration Verification:** The initial calibration is verified every 12 hours by analyzing a DFTPP tune verification as described in Section 10.1, followed by a Continuing Calibration Verification (CCV) standard.

11.1.15 If the % difference (%D) or % Drift for each CCC is ≤20%, then the initial calibration is assumed to be valid. The response factors for all SPCCs in the CCV standard must meet the criteria in Section 10.1.6. The % difference (%D) or % Drift for each non-CCC compound should be <40%. If non-CCC compounds fail to meet this criteria, the concentrations above the reporting limit in associated samples must be qualified as estimated.

$$\% \text{ Difference} = [RF_v - RF_{avg}] / RF_{avg} * 100$$

11.1.16 The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

11.1.17 **CCV Corrective Action:** If a CCV fails the acceptance criteria, check the instrument operating conditions and if necessary, restore them to the original settings, and analyze another CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.2 Calibration for 8270-SIM Analysis

11.2.1 The instrument is auto-tuned periodically using PFTBA according to manufacturer's instructions to maximize detector sensitivity. Verification of hardware tuning is accomplished through the analysis of initial and continuing calibration standards in which the known ions are selected for data acquisition.

11.2.2 **Initial Calibration:** Initial Calibration standards are introduced into the GC from the lowest to highest concentration by direct injection of 1uL of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. The 12 hour clock during which standards and samples may be analyzed begins with the injection of the first calibration standard. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard using the calculation in Section 10.1.2.

11.2.3 The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

11.2.4 The percent relative standard deviation (%RSD) of the response factors is calculated using the calculation in Section 10.1.4.

11.2.5 All reported compounds in the calibration must be evaluated as CCC compounds. The %RSD of the RFs for each compound must be ≤30%.

11.2.6 All reported compounds in the calibration must be evaluated as SPCC compounds. Each compound must meet a minimum average response factor of 0.050.

11.2.7 If the %RSD of the RFs for a compound is ≤15% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

11.2.8 If the %RSD of the RFs for any compound is >15%, the analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for

quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.2.9 Initial Calibration Corrective Action: If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards should also be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.2.10 Each day that analysis is performed, the calibration standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably. Additionally, examination of signal-to-noise ratio of analytes in low-level standards may be a useful diagnostic tool to monitor instrument performance. Signal-to-noise ratio is defined as the ratio of the analyte signal to the noise measured on a blank. It is recommended but not required that the signal-to-noise ratio be at least three to confirm detection.

11.2.11 Initial Calibration Verification (ICV): In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent recovery (%Rec) of the observed ICV according to the criteria in Section 10.1.12.

11.2.12 ICV Corrective Action: If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

11.2.13 Continuing Calibration Verification: The initial calibration is verified every 12 hours by analyzing a Continuing Calibration Verification (CCV) standard. The CCV is prepared using the same standard solution used for the initial calibration. The 12 hour clock during which standards and samples may be analyzed begins with the injection of the CCV.

11.2.14 If the % Difference (%D) or % Drift for each reported PAH compound is $\leq$20%, then the initial calibration is assumed to be valid. Each reported PAH compound must meet the SPCC criteria in Section 10.2.6. All reported compounds are considered CCCs and SPCCs for the evaluation of the CCV.

11.2.15 The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

11.2.16 CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

12 Procedures

12.1 All sample extracts must be analyzed at room temperature and the instrument systems must be tuned (if required), calibrated as per Section 11, and free of contamination before samples are analyzed.

12.2 **Gas Chromatography conditions**: Configure the GC/MS per manufacturer's instructions.

12.3 **For 8270 Scan/8270-SIM extracts:** The extract obtained from the sample preparation laboratory is divided equally into two separate vials. Each vial must be fortified with internal standard compounds at the appropriate concentration for the intended analysis. Inject the extract into the GC/MS system using the same injection volume and operating conditions that were used for the calibration.

12.4 **Qualitative Analysis:** Compounds are identified as present when the following criteria are met:

12.4.1 The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.

12.4.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Refer to Table 2 for characteristic ions.

12.4.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.5 **Quantitative analysis:** Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique. See Sections 11.9, 11.10, and 11.11 for equations to calculate the amount or analyte or surrogate introduced into the instrument. Calculations are subject to change based on the data reduction software used. Refer to the current Manual Integration SOP for manual integration guidelines.

12.6 Refer to the Manual Integrations SOP for guidance on performing and documenting manual integrations.

12.7 If the sample concentration exceeds the linear range of the analysis, the sample must be diluted and reanalyzed or reported as an estimated concentration.

13 Quality Control

13.1 Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 3) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits. Refer to Sections 12.1.1 and 12.1.2 for additional information.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogates	Applicable surrogate compounds	Added to each standard, sample, and method blank.	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported. The surrogate must be qualified. 3) If only one surrogate fails and it is >10% recovery, re-extraction is not required but data must be qualified. 4) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.
As required by client only: **Internal Standards**	Applicable Internal Standard compounds	Added to each standard, sample, and method blank.	Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV.	Samples with internal standard failures must be reanalyzed undiluted or more concentrated. The laboratory may only dilute a sample prior to reanalysis if matrix interference is present. *Exception:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified.

13.1.1 The matrix spike may be used in place of the LCS as long as the acceptance criteria are as stringent as for the LCS.

13.1.2 **Allowable Marginal Exceedances**: If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. A marginal exceedance (ME) is defined as being beyond the LCS control limit of +/-3 standard deviations, but within the ME limits of +/-4 standard deviations around the mean. The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and correction action is necessary. If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken.

The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

NOTE: As allowed by client, the LCS shall be allowed to be outside the control limits but ≥10% for hexachlorocyclopentadiene, N-nitrosodimethylamine, pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, and 4-chloroaniline without corrective action. The LCS shall be allowed to be outside the control limits but >10% for up to four additional compounds, with the exception of any PAH, without corrective action. All PAH compounds must be within control limits.

14 Data Analysis and Calculations

14.1 Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_t)(D)}{(V_i)(V_s)} \qquad \text{Solid Sample (ug/Kg)} = \frac{(X_s)(V_t)(D)}{(V_i)(W_s)}$$

Where:
X_s = On-column concentration of the analyte in the sample aliquot injected
V_t = Total volume of concentrated extract
D = Dilution factor
V_i = Volume of the extract injected in uL
V_s = Volume of aqueous sample extracted in milliliters
W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100- \%Moisture)}} \times 100$$

14.2 LCS equation

R = (C/S) * 100

Where R = percent recovery
C = observed LCS concentration
S = concentration of analyte added to the clean matrix

14.3 MS/MSD equation

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = observed spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4 RPD calculations:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15 Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 Refer to Sections 11 and 13.

16 Corrective Actions for Out-of-Control Data

16.1 Refer to Sections 11 and 13.

17 Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 Refer to Sections 11 and 13.

18 Method Performance

18.1 An MDL study and/or LOD/LOQ verification must be conducted annually for 8270 Scan LVE and 8270 SIM LVE per instrument per matrix.

18.2 Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) for each component per matrix.

19 Method Modifications

19.1 Standards are purchased as certified stock solutions and not prepared from neat materials.

19.2 Phenol-d5 is used as a surrogate instead of Phenol-d6.

19.3 Extract final volumes, volume of internal standards added to extracts, and volume of extract injected into the instrument may vary from those identified in Method 8270C.

20 Instrument/Equipment Maintenance

20.1 Refer to maintenance log and/or instrument manufacturer's instructions.

21 Troubleshooting

21.1 Refer to maintenance log and/or instrument manufacturer's instructions.

22 Safety

22.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3 **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23 Waste Management

23.1 Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

24 Pollution Prevention

24.1 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24.2 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25 References

25.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000C and 8270C.

25.2 Pace Analytical Quality Manual; latest revision.

25.3 TNI Standard; Quality Systems section; latest revision.

26 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1 Table 1: 8270 Scan Target Compounds and Reporting Limits

26.2 Table 2: 8270 Characteristic Ions of Target Compounds

26.3 Table 3: 8270 SIM Target Compounds and Reporting Limits

26.4 Table 4: 8270 SIM Characteristic Ions of Target Compounds

27 Revisions

Document Number	Reason for Change	Date
S-IN-O-163-rev.02	1. Cover page: added actual effective date. 2. Section 9.2.3.14: added info for SIM LVE ICAL standard 9. 3. Section 10.1.8 and 10.1.9: removed calculations and made reference to 8000C. 4. Section 10.1.11: added as guidance for evaluation of ICAL standards. 5. Section 10.1.12: added that alternative limits may be appropriate for some ICV compounds. 6. Section 10.1.15: added criteria of <40% for non-CCC compounds in CCV. 7. Section 10.2.8 and 10.2.9: added reference to 8000C. 8. Section 10.2.11: added as guidance for evaluation of ICAL standards. 9. Section 10.2.15: clarified that CCC and SPCC criteria apply to all reported PAHs. 10. Section 11.7: added as a reference to the Manual Integrations SOP. 11. Section 11.8: added to require rerun or estimation of sample concentrations that exceed the linear range. 12. Sections 11.9-11.11: replaced Target calculations with 8000C calculations. 13. Table 12.1 and Section 12.1.2 Note: removed client-specific reference. 14. Section 13.1: added optional LOD/LOQ verification. 15. Section 16.1: removed reference to 8000B.	04Nov2013
S-IN-O-163-rev.03	1. Cover page: changed phone number and revised document control format. 2. Section 3: added a list of compounds that are analyzed but not listed in the method. 3. Section 8.2: updated column details 4. Table 9.3a: updated standard details and storage conditions 5. Table 9.3b: updated standard details and storage conditions 6. Section 9.2.3: updated several standard preparation procedures 7. Section 10.2.1: added PFTBA detail for auto tune. Removed ambiguous sentences regarding scan mode versus SIM mode for better clarity. 8. Section 10.2: removed quadratic curve fit option. 9. Section 11: removed references to 8270 Scan/8270 SIM extracts for full volume extractions. Removed calculations for curve fit types. Removed calculations for soil samples. 10. Updated tables 1-4.	27Oct2015
S-IN-O-163-rev.04	1. Converted to SOT format with 27 sections. 2. Section 4.1: added information and extraction method for solids. 3. Table 7.1: added collection, storage and holding time for solids. Changed storage conditions to “Cool to 6°C.” 4. Section 12.3: removed specific volumes and concentrations. 5. Section 14: added calculations for solids. 6. Section 18: added “per matrix” to DOC and MDL requirements. 7. Table 1: added RLs for solids and removed PAH compounds. 8. Table 3: added RLs for solids.	16Mar2016

Table 1: 8270 Scan Target Compounds and Reporting Limits[1]

Analyte	RL water (ug/L)	RL soil (ug/kg)
Phenol	10	330
Bis (2-chloroethyl) ether	10	330
2-Chlorophenol	10	330
Benzyl Alcohol	20	660
2-Methylphenol (o-Cresol)	10	330
Bis (2-chloroisopropyl)ether	5	330
3&4-Methylphenol (m&p-Cresol)	20	330
N-Nitroso-di-n-propylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
Bis(2-chloroethoxy)methane	10	330
Bis(2-chloro-1-methylethyl) ether	10	330
2,4-Dichlorophenol	10	330
4-Chloroaniline	20	660
Hexachloro-1,3-butadiene	5	330
4-Chloro-3-methylphenol	20	660
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	10	330
2-Chloronaphthalene	10	330
2-Nitroaniline	50	1600
Dimethyl phthalate	10	330
2,4-Dinitrophenol	50	1600
4-Nitrophenol	50	1600
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
2,6-Dinitrotoluene	10	330
3-Nitroaniline	50	1600
Diethyl phthalate	10	330
4-Chlorophenyl phenyl ether	10	330
4-Nitroaniline	50	1600
4,6-Dinitro-2-methylphenol	50	1600
N-Nitrosodiphenylamine	10	330
4-Bromophenyl phenyl ether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	50	1600
Di-n-butyl phthalate	10	330
Butyl benzyl phthalate	10	330
3,3'-Dichlorobenzidine	20	660
Bis(2-ethylhexyl)phthalate	5	330
Di-n-octyl phthalate	10	330

[1] Target Compounds and Reporting Limits are subject to change.

Table 2: 8270 Characteristic Ions of Target Compounds[2]

Analyte	Primary Ion	Secondary Ion(s)
Group 1-1,4-Dichlorobenzene-d4 (IS)	152	150, 115
N-Nitrosodimethylamine	42	74,44
Pyridine	79	52
2-Picoline	93	66, 92
4-Chlorobenzotrifluoride[3]	180	161,182
2-Fluorophenol (S)	112	64
Phenol-d5 (S)	99	71, 42
Benzaldehyde	77	105, 106
Phenol	94	65.66
Aniline	93	66, 65
Bis(2-Chloroethyl) ether	93	63.95
2-Chlorophenol	128	64,130
n-Decane[3]	57	43, 142
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
Benzyl alcohol	108	79, 77
1,2-Dichlorobenzene	146	148, 111
Bis(2chloro1methylethyl) ether	45	77, 121
Bis(2-chloroisopropyl) ether	45	77, 121
3&4-methylphenol (m&p cresol)	108	107,77
Acetophenone	105	77, 120
N-Nitroso-di-n-propylamine	70	130, 101
Hexachloroethane	117	201, 199
Group 2-Naphthalene-d8 (IS)	136	68
Nitrobenzene-d5 (S)	82	128, 54
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	109, 65
2,4-Dimethylphenol	122	107, 121
Bis(2-chloroethoxy) methane	93	95, 123
Benzoic Acid	105	122, 77
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 102
Apha-Terpineol	59	93, 121
2-Chloroaniline	127	65, 92
Hexachlorobutadiene	225	223, 227
Caprolactam	113	55, 56
Diethyl Aniline[3]	134	106, 77
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141, 115
1-Methylnaphthalene[3]	142	141, 115

[2] Target Compounds and primary ions are subject to change.
[3] Compound is not listed in Method 8270C.

Table 2: 8270 Characteristic Ions of Target Compounds[2]

Analyte	Primary Ion	Secondary Ion(s)
Group 3-Acenaphthene-d10 (IS)	164	160, 162
Hexachlorocyclopetadiene	237	235, 272
1,2,4,5-Tetrachlorobenzene	216	179, 108
2,3-Dichloroaniline[3]	161	163, 90
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Fluorobiphenyl (S)	172	171
2-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
1,3-Dinitrobenzene	168	76, 50
2,6-Dinitrotoluene	165	63, 89
Acenaphthylene	152	150, 153
3-Nitroaniline	138	108, 92
Biphenyl (Diphenyl)	154	153, 152
Acenaphthene	153	154, 152
2,4-Dinitrophenol	184	154, 63
4-Nitrophenol	109	139, 65
2,4-Dinitrotoluene	165	63, 89
Dibenzofuran	168	139, 169
2,3,4,6-Tetrachlorophenol	232	131, 230
Diethylphthalate	149	177, 150
4-Chloropheyl-phenylether	204	206, 141
Fluroene	166	165, 139
4-Nitroaniline	138	108, 65
Group 4-Phenanthrene-d10 (IS)	188	80,94
4,6-Dinitro-2-methylphenol	198	51,105
N-Nitrosodiphenylamine	169	168, 167
Azobenzene[3]	77	182, 105
1,2-Diphenylhydrazine	77	105, 182
2,4,6-Tribromophenol (S)	330	332, 141
4-Bromophenyl-phenyl ether	248	250, 141
Hexachlorobenzene	284	142, 249
Atrazine	200	215, 202
Pentachlorophenol	266	264, 268
n-Octadecane[3]	57	43, 71
Phenanthrene	178	179, 176
Anthracene	178	176, 179
Carbazole[3]	167	166, 139
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 203
Benzidine	184	92, 185

[2] Target Compounds and primary ions are subject to change.
[3] Compound is not listed in Method 8270C.

Table 2: 8270 Characteristic Ions of Target Compounds[2]

Analyte	Primary Ion	Secondary Ion(s)
Group 5-Chysene-d12 (IS)	240	120, 236
Pyrene	202	101, 203
p-Terphenyl-d14 (S)	244	122, 212
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Bis(2-Ethylhexyl) phthalate	149	167, 279
Benzo(a)athracene	228	229, 226
Chyrsene	228	226, 229
Group 6-Perylene-d12 (IS)	264	260, 265
Di-n-ocytlphthalate	149	279, 43
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluroanthene	252	253, 125
Benzo(a)pyrene	252	253,125
Indeno (1,2,3-cd)pyrene	276	138, 277
Dibenz (a,h) athracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277

[2] Target Compounds and primary ions are subject to change.
[3] Compound is not listed in Method 8270C.

Table 3: 8270 SIM Target Compounds and Reporting Limits[1]

Analyte	RL water (ug/L) 8270 SIM	RL soil (ug/kg) 8270 SIM
Acenaphthene	1	5
Acenaphthylene	1	5
Anthracene	0.1	5
Benzo(a)anthracene	0.1	5
Benzo(a)pyrene	0.1	5
Benzo(b)fluoranthene	0.1	5
Benzo(g,h,i)perylene	0.1	5
Benzo(k)fluoranthene	0.1	5
Chrysene	0.5	5
Dibenz(a,h)anthracene	0.1	5
Fluoranthene	1	5
Fluorene	1	5
Indeno(1,2,3-cd)pyrene	0.1	5
1-Methylnaphthalene	1	5
2-Methylnaphthalene	1	5
Naphthalene	1	5
Phenanthrene	1	5
Pyrene	1	5

[1] Target Compounds and Reporting Limits are subject to change.

Table 4: 8270 SIM Characteristic Ions of Target Compounds[2]

Analytes	Primary Ion	Secondary Ion
Group 1-Naphthalene-d8 (IS)	**136**	**68**
Naphthalene	128	129
2-Methylnaphthalene	142	141
1-Methylnaphthalene[3]	142	141
Group 2-Acenaphthalene-d10 (IS)	**164**	**162**
2-Fluorobiphenyl (S)	172	171
Acenaphthylene	152	151
Acenaphthene	154	153
Fluroene	166	165
Group 3-Phenanthrene-d10 (IS)	188	94
Phenanthrene	178	179
Anthracene	178	176
Fluoranthene	202	101
Group 4-Chysene-d12 (IS)	**240**	**120**
Pyrene	202	200
Benzo(a)anthracene	228	229
Terphenyl-d_{14} (S)	244	122
Chrysene	228	226
Group 5-Perylene-d12 (IS)	**264**	**260**
Benzo(b)fluoranthene	252	253
Benzo(k)fluoranthene	252	253
Benzo(a)pyrene	252	253
Indeno(1,2,3-cd)pyrene	276	138
Dibenz(a,h)anthracene	278	139
Benzo(g,h,i)perylene	276	138

[2] Target Compounds and primary ions are subject to change.
[3] Compound is not listed in Method 8270C.

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF 1,4-DIOXANE BY GC/MS SELECTED ION MONITORING (SIM)

REFERENCE METHOD: EPA SW-846 METHODS 3510C AND 8270C

SOP NUMBER:	S-IN-O-171-rev.01
EFFECTIVE DATE:	February 20, 2017
SUPERSEDES:	S-IN-O-171-rev.00

APPROVAL

General Manager — February 9, 2017 Date

Quality Manager — February 8, 2017 Date

Department Manager — February 8, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of 1,4-Dioxane in sample extracts while meeting the requirements specified in EPA methods 3510C and 8270C, through selected ion monitoring (SIM).

2. Summary of Method

2.1. Semi-volatile compounds are introduced into a gas chromatograph by injection of a sample extract onto a narrow-bore capillary column for analysis. The column is temperature programmed to separate the analytes that are then detected with a mass spectrometer. Identification of the analytes is made by comparing their mass spectra with spectra of known standards. Quantitation is accomplished by comparing the response of a major ion relative to the internal standard response using a multi-point calibration curve. Selected ion monitoring (SIM) is the mode of scanning for this SOP.

2.2. Method 8270C SIM provides chromatographic conditions for the detection of semi-volatile compounds in organic extracts of groundwater, surface water, soil and sediment. Aqueous samples are extracted by using SW-846 method 3510C Separatory Funnel Extraction or other applicable method.

3. Scope and Application

3.1. This method is for the preparation and analysis of 1.4-dioxane by Selected Ion Monitoring (SIM).

3.2. Reporting limits, control limits, volumes used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC/MS equipment and the interpretation of GC/MS SIM data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. The sample preparation is for the extraction of water insoluble or slightly water soluble organic compounds from groundwater, surface water and other aqueous samples using methylene chloride as the extraction solvent.

5. Limits of Detection and Quantitation

5.1. The 1,4-Dioxane default reporting limit is 3ug/L. Other analytes may be analyzed by this method but must have quality control documentation to support the performance of this method for those analytes. Refer to LIMS for method detection limits.

6. Interferences

6.1. Glassware for the preparatory steps for this method should be thoroughly cleaned and rinsed. Soap

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber glass container with Teflon-lined lid, preferably 1L or 100mL widemouth.	None required	Cool to $\leq 6^{o}C$	Sample must be extracted within 7 days of collection date and extract must be analyzed within 40 days of extraction date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples. Sample extracts must be stored in the refrigerator or freezer until analysis is complete.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Description / Comments
Gas Chromatographs	Agilent 6890 or equivalent system to include autosampler and data system.
Mass Spectrometers	Must be capable of operating in **SIM** mode. Or equivalent system.

9.2. Chromatography Supplies

Item	Description
Analytical Column	Restek Rxi-5 Sil MS 15m x 0.25mm, or equivalent column

9.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Standard vials	2mL stop/go vials (clear vials)	Supelco or equivalent
Autosampler vials	1.8mL clear vials	Or equivalent

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Methylene Chloride	Pesticide grade or equivalent
Methanol	Pesticide grade or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	ICAL
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains all target analytes and is used to prepare MS/MSD sets.	Same solution can be used for the LCS and MS/MSD

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock 1,4-Dioxane calibration standard	Supelco; catalog #CRM48367, 2000ug/ml or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Intermediate 1,4-Dioxane calibration standard	Refer to Sections 10.2.3.1	Solution good for 6 months from preparation	Refrigerate
Working 1,4-Dioxane calibration standards	Refer to Sections 10.2.3.2	Solution good for 6 months from preparation	Refrigerate
Stock 1,4-Dioxane ICV standard	Accustandard; catalog #APP-9-096, 100ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 1,4-Dioxane ICV standard w/surrogate	Refer to Sections 10.2.3.3	Solution good for 6 months from preparation	Refrigerate
Stock 1,4-Dioxane-d8 Surrogate standard	Supelco; catalog # 4M4829-U; 2000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 1,4-Dioxane-d8 surrogate standard	Refer to Sections 10.2.3.4	Solution good for 6 months from preparation	Refrigerate
Stock Internal standard	Supelco; catalog #CRM48902; 2000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working Internal standard	Refer to Sections 10.2.3.5	Solution good for 6 months from preparation	Refrigerate
Working 1,4-Dioxane spike standard	Refer to Sections 10.2.3.6	Solution good for 6 months from preparation	Refrigerate

10.2.3. Standard Preparation Procedures

10.2.3.1. Intermediate 1,4-Dioxane Calibration Standard Preparation

Dilute 50uL of the Stock 1,4-Dioxane Calibration Standard (2000ug/mL) plus 50uL of the Stock 1,4-Dioxane Surrogate Standard (2000ug/mL) to 1mL with methylene chloride for a final concentration of 100ug/mL.

10.2.3.2. Working 1,4-Dioxane Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Working Calibration Standard	Intermediate Standard amount	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Working Internal Standard amount added to Cal. Std.	Internal Standard Concentration
Cal Std 1	10uL	1mL	1ug/mL	10uL	4ug/mL
Cal Std 2	20uL	1mL	2ug/mL	10uL	4ug/mL
Cal Std 3	50uL	1mL	5ug/mL	10uL	4ug/mL
Cal Std 4 (CCV)	100uL	1mL	10ug/mL	10uL	4ug/mL
Cal Std 5	200uL	1mL	20ug/mL	10uL	4ug/mL
Cal Std 6	500uL	1mL	50ug/mL	10uL	4ug/mL

10.2.3.3. Working 1,4-Dioxane ICV Standard with Surrogate

Dilute 100uL of the Stock ICV standard (100ug/mL) plus 100uL of Working Surrogate Standard (100ug/mL) to 1mL with methylene chloride for a final concentration of 10ug/mL.

10.2.3.4. Working Surrogate Spike Standard Preparation

Dilute 1mL of the Stock Surrogate Standard (2000ug/mL) to 20mL with methanol for a final concentration of 100ug/mL.

10.2.3.5. Working Internal Standard Preparation

Dilute 200uL of the Stock Internal Standard (2000ug/mL) to 1mL with methylene chloride for a final concentration of 400ug/mL.

10.2.3.6. Working 1,4-Dioxane Spike Standard Preparation

Dilute 250uL of the Stock Calibration Standard (2000ug/mL) to 5mL with methanol for a final concentration of 100ug/mL.

11. Calibration

11.1. The instrument is auto-tuned periodically according to manufacturer's instructions to maximize detector sensitivity. DFTPP tuning is not performed for this method. DFTPP tune data is acquired in scan mode. For SIM analysis, the instrument is set to selected ion monitoring (SIM) mode. Verification of hardware tuning is accomplished through the analysis of initial and continuing calibration standards in which the known ions are selected for data acquisition.

11.2. **Initial Calibration:** Initial Calibration standards are introduced into the GC from the lowest to highest concentration by direct injection of 2uL of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. The 12 hour clock during which standards and samples may be analyzed begins with the injection of the first calibration standard. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
A_{IS} = Area of the quantitation ion for the internal standard.
C_{IS} = Concentration of the internal standard
C_x = Concentration of the compound being measured.

11.3. The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

11.4. The percent relative standard deviation (%RSD) of the response factors is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of the RFs for a compound
RF_{avg} = Mean of RFs for a compound

11.5. If the %RSD of the RFs for a compound is $\leq$15% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

11.6. If the %RSD of the RFs for any compound is >15%, the analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.7. Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results.

The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be greater than or equal to 0.99. Refer to Method 8000C for additional information regarding calibration.

11.8. Initial Calibration Corrective Action: If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards should also be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.9. Each day that analysis is performed, the calibration standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably. Additionally, examination of signal-to-noise ratio of analytes in low-level standards may be a useful diagnostic tool to monitor instrument performance. Signal-to-noise ratio is defined as the ratio of the analyte signal to the noise measured on a blank. It is recommended but not required that the signal-to-noise ratio be at least three to confirm detection.

11.10. Initial Calibration Verification (ICV): In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent recovery (%Rec) of the observed ICV according to the following equation:

$$\% \text{ Recovery} = \frac{\text{Observed concentration}}{\text{Theoretical concentration}} \times 100$$

The ICV is analyzed immediately following the initial calibration curve. The ICV recoveries are evaluated against a default acceptance range of 70-130% recovery. Alternative acceptance limits may be appropriate for some compounds.

11.11. ICV Corrective Action: If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

11.12. Continuing Calibration Verification: The initial calibration is verified every 12 hours by analyzing a Continuing Calibration Verification (CCV) standard. The CCV is prepared using the same standard solution used for the initial calibration. The 12 hour clock during which standards and samples may be analyzed begins with the injection of the CCV.

11.13. If the % Difference (%D) or %Drift for each compound and surrogate is $\leq$20%, then the initial calibration is assumed to be valid.

$$\% \text{ Difference} = [RF_v - RF_{avg}] / RF_{avg} * 100$$

11.14. The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

11.15. CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

12. Procedures

12.1. All sample extracts must be analyzed at room temperature and the system must be calibrated and free of contamination before samples are analyzed.

12.2. Sample Preparation and Handling

12.2.1. Aqueous Sample Extraction

12.2.1.1. Make sure that all glassware and Teflon separatory funnels used for this procedure have been properly washed. All washed glassware must be rinsed prior to use with acetone to remove residual water and with methylene chloride to remove any residual contaminants.

12.2.1.2. Measure the initial pH of each sample using wide range pH paper. Dip a clean disposable Pasteur pipette into each sample and touch the pipette to a piece of pH paper. Record the initial pH in the extraction log.

12.2.1.3. A nominal volume of 1L of aqueous sample is normally extracted. For samples expected to contain high concentrations of analytes, use a smaller aliquot of sample diluted to 1L with reagent water.

12.2.1.4. For each batch of 20 or fewer samples, prepare and method blank and an LCS by using a 1L aliquot of reagent water. The method blank will be used to check for contamination in the system. The LCS will be used to determine the efficiency of the extraction method in extracting target compounds. MS/MSD should also be prepared if sample volume allows.

12.2.1.5. Using a Class A graduated cylinder, measure the desired volume of sample to be used for the extraction and record the volume in mLs. Transfer the sample into a clean separatory funnel in a ring stand on a secured rack.

12.2.1.6. Add the appropriate surrogate solution to each method blank, sample, LCS and MS/MSD. Add the appropriate spiking solution to the LCS and MS/MSD. Refer to the standard preparation log and the sample preparation log for details regarding the appropriate spiking solution and volume to be used.

12.2.1.7. Adjust the sample pH if necessary, to the pH indicated Table 2 using 1:1 Sulfuric Acid or 10N Sodium Hydroxide. The pH is checked by dipping the tip of the disposable pipet into each well-mixed sample and placing the tip onto the pH paper to obtain a pH measurement.

12.2.1.8. Rinse the graduated cylinder or sample bottle with a 60mL portion of extraction solvent for a 1L sample and transfer the rinsate to the separatory funnel.

12.2.1.9. Seal the separatory funnels with Teflon lids and shake for two minutes with periodic venting. This can be done manually or on an automatic shaker. NOTE: Methylene Chloride may cause excessive pressure in the separatory funnel. It is recommended to shake slightly and vent before placing funnels on an automatic shaker.

12.2.1.10. Return the 2L separatory funnels to their ring stands and allow the solvent layer to separate for draining. If an excessive emulsion is present in the solvent layer, it can be broken up manually by using a clean glass stirring rod. If this is not successful, the sample can be drained into a clean secondary container (i.e. VOA vial) and transferred to a centrifuge tube. The extract can be centrifuged and then decanted into the drying funnel.

12.2.1.11. Drain the solvent layer through a drying funnel consisting of a clean funnel containing a plug of clean glass wool topped with a portion of clean sodium sulfate. The solvent should be collected in labeled beakers, labeled KD glassware, or labeled glass tubes.

12.2.1.12. Repeat the extraction two additional times with 60mL of solvent. Drain the solvent through the drying funnel after each extraction. After the final solvent extraction has been collected, rinse the funnel with methylene chloride and remove the drying funnel.

12.2.1.13. If extraction at a secondary pH is required, add acid or base as necessary and serially extract the sample, as described in Sections 12.2.1.9 through 12.2.1.12, at the adjusted pH. Collect all sample extract fractions together for concentration. Refer to Table 3 for extraction conditions.

12.2.1.14. **Concentration procedure:** Pour the extract into a labeled Kuderna-Danish concentrator with a concentrator tube securely attached. Add one or two clean boiling chips to the KD flask and attach a 3-ball Snyder column. Place the KD apparatus on a hot water bath so that the flask is partially immersed in the water. At the proper rate of distillation, the balls of the Snyder column should actively chatter but the chambers should not flood with solvent. Adjustment of the angle of the apparatus and the water temperature may be necessary to make the boiling more efficient. When the apparent volume of the extract reaches 4-6mL, remove the apparatus from the water bath and allow it to cool. Once cooled, carefully disassemble the KD apparatus rinsing each joint into the concentrator tube with a small amount of extraction solvent. Place the concentrator tube into the N-Evap and further concentrate the extract until the apparent volume is slightly below 1mL.

12.2.1.15. Prepare a calibrated vial by volumetrically dispensing the required volume of the solvent being used into a vial and securely capping the vial to eliminate evaporation. The calibrated vial must be prepared daily using a Class A pipet. Quantitatively transfer the sample extract from the concentrator tube to a vial. Bring the sample extract in the vial to the required final volume listed in Table3 by visually comparing the sample extract vial volume to the calibrated vial volume. Securely cap the sample extract vial. Store all extracts in the appropriate storage cooler. For extracts that will not concentrate to the usual final volume, use the procedure described above to bring the extract to the next higher practical volume for which a calibrated vial can be prepared. **Note:** Samples can be prepared for both 8270 full list and 1,4-Dioxane as a combo and the extract split for analysis.

12.3. **Gas Chromatography conditions**: Configure the GC/MS per manufacturer's instructions.

12.4. **Qualitative Analysis:** Compounds are identified as present when the following criteria are met:

12.4.1. The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.

12.4.2. The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. See Table 1 for primary and secondary ions.

12.4.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.5. **Quantitative analysis:** Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique.

12.6. When compound concentrations exceed the calibration range, the sample extract should be rerun at a dilution or reported as an estimated concentration.

12.7. Refer to the Manual Integrations SOP for guidance on performing and documenting manual integrations.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 3) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Target analyte	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified. 3) A Matrix Spike may be used in place of the LCS if it passes the LCS acceptance criteria.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Target analyte	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogates	Applicable surrogate compounds	Added to each standard, sample, and method blank.	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.
As required by client or program only: **Internal Standards**	Applicable Internal Standard compounds	Added to each standard, sample, and method blank.	Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV.	Samples with internal standard failures must be reanalyzed undiluted or more concentrated. The laboratory may only dilute a sample prior to reanalysis if matrix interference is present. *Exception:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_f)(D)}{(V_i)}$$

Where: X_s = On-column concentration of the analyte in ug/mL
V_f = Final volume of concentrated extract in mL
D = Dilution factor
V_i = Volume of sample extracted in L

14.2. LCS equation:

$$\mathbf{R = (C/S) * 100}$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. An MDL study and/or LOD/LOQ verification must be conducted annually per matrix per instrument if reporting below the default reporting limit.

18.2. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Method adapted for the extraction analysis of 1,4-Dioxane by selected ion monitoring (SIM).

19.2. Hardware tuning using DFTPP is not performed because sample analysis is conducted in SIM mode.

20. Instrument/Equipment Maintenance

20.1. Refer to maintenance log and/or instrument manufacturer's instructions.

21. Troubleshooting

21.1. Refer to maintenance log and/or instrument manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in the Waste Handling or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000C, 3510C and 8270C

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003, 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Table 1: 1,4-Dioxane Primary and Secondary Ions

26.2. Table 2: 1,4-Dioxane Extraction Conditions

27. Revisions

Document Number	Reason for Change	Date
S-IN-O-171-rev.00	1. First issue.	02Feb2015
S-IN-O-171-rev.01	1. Cover page: revised document control format. 2. Converted to 27-section format. 3. Table 7.1: updated storage temperature format. 4. Table 10.3: updated storage conditions. 5. Section 10.2.3.3: updated surrogate concentration and volume. 6. Sections 10.2.3.4 and 10.2.3.6: changed methylene chloride to methanol. 7. Section 25: added years to TNI reference.	06Feb2017

Table 1

8270C 1,4-Dioxane Primary and Secondary Ions

Analytes	Primary Ion	Secondary Ion
1,4-Dioxane	88	58
Internal Standards	**Primary Ion**	**Secondary Ion**
1,4-Dichlorobenzene-d4	152	115
Surrogates	**Primary Ion**	**Secondary Ion**
1,4-Dioxane-d8	96	64

Table 2
8270C 1,4-Dioxane Extraction Conditions

Determinative Method	Initial Extraction pH (if required)	#of extractions at Initial pH (if required)	Secondary Extraction pH	# of extractions at Secondary pH	Extraction Solvent	Exchange Solvent	Final Extract Volume (mL)
8270C 1,4-Dioxane	<2	3	>11	3	Methylene Chloride	N/A	1

Attachment B-2

Advanced GeoServices Organzational Chart

Attachment B-2
Organizational Chart
Exide Technologies
Frankfort, Indiana