Appendices

Appendix A

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

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Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Hudson River PCBs Superfund Site

Revised September 2015

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Hudson River PCBs
Superfund Site

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Acronyms and Abbreviations

ARCADIS	ARCADIS of New York, Inc.
BBL	Blasland, Bouck & Lee, Inc.
bgs	below ground surface
CLP	Contract Laboratory Program
cm	centimeters
COC	chain of custody
DPT	direct-push technology
DRO	diesel range organics
ELAP	Environmental Laboratory Accreditation Program
EPA	United States Environmental Protection Agency
Facility	Sediment Processing Facility
FML	flexible membrane liner
FSP	Field Sampling Plan
GE	General Electric Company
GPS	global positioning system
GRO	gasoline range organics
HDPE	high-density polyethylene
ID	identification
JSA	Job Safety Analysis
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LD	Laboratory duplicates
MDLs	method detection limits
MS/MSD	matrix spike/matrix spike duplicate
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health

PCB	polychlorinated biphenyl
PID	photoionization detector
PPE	personal protective equipment
QA/QC	quality assurance/quality control
RLs	reporting limits
SOP	standard operating procedure
SPF Demobilization FSP	Sediment Processing Facility Demobilization Field Sampling Plan
SPF Demobilization Plan	Sediment Processing Facility Demobilization and Restoration Plan
STARS	Spills Technology and Remediation Series
SVOC	semi-volatile organic compound
TCL	target compound list
TCLP	Toxicity Characteristic Leaching Procedure
TOC	total organic carbon
TPH	total petroleum hydrocarbons
USCS	Unified Soil Classification System
VOC	volatile organic compound
WTP	water treatment plant

1. Introduction

On behalf of General Electric Company (GE), ARCADIS of New York, Inc. (ARCADIS) has prepared this Phase 2 *Sediment Processing Facility Demobilization and Restoration Field Sampling Plan* (SPF Demobilization FSP) to present field procedures and sample collection methods for sampling to be performed in connection with demobilization of the Hudson River Sediment Processing Facility (the Facility) (Figure A-1). This document serves as an appendix to the Phase 2 *Sediment Processing Facility Demobilization and Restoration Plan* (SPF Demobilization Plan, ARCADIS 2015), which describes plans for demobilization and restoration of the Facility.

The field sampling and associated analytical activities described in this SPF Demobilization FSP will be performed in conformance with the applicable portions of the *Phase 2 Remedial Action Monitoring Quality Assurance Project Plan* (Phase 2 RAM QAPP, Anchor QEA and ESI 2012), including the October 2014 revision to Attachment A, Analytical Approach and Procedures (Revised Attachment A, ESI 2014). Where procedures, methods, and other information associated with the sampling identified in this SPF Demobilization FSP are not currently covered by the Phase 2 RAM QAPP, such information is included in attachments to this plan to supplement the Phase 2 RAM QAPP.

In general, the demobilization sampling program will include sampling of equipment, structures, Facility construction materials, and environmental media. Sampling of equipment, structures, and construction materials will be conducted in connection with Facility decontamination efforts within the exclusion zone to verify that items have been properly decontaminated and to determine appropriate disposition requirements. Environmental media sampling will occur after decontamination processes have been completed as described in the SPF Demobilization Plan. The environmental media sampling results will be compared with analytical data from sampling conducted prior to construction and operation of the Facility, as well as applicable New York State Department of Environmental Conservation (NYSDEC) standards and guidance values.

The remainder of this plan is organized as follows.

- Section 2 describes the field activities and procedures that will be used for sampling of equipment, structures, and construction materials in connection with the Facility decontamination efforts.

- Section 3 describes the field activities and procedures that will be used for environmental media sampling to be performed after Facility decontamination.
- Section 4 presents a description of sample handling and documentation requirements.
- Section 5 identifies the quality assurance/quality control (QA/QC) sampling and validation requirements.
- Section 6 describes the methods that will be used to decontaminate field sampling equipment and manage investigation-derived waste.

2. Facility Decontamination-Related Sampling

2.1 General

Sampling of equipment, structures, and construction materials will be conducted in connection with Facility decontamination efforts in the exclusion zone to verify that items have been properly decontaminated and to determine appropriate disposition requirements.

It is anticipated that the Facility decontamination-related sampling will include the collection of:

- wipe samples from painted and non-painted surfaces;
- pulverized core samples from asphalt and concrete materials;
- bulk material samples from construction materials (e.g., sand and sub-base gravel); and
- stormwater samples from drainage piping.

Field sampling Standard Operating Procedures (SOPs) have been developed with the goal of standardizing methodology so that data is collected utilizing consistent practices. However, it should be recognized that some deviations to the SOPs may occur depending on field-specific conditions. The following equipment and material sampling SOPs are provided as attachments to this FSP:

- Attachment A – SOP: Wipe Sample Collection
- Attachment B – SOP: Concrete/Asphalt Sampling
- Attachment C – SOP: Subsurface Media Sampling Using Manual Methods

2.2 Pre-Sampling Activities

Prior to sampling, a project-specific Job Safety Analysis (JSA) will be developed for each different sampling task. The JSA will identify the anticipated hazards and controls associated with the proposed sampling activities.

Field reconnaissance efforts will also be undertaken prior to sampling to review field conditions, potential evidence of impacts (e.g., visible staining or residue), obstructions, potential subsurface utilities, potential energy sources, or other obstacles and determine the final sampling locations.

The sampling activities will be coordinated with the construction manager and other contractors working in the area to minimize disturbance to other operations and to

provide a clear sampling area. In addition, before any sampling, potential nearby energy sources (including electrical and mechanical) must be de-energized, disconnected, and disabled, as applicable, in accordance with the project's energy control and lockout procedures.

Project-specific excavation permits will also be issued by the construction manager, as necessary, prior to conducting the sampling activities.

2.3 Equipment, Structure, and Appurtenance Sampling

Where required by the SPF Demobilization Plan, equipment, tanks, piping, pumps, supports, and associated appurtenances in the exclusion zone that have come into contact with polychlorinated biphenyl- (PCB-) containing materials, process water, or stormwater will be decontaminated after sediment processing activities are completed. In addition, the dewatering building and filter cake staging enclosures will be decontaminated and sampled as part of the demobilization process. The water treatment plant (WTP) building, which has not come into contact with PCB-containing sediment, will be cleaned, and limited sampling will be performed to verify that PCB concentrations are below the criteria described in the SPF Demobilization Plan. The rail yard support building, which is located outside of the exclusion area and has not come into contact with PCB-containing sediment, will not be subject to decontamination or sampling.

Following decontamination, verification samples will be collected from moveable and stationary equipment, tanks, steel piping, pumps, structures, and associated appurtenances that will be reused or sent off-site for salvage/recycling. In addition, for such items that are designated for off-site disposal, sampling may be performed to determine appropriate disposal requirements, as specified in the SPF Demobilization Plan.

Sampling of equipment, steel piping, structures, and related appurtenances will include the collection of wipe samples. In addition, pulverized core samples will be collected from the concrete floor slab in the dewatering building and the asphalt pavement and concrete retaining walls associated with the filter cake staging structures. The wipe sampling and concrete/asphalt core sampling will be conducted in accordance with the SOPs included in Attachment A and Attachment B, respectively.

Table A-1 outlines the proposed sample types and sampling frequencies for items to be decontaminated and sampled as part of the Facility demobilization program. Figure A-2 shows the proposed core sampling locations for the concrete floor slab within the dewatering building and the asphalt pavement within the filter cake staging structures.

For tanks, the dewatering building, the filter cake staging structures, and the water treatment plant (WTP) building, Table A-1 specifies the number of samples to be collected after decontamination. However, the number of wipe samples to be collected from individual pieces of equipment, steel piping, and related appurtenances will be determined in the field based on the sampling types and frequencies presented in Table A-1 and will also be dependent on the item to be sampled and the size of the item.

As summarized in Table A-1, equipment items that are intended for potential reuse or salvage/recycling will be sampled based on the estimated surface area of the item. For sampling purposes, equipment surface areas (and the number of samples to be collected) will be estimated based on a “hypothetical box” with sufficient size to contain the item. For oddly shaped pieces, multiple “hypothetical boxes” may be used to estimate equipment surface area. The equipment surface areas will be estimated by summing the surface areas for all six sides of each “hypothetical box” used to “contain” the item. The total number of samples (inclusive of samples from exterior surfaces and interior surfaces, where applicable) to be collected from each item will then be determined based on the sampling frequency presented in Table A-1.

For small equipment items (e.g., motors, etc.), pumps, and appurtenances (e.g., valves, instrumentation) that are designated for reuse or potential salvage/recycling, the sampling approach will consist of sampling a representative number of items instead of sampling each individual item. Steel piping, if designated for potential salvage/recycling, will be wipe sampled on the interior surfaces of the piping at a frequency of two samples per 150 linear feet. If high density polyethylene (HDPE) piping is designated for potential recycling, wipe samples will be collected from interior pipe surfaces at a frequency of two samples per 150 linear feet (for whole pipe sections) or, if the piping is shredded, bulk material composite samples will be collected for each approximately 100 cubic yards of shredded HDPE material. Table A-1 presents the proposed sampling frequencies.

When sampling a particular component, the sampling will focus on locations showing potential evidence of impacts (e.g., visible staining or residue) (if any) and locations where the greatest potential exposure to PCB-containing materials occurred (i.e., interior or exterior surfaces used for processing/handling PCB-containing sediment or where PCB-containing sediment accumulated during processing operations). This will include, but is not limited to, the collection of wipe samples from the interior surfaces of equipment, tanks, piping, pumps, and appurtenances. Sampling of structures will target surfaces within 6 feet of the ground and other areas if known to have had direct contact with PCB-containing sediment.

To aid in tracking the decontamination and sampling status, each equipment item and appurtenance sampled will be tagged for documentation and tracking purposes. The equipment tags will reference a distinct tracking identification (ID) generated for each item, the date when the item was decontaminated, the date when the item was sampled, and other equipment-specific information. The equipment tracking ID will be incorporated into the sample ID and will be recorded in the sampling field book to aid in data review and tracking.

As identified in Section 5, quality assurance/quality control (QA/QC) samples will also be collected as part of the sampling.

2.4 Construction Material Sampling

As described in the SPF Demobilization Plan, concrete and asphalt surfaces in the exclusion zone that have come into contact with PCB-containing sediment, process water, or stormwater will be decontaminated after sediment processing activities are completed, as provided in the SPF Demobilization Plan.

Following decontamination, verification samples will be collected from the concrete/asphalt to determine the PCB concentrations in the materials. In addition, the underlying sub-base stone and sand layers above the HDPE flexible membrane liner (FML) will be sampled only in areas where substantial sections of overlying asphalt or concrete pavements have been removed based on requests from the property owner and in areas where the full depth of overlying asphalt or concrete pavements has been removed to address PCB concentrations. A representative number of subsurface samples will also be collected proximate to the drainage piping and structures.

Table A-1 outlines the proposed sample types and sampling frequencies. As summarized in Table A-1, in-place sampling of concrete/asphalt pavement surfaces (and underlying construction materials above the FML) will be conducted on a 150-foot hypothetical grid established across the exclusion zone at the Facility. Figure A-2 shows the layout of the Sediment Processing Facility, the hypothetical sampling grid, and the approximate in-place sampling locations for concrete and asphalt surfaces and underlying aggregate materials based on the proposed sampling grid. The proposed sampling locations shown on Figure A-2 are not solely located at the centroid of each sampling grid cell. Instead, the proposed sampling locations within each sampling grid cell were selected to focus on areas where the greatest potential exposure to PCB-containing materials occurred (i.e., areas used for processing/handling PCB-containing sediment or where PCB-containing sediment was staged or accumulated during processing operations) and for spatial distribution considerations. As described in the SPF Demobilization Plan and as summarized in Table A-1, core samples will also be collected from in-place concrete/asphalt walls and below-grade structures and bulk

composite samples will be collected from concrete and asphalt that is removed, crushed, and placed in staging piles (i.e., associated with above-grade foundations, stormwater basins, pads, pavement, and other areas within the exclusion zone) (these sampling locations are not shown on Figure A-1).

In-place sampling of construction materials will include collection of pulverized core samples from concrete and asphalt surfaces and bulk material samples from underlying aggregate materials (e.g., sand and sub-base gravel), where applicable. The pulverized concrete and asphalt core samples will be collected to a depth of 7½ centimeters (cm) (or other depth agreed to by GE and EPA) from the upper concrete or asphalt surface.

As summarized in Table A-1, bulk composite sampling of concrete associated with the stormwater basins and other removed concrete/asphalt materials will be performed after removal. The concrete lining removed from the stormwater basins and concrete/asphalt removed from other locations (i.e., above-grade foundations, pads, pavement) will be crushed and placed in staging piles. Composite samples will be collected from the crushed concrete/asphalt at a frequency of 1 sample per 500 cubic yards.

The bulk material sampling of sub-base stone and sand layers will be collected using a hand auger after coring through the upper concrete/asphalt surface, where present.

The concrete/asphalt core sampling and bulk material construction media sampling will be conducted in accordance with the SOPs included in Attachment B and Attachment C, respectively.

Based on the sampling approach described above and presented in Table A-1, more than 100 samples are anticipated to characterize the concrete and asphalt surfaces within the exclusion zone.

Prior to sampling, field reconnaissance efforts will be undertaken to review field conditions, potential evidence of impacts (e.g., visible staining or residue), obstructions, potential subsurface utilities, or other obstacles and make adjustments, as necessary, to the proposed sampling locations shown on Figure A-2.

Depending on the sample analytical results, additional sampling may be performed to further delineate areas for subsequent decontamination or removal.

As identified in Section 5, QA/QC samples (i.e., duplicate samples, equipment blanks, and MS/MSDs) will also be collected as part of the sampling.

2.5 Stormwater Discharge Sampling

As described in the SPF Demobilization Plan, wet-weather stormwater sampling will be performed after the final decontamination of the drainage structures and piping is completed to document whether PCBs are detected in the stormwater that drains through the system.

Two wet-weather stormwater sampling events will be performed to verify the sampling results. During each sampling event, stormwater samples will be collected from each stormwater outfall pipe that discharges to the south and north stormwater basins. The stormwater samples will be analzed for PCBs.

As identified in Section 5, QA/QC samples (i.e., duplicate samples, equipment blanks, and MS/MSDs) will also be collected as part of the sampling.

2.6 Analytical Methods

The analytical approach and procedures for Facility decontamination-related samples are documented in Attachment O. This attachment identifies the laboratory selected for analysis of the Facility-decontamination-related samples, as well as the certifications, method detection limits (MDLs), reporting limits (RLs), method performance criteria, and SOPs relevant to these analyses.

Table A-2 provides a summary of the samples anticipated to be collected as part of the decontamination-related sampling program, including field QA/QC sample requirements (see Section 5). Table A-4 summarizes the analytical parameters and laboratory methods for each sampling matrix.

3. Environmental Media Sampling

After termination of sediment processing/handling activities and after completion of decontamination processes as described in the SPF Demobilization Plan, an environmental sampling program will be implemented. The post-decontamination environmental sampling program will include the collection and analysis of soil, groundwater, sediment, and surface water samples at and immediately adjacent to the Facility. Analytical data from the environmental sampling program will be compared with analytical data from sampling conducted prior to construction and operation of the Facility, as well as applicable NYSDEC standards and guidance values, to determine whether additional sampling or other actions are needed.

Figure A-3 shows the layout of the Sediment Processing Facility and the proposed environmental media sampling locations. Soil sampling locations were selected to target areas used to process, handle, and stage PCB-containing sediment during Facility operations. Groundwater, surface water, and sediment sampling locations were selected to generally correspond to locations sampled as part of baseline characterization sampling programs conducted between 2003 and 2009 prior to construction and operation of the Facility.

Field sampling SOPs developed for the environmental sampling activities are provided as attachments to this FSP and include the following:

- Attachment D – SOP: Soil Borings and Soil Sampling
- Attachment E – SOP: Groundwater Monitoring Well Development
- Attachment F – SOP: Water Level Measurement
- Attachment G – SOP: Groundwater Purging and Sampling
- Attachment H – SOP: Sediment Sampling
- Attachment I – SOP: Surface Water Sampling
- Attachment J – SOP: Field Parameter Measurement
- Attachment K – SOP: Photoionization Detector Field Screening

3.1 Pre-Sampling Activities

Prior to sampling, JSAs will be developed to identify the anticipated hazards and controls associated with the sampling activities.

In addition, field reconnaissance efforts will be undertaken prior to sampling to review field conditions, potential evidence of impacts (e.g., visible staining or residue), obstructions, potential subsurface utilities, or other obstacles and determine the final sampling locations.

The sampling activities will be coordinated with the construction manager and other contractors working in the area to minimize disturbance to other operations and to provide a clear sampling area.

Project-specific excavation permits, as applicable, will also be issued by the construction manager prior to conducting the sampling activities.

3.2 Surface and Subsurface Soil Sampling

Surface and subsurface soil samples will be collected at various locations across the Facility to characterize soils beneath and immediately surrounding areas used to process, handle, and stage PCB-containing sediment during Facility operations. The proposed soil sample locations are illustrated on Figure A-2. It is anticipated that surface soil sampling will be conducted at 17 locations surrounding PCB sediment handling areas. Subsurface soil samples will be collected from soil borings completed at 22 locations.

A description of the sample collection methods and protocols are presented in the soil boring and soil sampling SOP included as Attachment D of this FSP.

Where only surface soil samples will be collected, the sampling will be performed using a hand auger sampling device that is manually advanced to a depth of 6 inches below ground surface (bgs).

Where subsurface samples will be collected, soil borings will be completed using direct-push technology (DPT) sampling methods (e.g., Geoprobe®-type rig) – the same method used to collect soil samples during the baseline site characterization program. In areas with concrete or asphalt pavement, coring will be performed to provide access to underlying soil for sampling. The DPT borings will be advanced to a depth of 4 feet below ground surface or 4 feet below the containment liner system, where present.[1] Soil samples will be collected with a 4-foot-long Macro-Core® sampling device using disposable acetate sleeves. In the event that insufficient sample recovery (i.e., consistently less than 50%) is encountered, the sampling will continue with the 4-foot Macro-Core® device, but the sampling intervals will be reduced to 2 feet.

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[1] In general, the profile of the containment system from bottom to top consists of a non-woven geotextile, a high-density polyethylene (HDPE) flexible membrane liner (FML), a non-woven geotextile, a sand drainage layer, a woven geotextile, a sub-base aggregate layer, and upper pavement courses (i.e., asphalt or concrete).

Field personnel will record a detailed description of each sample interval using the Unified Soil Classification System (USCS), including lithologic description, moisture content, color, percent recovery, grain size distribution, and any visual or olfactory observations. A portion of each sampling interval will be split for headspace screening for volatile organic vapors using a properly calibrated field photoionization detector (PID) (see Attachment K). The headspace readings will be recorded in the field notes.

At surface soil sample locations, one soil sample from the 0- to 6-inch depth interval will be collected for laboratory analysis for PCBs.

At subsurface soil boring locations below the containment liner system, one soil sample will be collected for PCB analysis from the uppermost 0- to 2-foot sample interval below the containment liner system. Subsurface soil samples collected from the size separation area (SB-301 through SB-305) will also be analyzed for target compound list (TCL) semi-volatile organic compounds (SVOCs) (including nitrobenzene, 2,4-dinitrotoluene, and 2,6-dinitrotoluene) and total petroleum hydrocarbons (TPH) diesel range organics (DRO) and TPH gasoline range organics (GRO) (based on the potential for explosive or propellant residues and petroleum impacts on the property). Field PID measurements will be obtained from soil samples collected from the 0- to 2-foot and 2- to 4-foot subsurface intervals below the containment liner system at each boring. If a subsurface sample has the potential presence of volatile organic vapors through PID screening or based on visual observations, a sample will also be collected and submitted from the boring for laboratory analysis for NYSDEC Spills Technology and Remediation Series (STARS) list volatile organic compounds (VOCs) and STARS list SVOCs.

At subsurface soil boring locations SB-321 and SB-322, where temporary above-ground fuel storage occurred during the Facility operations, field PID measurements will be obtained from soil samples collected from the 0- to 2-foot and 2- to 4-foot depth intervals at each boring. If a subsurface sample indicates the potential presence of volatile organic vapors through PID readings above background or based on visual observations, a sample will also be collected from that depth interval and submitted for laboratory analysis for NYSDEC STARS list VOCs, STARS list SVOCs, TPH DRO, and TPH GRO. If there are no indications of the potential presence of volatile organic vapors (through PID screening or based on visual observations), one sample will be collected and submitted from the 0- to 2-foot depth interval for laboratory analysis for NYSDEC STARS list VOCs, STARS list SVOCs, TPH DRO, and TPH GRO.

Following completion, each core/boring location will be abandoned by backfilling with a bentonite/cement grout. Concrete/asphalt surfaces will be restored using a fast-setting concrete mix.

As described in Section 5, QA/QC samples will also be collected as part of the soil sampling.

3.3 Groundwater Sampling

Groundwater samples will be collected from 12 existing groundwater monitoring wells that were installed in 2005 as part of the baseline characterization sampling program. The existing monitoring wells to be sampled include: MW-01, MW-01D, MW-02, MW-02D, MW-08, MW-09, MW-10, MW-11, MW-12, MW-13, MW-15, and MW-17.

The total depths of the existing 10 shallow monitoring wells range from approximately 15 to 23 feet bgs. The total depths of the two deep monitoring wells are approximately 51 and 75 feet bgs. Well construction and gauging details for the monitoring wells installed as part of the 2005 baseline event were presented in the *New York State Canal Corporation Baseline Characterization Data Summary Report* (Blasland, Bouck & Lee, Inc. [BBL] 2006a) and the *Energy Park/Longe Baseline Characterization Data Summary Report* (BBL 2006b). A table presenting a monitoring well construction and gauging summary is included in Attachment N of this FSP.

Initially, each of the 12 existing monitoring wells to be sampled will be redeveloped in accordance with the SOP presented in Attachment E. After development, the monitoring wells will be allowed to stabilize for a minimum of one week prior to groundwater sampling.

Before groundwater sampling, groundwater levels at each existing monitoring well will be measured and recorded to the nearest 0.01 foot in accordance with the SOP presented in Attachment F. Groundwater samples will then be collected from each monitoring well utilizing the low-flow groundwater sampling techniques described in the SOP presented in Attachment G. Field water quality measurements (i.e., for turbidity, specific conductivity, pH, oxidation/reduction potential, and temperature) will be measured and recorded during the purging/sampling process following the SOP presented in Attachment J.

Consistent with the baseline sampling events, the groundwater samples will be analyzed for PCBs, VOCs, SVOCs, pesticides, metals, and oil & grease. As described in Section 5, QA/QC samples will also be collected during the groundwater sampling.

3.4 Sediment Sampling

Sediment samples will be collected from 11 locations, as shown on Figure A-3. The sediment sampling locations were selected to generally correspond to locations sampled during the baseline sampling program. As shown on Figure A-3, sediment samples will be collected from the following locations:

- Three locations within the Champlain Canal
- Five locations within Bond Creek
- Three locations within the Lock Diversion Canal

The sediment sampling will be performed in accordance with the SOP presented in Attachment H. It is anticipated that Lexan® tubing (or similar) will be used to collect the sediment core samples. The tubing will be inserted with a vertical entry into the sediment bed to secure a reliably representative cross-section sample. The core tubes will be advanced to a depth of approximately 3 feet below the sediment surface, or refusal if encountered first. At each sampling location, one sample will be collected for laboratory analysis from the 0- to 6-inch surface depth interval and one composite sediment sample will be collected for laboratory analysis from the entire length of the recovered core.

The sediment samples will be analyzed for PCBs and total organic carbon (TOC). As identified in Section 5, QA/QC samples will also be collected as part of the sediment sampling.

3.5 Surface Water Sampling

Surface water samples will be collected from four locations adjacent to the Sediment Processing Facility. The proposed surface water sampling locations are depicted on Figure A-3 and generally correspond with locations sampled during the baseline sampling program. As shown on Figure A-3, two surface water samples will be collected from Bond Creek, and two surface water samples will be collected from the Lock Diversion Channel.

The surface water sampling will be performed following the sample collection methods and protocols presented in the SOP included as Attachment I. The surface water sampling will be in conjunction with the sediment sampling activities (see Section 3.4) and will generally proceed in a downstream to upstream direction.

The surface water samples will be analyzed for PCBs. In addition, as identified in Section 5, QA/QC samples will be collected as part of the surface water sampling.

3.6 Sample Location Survey

Each sample location will be marked and surveyed in the field using either survey-grade global positioning system (GPS) or conventional survey methods.

3.7 Analytical Methods

The analytical approach and procedures for the soil, sediment, groundwater, and surface water samples will be provided in Attachment P after the laboratory(ies) is selected. This attachment will be updated to identify the laboratory(ies) selected for analysis of the environmental media samples, as well as the certifications, MDLs, RLs, method performance criteria, and SOPs relevant to these analyses.

Table A-3 provides a summary of the samples anticipated to be collected as part of the environmental media sampling program, including field QA/QC sample requirements (see Section 5). Table A-4 summarizes the analytical parameters and laboratory methods for each sampling matrix.

4. Sample Handling and Documentation

Sample handling and documentation procedures will be conducted in accordance with applicable portions of the Phase 2 RAM QAPP. Additional details concerning these procedures are discussed below.

4.1 Sample Labeling, Packing, and Shipping

Sample labeling, packing, and shipping methods will be completed in accordance with *Appendix 2.6-4 Standard Operating Procedure for Handling, Packing, and Shipping Samples*, located in the Phase 2 RAM QAPP. The analytical laboratory will supply appropriate sample containers and preservatives, as necessary. The field personnel will be responsible for properly sealing and labeling the sample containers and packing the samples for transport to the analytical laboratory.

Sample labeling will follow the procedures used for other sampling on the project and will be completed electronically through a sample tracking database. Unique sample identifications will be provided for each sample. The sample identification nomenclature for the construction media and structures is presented in Attachment M. The sampling identification nomenclature for equipment items will be similar, but will also include the distinct tracking ID generated for each item as described in Section 2.3. The sampling identification nomenclature for environmental media sampling will also be similar, but will also include reference to the sampling location ID shown on Figure A-3.

The samples collected each day will be packaged by the field personnel in a cooler with ice and hand delivered by either direct courier or 24-hour delivery courier at the end of each day’s sample collection and processing activities under chain-of-custody to GE’s analytical laboratory for analysis.

4.2 Documentation

Appropriate documentation procedures will be employed throughout the field sampling program. Documentation will be completed in accordance with the Phase 2 RAM QAPP where applicable, and will constitute a record which allows reconstruction of field events to aid in the data review and interpretation process. Documents, records, and information relating to the performance of the field work will be retained in the project file.

The various forms of documentation to be prepared and maintained throughout the sampling program include:

- Daily Production Documentation: Field notebooks will contain a record of the sampling activities performed at the Facility. The field notebooks will be maintained by the field team leader and other team members to provide a daily record of significant events, observations, and measurements during the field sampling event. Information pertinent to the field investigation and/or sampling activities will be recorded in the field notebooks.

- Sampling Notes: Detailed notes will be made related to the item sampled, sampling locations, physical observations, sample depths, field measurements, and weather conditions.

- Sampling Sketches: Sketches will be prepared to depict the sampling locations and field measurements for equipment items and structures sampled.

- Equipment Tags: As described in Section 2.3, each equipment item and appurtenance sampled will be tagged for documentation and tracking purposes. The equipment tags will reference a distinct tracking ID generated for each item, the date when the item was decontaminated, the date when the item was sampled, and other equipment-specific information (e.g., equipment name, manufacturer, model number, location).

- Photographs: Photographs of the sampling locations and collected samples will be taken.

- Sample Chain-of-Custody: Chain-of-custody (COC) forms will provide the record of responsibility for sample collection, transport, and submittal to the analytical laboratory. COC forms will be filled out by the sampling personnel, and will accompany the samples to the laboratory as described in Section 10 and Appendix 2.6-4 of the Phase 2 RAM QAPP.

- Field Equipment, Calibration, and Maintenance Logs: Logs will be maintained to document the calibration and maintenance of field instrumentation. Calibration and maintenance logs will be maintained for each piece of field equipment that is not factory-calibrated.

5. Quality Assurance/Quality Control

QA/QC procedures will be conducted in accordance with the Phase 2 RAM QAPP where applicable. Additional details concerning QA/QC measures employed in connection with the field sampling and analytical procedures are discussed below.

5.1 Quality Assurance and Quality Control (QA/QC) Sampling

This subsection summarizes the QA/QC requirements for the field sampling activities. Tables A-2 and A-3 summarize the QC sample requirements. The QA/QC sampling requirements are described below. The results of the field QA/QC samples will be compared to established measurement performance criteria in order to assess the accuracy and precision of the data generated during the SPF demobilization and restoration program. Measurement performance criteria for the following QA/QC samples are provided in Attachment O for the decontamination-related samples. Measurement performance criteria for QA/QC sampling associated with the environmental media samples (i.e., soil, sediment, groundwater, and surface water) are provided in Attachment P.

Duplicate Samples

Blind duplicate samples will be sent for laboratory analysis to evaluate the reproducibility of the sampling technique used. Five percent (i.e., one for every 20 samples) of each matrix will be duplicated (i.e., concrete, groundwater, subbase, soil, etc.). The duplicate samples will be collected using methods to maximize the compatibility of the samples. For example, material collected from a particular location will be thoroughly homogenized and then divided between the sample and duplicate sample laboratory containers. Wipe field duplicate samples will consist of co-located samples since it is not possible to collect true field duplicates for wipe samples.

Matrix Spike/Matrix Spike Duplicate

Triple sample volumes from designated sample locations will be collected for each matrix in order to perform MS/MSD analysis at a frequency of five percent (i.e., one for every 20 samples) of each matrix (with the exception of wipe samples). Laboratory duplicates (LDs) are typically substituted for MSDs for inorganic and conventional parameter analysis. Since triplicate volumes cannot be collected for wipe samples, the laboratory will prepare a laboratory control sample (LCS) and a laboratory control sample duplicate (LCSD) instead (refer to Attachment O).

Equipment Field Blanks

When non-disposable equipment is used and decontamination efforts on non-dedicated equipment are needed, the decontamination procedure will be assessed by preparing field blanks. Equipment field blanks will not be collected when dedicated sampling devices are used or when dedicated sample containers are used to collect the samples.

For wipe samples, field blanks will consist of an unused wipe placed in a sample jar with the same volume of hexane that is used in collection of a wipe sample.

For solid sample collection methods (i.e., soil, sediment, concrete/asphalt, and aggregate material), field blanks will be prepared in the field by contacting clean "builder's sand" with decontaminated sampling equipment and then the sand will be transferred directly into the laboratory-supplied sample bottles to verify that the equipment will not lead to cross contamination of samples. The intent is for the clean "builder's sand" making up the blank to follow the same path, and therefore, come in contact with the same equipment as the samples. Equipment blanks are not applicable to TOC analysis.

For water samples (i.e., groundwater and surface water), field blanks will consist of rinse blanks prepared by passing deionized water through or over the decontaminated sample device and collecting the rinse blank in sample containers.

If non-disposable equipment is used, an equipment field blank will be collected once per day for each type of sampling equipment used per 20 samples submitted to the laboratory. Equipment field blanks may be performed on non-dedicated decontaminated sampling equipment and other equipment such as bowls or pans if used to homogenize samples. Equipment field blanks must accompany the samples collected that day.

Trip Blanks

Trip blanks will be used to assess whether site samples have been exposed to non-site-related volatile constituents during sample storage and transport. Trip blanks will be submitted at a frequency of one per cooler of aqueous samples to be analyzed for VOCs and TPH GRO. A trip blank will consist of a container filled with analyte-free water (supplied by the laboratory), which remains unopened with field samples throughout the sampling event. Aqueous trip blanks will only be analyzed for VOCs. A soil trip blank will consist of vials containing preservatives only from an unused Terra Core kit. Soil trip blanks will only be analyzed for VOCs and TPH GRO.

Temperature Blanks

The purpose of preparing temperature blanks and sending the temperature blanks in the sample coolers on location is to enable the laboratory to monitor the temperature of the coolers (and field samples) upon receipt at the laboratory. A temperature blank will be provided in each cooler.

5.2 Data Review / Validation

Consistent with the baseline characterization, approximately 50 percent of the laboratory data associated with the environmental sampling will be reviewed to evaluate the data quality and usability. A Tier III validation will be conducted in accordance with the United States Environmental Protection Agency's (EPA's) *National Functional Guidelines for Organic Data Review* (USEPA 1999), EPA's *National Functional Guidelines for Inorganic Data Review* (USEPA 2004), and EPA Region 2 RCRA and CERCLA Data Validation SOPs, where applicable.

A complete record of each of these field samples' history will be available for documenting its progress from the time of sample collection, to arrival at the laboratory, processing and analysis at the laboratory, and sample receipt and reporting. The data usability summary review will include the use of dated entries, signed by analysts and supervisors on worksheets and logbooks used for all samples; the use of sample tracking and numbering systems to logically follow the progress of samples through the laboratory; use of calibration, QC, and sample raw instrument data, and the use of quality control criteria to reject, accept, or qualify specific data. The requirements that will be checked during the review include: holding times, blanks, surrogate recovery, MS/MSD and/or laboratory control samples, field duplicates, instrument calibration and verification, instrument mass tuning internal standard responses, instrument-specific quality control sample analyses, compound identification, compound quantitation, reported detection limits, and overall assessment of the data.

6. Equipment Decontamination and Management of Investigation-Derived Waste

6.1 Equipment Decontamination

During the sampling activities, both disposable and non-disposable equipment is anticipated to be used. For non-disposable field sampling equipment being used, the equipment will be decontaminated using the decontamination procedures described in the SOP presented in Attachment L.

Non-disposable sampling equipment will be cleaned before collecting each sample, between sampling events, and prior to leaving the site. Cleaning of sampling tools will be conducted at an established equipment decontamination area on site. Equipment blank samples will be collected from the decontaminated equipment as described in Section 5.

Dedicated and/or disposable (i.e., not to be re-used) sampling equipment will not require decontamination.

6.2 Investigation-Derived Waste Management

Investigation-derived waste generated by the sampling activities (e.g., core cuttings, sampling supplies, decontamination water, personal protective equipment [PPE], etc.) will be managed for appropriate treatment and/or disposal. Rinsate from decontamination activities will be containerized for appropriate treatment and/or disposal. PPE (i.e., gloves, disposable clothing) and other disposable equipment and debris resulting from sampling operations (i.e., plastic sheeting, sampling equipment, and other equipment not reused in the investigation) will be placed in plastic bags or a drum for appropriate disposal.

7. References

Anchor QEA and ESI, 2012. Phase 2 Remedial Action Monitoring Quality Assurance Project Plan. Hudson River PCBs Superfund Site (Phase 2 RAM QAPP, May 2012) with Analytical Program Approach and Procedures (Attachment A, revised October 2014).

BBL. 2006a. New York State Canal Corporation Baseline Characterization Data Summary Report. Prepared for General Electric Company, Albany, New York. September.

BBL. 2006b. Energy Park/Longe Baseline Characterization Data Summary Report. Prepared for General Electric Company, Albany, New York. October.

EPA. 1999. National Functional Guidelines for Organic Data Review. October.

EPA. 2004. National Functional Guidelines for Inorganic Data Review. October.

Parsons 2013. Phase 2 Facility Operations and Maintenance Plan for 2013. Hudson River PCBs Superfund Site. April 2013.

Tables

Table A-1
Decontamination-Related Sampling Types and Frequencies [1,2]

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan
General Electric Company – Hudson River PCBs Superfund Site

Item	Item Description	Sampling Method[3]	Sampling Frequency[16]
Equipment, Pumps, and Appurtenances			
Major processing equipment	Trommel screen, intermediate vibratory dewatering screen, triple-deck scalping screen, double-deck fine/mid screens, sand wheel screens, log washer, hydrocyclones, conveyors, filter presses, container handling system, air handling system blowers, vapor-phase GAC vessels, WTP clarifiers, WTP multi-media filters, WTP GAC vessels, WTP bag filter units	Wipe sampling	1 wipe sample per 300 square feet of surface area[4]
Unloading buckets	Clamshell unloading buckets	Wipe sampling	3 samples per bucket
Roll-off containers	Roll-off containers	Wipe sampling	1 wipe sample per 300 square feet of surface area[4]
Pumps	Slurry pumps, process/storm water pumps, etc.	Wipe sampling	2 wipe samples per pump
Other appurtenances	Motors, mixers, drives, gears, valves, meters, etc.	Wipe sampling	1 wipe sample for 25% of items[5]
Movable equipment	Sennebogen unloaders, front-end loaders, trucks, skid steers, etc.	Wipe sampling	1 wipe sample per 300 square feet of surface area[4] * Minimum of 3 wipe samples per item
Steel rails	Rail yard loading platform (Track #7)	Wipe sampling	1 wipe sample per 150 feet of steel rail
Tanks			
Sediment slurry tank	Size separation area	Wipe sampling (interior surfaces)	5 wipe samples per tank
Intermediate screen underflow tank	Size separation area	Wipe sampling (interior surfaces)	5 wipe samples per tank
Hydrocyclone underflow tanks	Size separation area	Wipe sampling (interior surfaces)	5 wipe samples per tank
Hydrocyclone overflow tanks	Size separation area	Wipe sampling (interior surfaces)	5 wipe samples per tank
Process water tanks	Size separation area	Wipe sampling (interior surfaces)	5 wipe samples per tank
Gravity thickeners	Thickening/dewatering area	Wipe sampling (interior surfaces)	12 wipe samples per tank
North gravity thickener flocculation tank	Thickening/dewatering area	Wipe sampling (interior surfaces)	5 wipe samples per tank
North gravity thickener overflow tank	Thickening/dewatering area	Wipe sampling (interior surfaces)	5 wipe samples per tank
Recycle water equalization tank	Thickening/dewatering area	Wipe sampling (interior surfaces)	10 wipe samples per tank
WTP 4th treatment train holding tank	Thickening/dewatering area	Wipe sampling (interior surfaces)	5 wipe samples per tank
WTP equalization tanks	Thickening/dewatering area	Wipe sampling (interior surfaces)	5 wipe samples per tank
WTP backwash holding tank	Thickening/dewatering area	Wipe sampling (interior surfaces)	5 wipe samples per tank
Frac tanks	Various locations	Wipe sampling (interior surfaces)	5 wipe samples per tank

Table A-1
Decontamination-Related Sampling Types and Frequencies [1,2]

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan
General Electric Company – Hudson River PCBs Superfund Site

Item	Item Description	Sampling Method[3]	Sampling Frequency[16]
Piping			
Steel piping	Slurry and water piping throughout the Facility	Wipe sampling (interior surfaces)	2 wipe samples per 150 linear feet for steel piping[6]
HDPE piping	HDPE piping removed and destined for off-site recycling	Wipe sampling (interior surfaces) or bulk material sampling (of shredded materials)	2 samples per 150 linear feet of whole piping or 1 composite sample per 100 cubic yards of shredded material[7]
Equipment Structure Steel Supports			
Steel equipment structural supports	North size separation equipment, filter press stands	Wipe sampling	1 wipe sample per 300 square feet of surface area[8]
Splash/spill plates/pans	Unloading wharf steel splash/spill plates/pans	Wipe sampling	1 wipe sample per 300 square feet of surface area[8]
Structures			
Dewatering (filter press) building	Metal structural members, metal siding (interior and exterior), and appurtenances (i.e., doors, overhead doors, overhead door lift mechanisms)	Wipe sampling	24 wipe samples from structural steel members/bracing[9] 24 wipe samples from interior metal siding[9] 24 wipe samples from inside appurtenances[9] 8 wipe samples from exterior metal siding[9]
Filter cake staging enclosures	Metal structural members and appurtenances (i.e., doors, overhead doors, overhead door lift mechanisms)	Wipe sampling	10 wipe samples per building from structural steel members/bracing[9] 6 wipe samples per building from inside appurtenances[9]
Water Treatment Plant (WTP) Building	Metal structural members and metal siding (interior and exterior)	Wipe sampling	4 wipe samples from structural steel members/bracing[9] 4 wipe samples from interior metal siding[9] 4 wipe samples from exterior metal siding[9]

Table A-1
Decontamination-Related Sampling Types and Frequencies [1,2]

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan
General Electric Company – Hudson River PCBs Superfund Site

Item	Item Description	Sampling Method[3]	Sampling Frequency[16]
Concrete/Asphalt Surfaces and Underlying Construction Materials (Items Planned to Remain In-Place)			
Concrete/asphalt pavement	Size separation area, material staging areas, exclusion area roads, rail yard loading platform, thickening/dewatering area, dewatering building concrete floor slab	Pulverized concrete core sampling[10]	1 core sample per 150-foot grid within the exclusion zone at the locations shown on Figure 3-1
Track #7 pre-cast concrete panels	Pre-cast concrete panels along Track #7	Pulverized concrete core sampling[10]	1 core sample per 150 feet of Track #7
Sub-base sand layer below concrete and asphalt	Areas where the overlying asphalt or concrete pavements have been removed	Bulk material sampling[12]	1 core sample per 150-foot grid where overlying asphalt or concrete pavements have been removed[13]
Sub-base gravel/stone below concrete and asphalt	Areas where the overlying asphalt or concrete pavements have been removed	Bulk material sampling[12]	1 core sample per 150-foot grid where overlying asphalt or concrete pavements have been removed[13]
Sub-base materials proximate to drainage structures and piping	Areas adjacent to drainage structures and piping	Bulk material sampling[12]	Locations to be determined in the field[14]
Concrete hydrocyclone overflow pump station	Size separation area	Pulverized concrete core sampling[10]	4 core samples from pump station concrete floor and walls
Concrete slurry force main encasement	Size separation area	Pulverized concrete core sampling[10]	3 core samples from floor
Concrete recycle water wet well	Thickening/dewatering area	Pulverized concrete core sampling[10]	4 core samples from wet well concrete walls and floor
Concrete filter cake staging enclosure bin walls	Material staging area	Pulverized concrete core sampling[10]	2 core samples along each wall
Concrete material staging area bin walls	Material staging area	Pulverized concrete core sampling[10]	2 core samples along each wall
Concrete/Asphalt Surfaces (Items Planned for Removal)			
Concrete lining from stormwater basins	North stormwater basin, south stormwater basin, waterfront stormwater basin	Composite sampling from crushed concrete staging piles[11]	1 composite sample per 500 cubic yards of crushed concrete
Crushed concrete/asphalt	Crushed concrete/asphalt from from various areas of the Facility (i.e., foundations, pads, pavement, and/or other areas)	Composite sampling from crushed concrete/asphalt staging piles[11]	1 composite sample per 500 cubic yards of crushed concrete/asphalt

Table A-1
Decontamination-Related Sampling Types and Frequencies [1,2]

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan
General Electric Company – Hudson River PCBs Superfund Site

Item	Item Description	Sampling Method[3]	Sampling Frequency[16]
Stormwater Drainage Structures/Piping			
Stormwater Drainage System	HDPE stormwater discharge piping to the south and south stormwater basins	Wet-weather stormwater flow sampling[15]	Two wet weather flow sampling events For each sampling event, one water sample will be collected from each stormwater outfall pipe that discharges to the stormwater basins.

Notes:

1. Decontamination and verification sampling activities will be performed where PCB-containing sediments have been processed, handled, and staged. Decontamination and sampling will not be required for surfaces that have not been exposed to PCB-containing materials. Throughout the demobilization processing, evaluations will be performed to determine the need and appropriate methods for decontamination and sampling based on the ultimate intended disposition of the items/materials. See Section 3.2 of this *Phase 2 Sediment Processing Facility Demobilization and Restoration Plan* (SPF Demobilization Plan).
2. Post-decontamination verification sampling may not be performed on items designated for off-site disposal as described in Section 3.2 of this SPF Demobilization Plan. If required, waste characterization sampling will be performed for disposal purposes.
3. When sampling a particular component, the sampling will focus on locations showing potential evidence of impacts (e.g., visible staining or residue) (if any) and locations where the greatest potential exposure to PCB-containing materials occurred (i.e., interior or exterior surfaces used for processing/handling PCB-containing sediment or where PCB-containing sediment accumulated during processing operations). In addition, additional waste characterization sampling will be performed if required for disposal purposes. Post-decontamination samples will be submitted for PCB analysis only.
4. Equipment items that are intended for potential reuse or salvage/recycling will be sampled based on the estimated surface area of the item. For sampling purposes, equipment surface areas (and the number of samples to be collected) will be estimated based on a “hypothetical box” with sufficient size to contain the item. For oddly shaped pieces, multiple “hypothetical boxes” may be used to estimate equipment surface area. The equipment surface areas will be estimated by summing the surface areas for all six sides of each “hypothetical box” used to “contain” the item. The total number of samples (inclusive of samples from exterior surfaces and interior surfaces, where applicable) to be collected from each item will then be determined based on the sampling frequency described above.
5. If designated for reuse or potential salvage/recycling, the sampling approach for small equipment items and appurtenances (e.g., motors, mixers, valves, drives, meters, sensors) will consist of sampling a representative number of items instead of sampling each individual item.
6. Steel piping, if designated for potential salvage/recycling, will be sampled on the interior surfaces of the piping for each 150 linear feet of piping. Sampling may also be performed to determine appropriate disposal methods.
7. HDPE piping will be sampled if designated for potential recycling. For pipe sections that remain whole, wipe sampling will be performed from interior pipe surfaces. For pipe sections that are shredded, bulk material composite samples will be collected for each approximately 100 cubic yards of shredded material. Sampling may also be performed to determine appropriate disposal methods.
8. Steel structural supports that were in contact with PCB-containing materials, process water, or stormwater (e.g., filter press stands, size separation equipment supports) will be sampled based on the estimated surface area of the item. For sampling purposes, surface areas (and the number of samples to be collected) will be estimated based on a “hypothetical box” with sufficient size to contain the item. The surface areas will be estimated by summing the surface areas for all six sides of each “hypothetical box” used to “contain” the item. The total number of samples (inclusive of samples from exterior surfaces and interior surfaces, where applicable) to be collected from each item will then be determined based on the sampling frequency described above.

Table A-1
Decontamination-Related Sampling Types and Frequencies [1,2]

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan
General Electric Company – Hudson River PCBs Superfund Site

9. Samples locations for the dewatering building, filter cake staging enclosures, and water treatment plant building will be determined in the field based on pre-sampling reconnaissance. Sampling of appurtenances in the dewatering building, filter cake staging enclosures, and water treatment plant building will only be performed on items that will remain in place or items planned for salvage/recycling. Additional sampling may be required depending on the sampling results.
10. The pulverized concrete and asphalt core samples will be collected to a depth of 7½ cm (or other depth agreed to by GE and EPA) from the upper concrete or asphalt surface in accordance with the SOPs in Attachment B of the SPF Demobilization FSP.
11. The concrete/geocell lining removed from the stormwater basins and concrete/asphalt removed from foundations, pads, pavement, and other areas will be crushed and placed in staging piles. Composite samples will be collected from the crushed concrete/asphalt at a frequency of 1 sample per 500 cubic yards. Fifteen discrete samples will be collected from each 500 cubic yard lot and composited into a single sample.
12. The bulk material samples (for sub-base materials) will be collected using a hand auger in accordance with the SOPs in Attachment C of the SPF Demobilization FSP.
13. The underlying sub-base stone and sand layers will be sampled only in areas where substantial sections of overlying asphalt or concrete pavements have been removed based on requests from the property owner and in areas where the full depth of overlying asphalt or concrete pavements has been removed to address PCB concentrations.
14. A representative number of subsurface samples will be collected proximate to underground drainage piping and structures. These subsurface sample locations will be determined in the field based on discussions with EPA.
15. After the final decontamination phase, wet-weather stormwater sampling will be performed to document whether PCBs are detected in the stormwater that drains through the system. Two wet-weather stormwater sampling events will be performed to verify the sampling results. During each sampling event, stormwater samples will be collected from each stormwater outfall pipe that discharges to the south and north stormwater basins and submitted for PCB analysis.
16. Additional sampling may be needed if conditions warrant or based on the results of prior sampling.

cm: centimeters
EPA: United States Environmental Protection Agency
FSP: Field Sampling Plan
GAC: granular activated carbon
PCBs: polychlorinated biphenyls
WTP: water treatment plant

Table A-2
Decontamination-Related Sampling – Sampling and Analysis Summary

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan
General Electric Company – Hudson River PCBs Superfund Site

Task	Sample Matrix	Laboratory Analysis[1]	Estimated Sample Quantity	Estimated QA/QC Sample Quantity				Estimated Total Samples
				Field Duplicates[2]	Equipment Blanks[3]	Trip Blanks	MS/MSD[4]	
Wipe Sampling	Wipe	PCBs	TBD[5]	1/20[5]	1/day/matrix	0	NA[6]	TBD[5]
Concrete/Asphalt Core Sampling	Solid	PCBs	134	7	1/day/matrix	0	7 / 7	155
Crushed Concrete/Asphalt Composite Sampling	Solid	PCBs	TBD[7]	1/20[7]	1/day/matrix	0	TBD[7]	TBD[7]
Sub-base Sand Bulk Sampling	Solid	PCBs	TBD[7]	1/20[7]	1/day/matrix	0	TBD[7]	TBD[7]
Sub-base Gravel Bulk Sampling	Solid	PCBs	TBD[7]	1/20[7]	1/day/matrix	0	TBD[7]	TBD[7]
HDPE Bulk Composite Sampling	Solid	PCBs	TBD[7]	1/20[7]	1/day/matrix	0	TBD[7]	TBD[7]
Stormwater Sampling Event #1	Water	PCBs	7	1	TBD	0	1 / 1	10
Stormwater Sampling Event #2	Water	PCBs	7	1	TBD	0	1 / 1	10

Notes:
1. See Table A-4 for the required analytical methods.
2. Field duplicate samples are to be collected at a rate of one duplicate sample per 20 samples (1/20). Wipe field duplicate samples will consist of co-located samples since it is not possible to collect true field duplicates for wipe samples.
3. Equipment blanks are to be collected at a rate of one equipment blank sample per 20 samples, or at least one sample per day per sample matrix (1/day/matrix).
4. MS/MSD samples are to be collected at a rate of one MS/MSD pair per 20 samples (1/20).
5. Sample quantities to be determined based on field measurements and the sampling frequencies presented on Table A-1.
6. Laboratory will perform laboratory control sample/laboratory control sample duplicate analyses for wipe samples since it is not possible to collect triplicate volumes for wipes.
7. Sample quantities to be determined in the field.

MS/MSD : matrix spike/matrix spike duplicate
NA: not applicable
PCBs: polychlorinated biphenyls
QA/QC: quality assurance/quality control
TBD: to be determined

Table A-3
Environmental Media – Sampling and Analysis Summary

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan
General Electric Company – Hudson River PCBs Superfund Site

Task	Sample Matrix	Laboratory Analysis[1]	Estimated Sample Quantity	Estimated QA/QC Sample Quantity				Estimated Total Samples
				Field Duplicates[2]	Equipment Blanks[3]	Trip Blanks[4]	MS/MSD[5]	
Surface Soil Sampling	Soil	PCBs	17	1	1/day/matrix	0	1 / 1	20
Soil Sampling	Soil	PCBs	20	1	1/day/matrix	0	1 / 1	23
		TCL SVOCs[6]	5	1	1/day/matrix	0	1 / 1	8
		TPH DRO[6]	7	1	1/day/matrix	0	1 / 1	10
		TPH GRO[6]	7	1	1/day/matrix	TBD	1 / 1	10
		STARS VOCs[7]	TBD	TBD	TBD	TBD	TBD	TBD
		STARS SVOCs[7]	TBD	TBD	TBD	0	TBD	TBD
Groundwater Sampling	Water	PCBs	12	1	1/day/matrix	0	1 / 1	15
		TCL VOCs[8]	12	1	1/day/matrix	1/cooler	1 / 1	15
		TCL SVOCs	12	1	1/day/matrix	0	1 / 1	15
		Pesticides	12	1	1/day/matrix	0	1 / 1	15
		TAL Metals	12	1	1/day/matrix	0	1 / 1	15
		Oil & Grease	12	1	1/day/matrix	0	1 / 1	15
Sediment Sampling	Soil	PCBs	11	1	1/day/matrix	0	1 / 1	14
		TOC	11	NA	1/day/matrix	0	1 / 1	13
Surface Water Sampling	Water	PCBs	4	1	1/day/matrix	0	1 / 1	7

Notes:
1. See Table A-4 for the required analytical methods.
2. Field duplicate samples are to be collected at a rate of one duplicate sample per 20 samples (1/20).
3. Equipment blanks will be collected at a rate of one equipment blank sample per 20 samples, and at least one sample per day per sample matrix (1/day/matrix).
4. Trip blanks will be analyzed for VOC parameters only at a rate of one per sample transport cooler (1/cooler).
5. MS/MSD samples are to be collected at a rate of one duplicate sample per 20 samples (1/20).
6. Analysis of soil samples for TCL SVOCs and TPH-DRO/TPH-GRO will be conducted for subsurface soil samples collected in the size separation area. Analysis for TPH-DRO/TPH-GRO will be conducted for subsurface soil samples collected from fuel storage areas.
7. Analysis of soil samples for STARS List VOCs and STARS List SVOCs will be conducted for subsurface soil samples collected from fuel storage areas and at other locations if elevated readings of volatile organic vapors are observed based on field photoionization detector screening.
8. TCL VOC analysis will include analysis for methyl tertiary butyl ether (MtBE).

DRO: diesel range organics
GRO : gasoline range organics
EPA: United States Environmental Protection Agency
MS/MSD : matrix spike/matrix spike duplicate
NA: Not applicable
PCBs: polychlorinated biphenyls
QA/QC: quality assurance/quality control
TPH: total petroleum hydrocarbons
STARS: New York State Department of Environmental Conservation Spills Technology and Remediation Series
SVOCs: semi-volatile organic compounds
TAL: target analyte list
TCL: target compound list
TOC: total organic carbon
VOCs: volatile organic compounds

Table A-4
Analytical Parameters and Methods

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan
General Electric Company – Hudson River PCBs Superfund Site

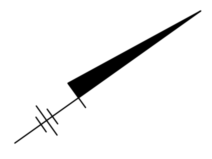
Parameter	Analytical Method
Wipe Samples, Concrete/Asphalt/Aggregate Samples	
PCBs	GEHR8082[1]
Soil Samples	
PCBs	GEHR8082[1]
TCL SVOCs[2]	SW-846 Method 8270D
STARS List VOCs[3]	SW-846 Method 8260C
STARS List SVOCs[3]	SW-846 Method 8270D
TPH DRO/GRO[2]	SW 846 Method 8015D
Sediment Samples	
PCBs	GEHR8082[1]
TOC	Lloyd Kahn Method
Groundwater Samples	
PCBs	SW-846 Method 8082A
TCL VOCs (including analysis for MtBE)	SW-846 Method 8260C
TCL SVOCs	SW-846 Method 8270D
Pesticides	SW-846 Method 8081B
TAL metals	SW-846 Method 6010C/7470B
Oil & grease	EPA Method 1664A HEM
Surface Water and Stormwater	
PCBs	SW-846 Method 8082A

Notes:

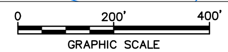
1. Method GEHR8082 analyses will be performed following the procedures in the Revised Attachment A (October 2014) to the Phase 2 RAM QAPP (Anchor QEA and ESI 2012), with the following exceptions: a) Performance Evaluation samples will not be prepared and analyzed as discussed in Section 11.2.1 of the Phase 2 RAM QAPP. Instead, matrix spike/matrix spike duplicate samples spiked with only Aroclor 1242 will be prepared and analyzed at a frequency of five percent of each matrix (i.e., one for every 20 samples); and b) Laboratory control spike will consist of only Aroclor 1242 instead of a combination of Aroclor 1221 and Aroclor 1242 at a ratio of 3:1 as discussed in Phase 2 RAM QAPP Revised Attachment A, Section 4.
2. Analysis of soil samples for TCL SVOCs (inclusive of nitrobenzene, 2,4-dinitrotoluene, and 2,6-dinitrotoluene) and TPH-DRO/GRO will be conducted for subsurface soil samples collected in the size separation area.
3. Analysis of soil samples for STARS List VOCs and STARS List SVOCs will be conducted for subsurface soil samples collected from fuel storage areas and at other locations if elevated readings of volatile organic vapors are observed based on field photoionization detector screening. STARS List constituents will include those listed in Tables 2 and 3 of the NYSDEC Final Commissioner Policy CP-51 Soil Cleanup Guidance (2010).

DRO: diesel range organics
EPA: United States Environmental Protection Agency
ESI: Environmental Standards, Inc.
GRO : gasoline range organics
MtBE: methyl tertiary butyl ether
NYSDEC: New York State Department of Environmental Conservation
PCBs: polychlorinated biphenyls
RAM QAPP: Remedial Action Monitoring Quality Assurance Project Plan
STARS: Spills Technology and Remediation Series
SVOCs: semi-volatile organic compounds
TAL: target analyte list
TCL: target compound list
TOC: total organic carbon
TPH: total petroleum hydrocarbons
VOCs: volatile organic compounds

Figures



RAIL SUPPORT BUILDING
RAIL YARD AREA
ACCESS GATE
TYPE II STORMWATER BASIN A
RAIL YARD LOADING PLATFORM (TRACK #7)
NORTH STORMWATER PUMP STATION
NORTH STORMWATER BASIN
RAILROAD SPUR (TRACK #8)
TYPE II STORMWATER BASIN B
WEIGH-IN-MOTION RAIL SCALE
RAIL YARD ACCESS ROAD
SOUTH STORMWATER BASIN
SOUTH EXTENSION ROAD
FILTER CAKE STAGING ENCLOSURES
COARSE MATERIAL STAGING AREA
NORTH EXTENSION ROAD
MAINTENANCE AREA
VEHICLE/EQUIPMENT DECON STATION
SITE PERSONNEL DECON STATION
CONSTRUCTION MANAGER TRAILERS
CONTRACTOR TRAILERS
ADMINISTRATIVE VEHICLE PARKING AREA
ACCESS GATE
MAIN HAUL ROAD
ABOVEGROUND FUEL STORAGE
ACCESS GATES
NORTH ACCESS ROAD
INTERSECTION SAFETY/DECON STATION
BOND CREEK
SECURITY STATION
SOUTH STORMWATER PUMP STATION
34.5 KV SUBSTATION
BACKWASH WATER HOLDING TANK
WATER TREATMENT PLANT
SLURRY FORCE MAIN ENCASEMENT
MAIN HAUL ROAD
RECYCLE WATER EQUALIZATION TANK
THICKENING/DEWATERING AREA
HYDROCYCLONES
HYDROCYCLONE PUMP STATION
HYDROCYCLONE SYSTEM
BOND CREEK
WATER FRONT STORMWATER PUMP STATION
LOCK DIVERSION CANAL
GRAVITY THICKENER TANKS NORTH AND SOUTH
VIBRATORY DEWATERING INTERMEDIATE SCREEN
LOCK DIVERSION CANAL
SIZE SEPARATION AREA
LOGWASHER SCREEN
SOUTH SERVICE ROAD
DEWATERING (FILTER PRESS) BUILDING
RECYCLE WATER WET WELL
TROMMEL SCREEN
ACCESS ROAD FOR EMERGENCY SERVICES
DECON STATION
DECK SCREENS
WATERFRONT STORMWATER BASIN
ABOVEGROUND FUEL STORAGE
FACILITY LIMITS
EAST STREET
WORK WHARF
PROCESS WATER TANK
SCALPING SCREEN
SAND WHEEL SCREENS
DREDGING OPERATIONS EQUIPMENT STAGING AREA
WORK WHARF CATWALK
CHAMPLAIN CANAL
UNLOADING WHARF (SOUTH)
PROCESS WATER TANK
UNLOADING WHARF (NORTH)
ACCESS GATES
GRAVEL PARKING AREA
SEDIMENT SLURRY TANK
LOCK 8
LOCK 8 ACCESS ROAD



LEGEND:

- APPROXIMATE EXTENT OF EXCLUSION ZONE
- APPROXIMATE EXCESS SOIL STAGING LOCATION
- PARCEL BOUNDARIES

NOTES:

1. PARCEL BOUNDARIES OBTAINED FROM WASHINGTON COUNTY IN JANUARY 2015.
2. EXCESS SOILS REMOVED DURING CONSTRUCTION OF THE FACILITY WERE STOCKPILED AT THE APPROXIMATE LOCATIONS SHOWN.

GENERAL ELECTRIC
HUDSON RIVER PCBS SUPERFUND SITE
PHASE 2 SEDIMENT PROCESSING FACILITY
DEMOBILIZATION AND RESTORATION FIELD SAMPLING PLAN

SEDIMENT PROCESSING FACILITY - FACILITY PLAN

FIGURE
A-1

CITY: SYRACUSE, NY DIV/GROUP: EBC-IM/DV DB/LD: L.POSENAUER PM: C.GUEST LYR:ON=*;OFF=*REF* PAGESETUP: ---- PLOTSTYLETABLE: ---- PLOTTED: 9/11/2015 10:06 AM BY: POSENAUER, LISA
V:\ENV\CAD\SYRACUSE\ACT\NB0031356\0000\00004\SPF_PH2DRFSP\31356B01.dwg LAYOUT: A-1 SAVED: 9/11/2015 10:05 AM
XREFS: IMAGES:
XGN-PL01
XGN-EX03
XGN-EX01
31356X00

GENERAL ELECTRIC
HUDSON RIVER PCBS SUPERFUND SITE
PHASE 2 SEDIMENT PROCESSING FACILITY
DEMOBILIZATION AND RESTORATION FIELD SAMPLING PLAN

SEDIMENT PROCESSING FACILITY - PROPOSED ASPHALT/CONCRETE PAVEMENT SAMPLING PLAN

LEGEND:

- APPROXIMATE EXTENT OF EXCLUSION ZONE
- PARCEL BOUNDARIES
- 150-FOOT SAMPLING GRID
- PROPOSED POST-DECONTAMINATION ASPHALT/CONCRETE PAVEMENT SAMPLING LOCATION

NOTES:

1. THIS FIGURE SHOWS ONLY THE PROPOSED POST-DECONTAMINATION ASPHALT/CONCRETE PAVEMENT SAMPLING LOCATIONS. POST-DECONTAMINATION SAMPLING LOCATIONS FOR CONCRETE WALLS, PITS, TRENCHES, PADS, AND FOUNDATIONS ARE NOT SHOWN ON THIS FIGURE.
2. LOCATIONS ARE APPROXIMATE AND MAY BE ADJUSTED IN THE FIELD.

ARCADIS

FIGURE
A-2

CITY: SYRACUSE, NY DIV/GROUP: EBC-IM/DV DB/LD: L.POSENAUER PM: C.GUEST LYR:ON=*;OFF=*REF*
V:\ENV\CADS\SYRACUSE\ACT\NB003135\6\0000\0004\SPF_PH2\DRFSP\31356P02.dwg LAYOUT: A-2 SAVED: 9/10/2015 4:21 PM PAGESETUP: ---- PLOTSTYLETABLE: ---- PLOTTED: 9/11/2015 8:53 AM BY: POSENAUER, LISA
XREFS: XGN-PL01 XGN-EX03 XGN-EX01 31356X00 IMAGES:

GENERAL ELECTRIC
HUDSON RIVER PCBS SUPERFUND SITE
PHASE 2 SEDIMENT PROCESSING FACILITY
DEMOBILIZATION AND RESTORATION FIELD SAMPLING PLAN

SEDIMENT PROCESSING FACILITY - PROPOSED ENVIRONMENTAL MEDIA SAMPLING PLAN

ARCADIS

FIGURE
A-3

LEGEND:

- APPROXIMATE EXTENT OF EXCLUSION ZONE
- PARCEL BOUNDARIES
- PROPOSED SURFACE SOIL SAMPLING LOCATION
- PROPOSED SOIL BORING LOCATION
- PROPOSED GROUNDWATER SAMPLING LOCATION
- PROPOSED SURFACE WATER SAMPLING LOCATION
- PROPOSED SEDIMENT SAMPLING LOCATION
- ABANDONED GROUNDWATER MONITORING WELL

NOTES:

1. THIS FIGURE SHOWS THE PROPOSED SAMPLING LOCATIONS FOR ENVIRONMENTAL SITE ASSESSMENT SAMPLING TO BE PERFORMED AFTER FACILITY DECONTAMINATION.
2. LOCATIONS ARE APPROXIMATE AND MAY BE ADJUSTED IN THE FIELD.

0 150' 300'
GRAPHIC SCALE

Attachments

Attachment A –

Standard Operating Procedure:

Wipe Sample Collection

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Standard Operating Procedure:
Wipe Sample Collection

Hudson River Sediment Processing Facility
Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 1

Rev Date: February 20, 2015

I. Introduction

This document describes procedures for collection of surface wipe samples from non-painted and painted surfaces.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading sampling activities should have a minimum of 5 years of previous environmental sampling experience. ARCADIS personnel providing assistance to sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences.

III. Equipment / Materials List

The following materials will be available, as required, during wipe sampling activities:

- personal protective equipment (PPE), as specified by the site Health and Safety Plan (HASP)
- latex/nitrile protective gloves
- 10-centimeter (cm) by 10-cm paper templates
- Hexane-moistened gauze pads provided by the analytical laboratory
- sample vials
- marker or paint pen
- transport containers with ice (sample storage and shipping for laboratory analysis)
- documentation forms and notebooks, including chain-of-custody forms, sample labels and seals, and field logbook

IV. Cautions / Hazards

Task-specific Job Safety Analysis (JSAs) identifying site hazards and controls associated with the sampling activities must be reviewed by all field crew members prior to the start of work. Safe Performance Self-Assessment (SPSA) to be performed by employees before performing a new task.

The work will be performed in coordination with the Construction Manager (Parsons) to minimize disturbance to ongoing facility operations and to provide a clear sampling area.

Before any sampling, potential energy sources (including electrical and mechanical) must be de-energized, disconnected, and disabled, as applicable, in accordance with the project's Energy Control and Lockout Procedures.

V. Health and Safety Considerations

Sample collection will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task specific JSA forms, copies of which will be present on site during such activities.

Review the material safety data sheet (MSDS) for the solvent (i.e., hexane) used in wipe sampling. Work in a well-ventilated area and stand upwind while applying solvent to sample area.

VI. Sampling Procedures

1. Proper personal safety equipment shall be worn in accordance with the site HASP.

2. Lockout/Tagout: Before sampling of any structures, equipment, or near equipment, all potential energy sources (including electrical and mechanical) must be de-energized, disconnected, and disabled, as applicable, in accordance with the project's Energy Control and Lockout Procedures.

3. Coordinate sampling work with the Construction Manager and other contractors working in the area. Prior to sample collection, confirm that the surface has been decontaminated (as applicable) in accordance with the approved demobilization and restoration plan.

4. Determine the number of samples required for each structure or equipment item based on the sampling frequency and locations described in the Field Sampling Plan (FSP). This may require the collection of field measurements and/or take-off measurements from equipment/structure drawings.

5. Put on clean latex/nitrile protective gloves for each new sample collection.

6. Place paper template with 10-cm x 10-cm cutout on the surface to be sampled.

7. Using hexane-soaked gauze pad, wipe the entire cutout area from one side to the opposite side. Refold the gauze pad and wipe from one side to the opposite side of entire cutout area in a direction perpendicular to the first wipe.

8. Place sample gauze pad into a pre-labeled sample vial.

9. Remove protective gloves while holding template and dispose of used gloves and template into appropriate container.

10. Repeat procedure for total number of samples required.

11. Complete chain-of-custody forms, prepare shipping containers, and send samples to the laboratory for analysis.

12. Sample observations will be recorded in a dedicated field log book and photographs will be taken.

13. Mark the four corners of the paper template onto the sample surface using a paint pen or marker or other appropriate means. Mark the sample ID above or next to the template corner markings.

14. Mark the sampling locations on a sketch, figure, or drawing of the item sampled.

VII. Waste Management

All investigation-derived waste (IDW) generated by the sampling activities (e.g., sampling supplies, PPE, etc.) will be transferred to appropriate DOT-approved waste containers for management and treatment/disposal by others.

Waste containers must be a sealed and labeled at the time of generation. Labels will indicate date, sample locations, site name, city, state, and description of the matrix (e.g., media, PPE).

VIII. Data Recording and Management

Field documentation entries and chain-of-custody records will be documented in accordance with the FSP. Chain-of-custody forms will be electronically transmitted each day. The field team leader will retain all site documentation while in the field and add to project files when the field mobilization is complete.

IX. Quality Assurance

Quality assurance samples (duplicates and MS/MSDs) will be collected at the frequency specified in the FSP. Any deviations from the SOP will be discussed with the project manager prior to changing any field procedures.

Attachment B –

Standard Operating Procedure:

Concrete / Asphalt Sampling

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Standard Operating Procedure:
Concrete / Asphalt Sampling

Hudson River Sediment Processing Facility
Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 2

Rev Date: February 20, 2015

I. Introduction

This document describes procedures for collection of pulverized concrete and asphalt samples at the Hudson River Sediment Processing Facility.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading sampling activities should have a minimum of 5 years of previous environmental sampling experience. ARCADIS personnel providing assistance to sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences.

III. Equipment / Materials List

The following equipment will be available, as required:

- personal protective equipment (PPE), as specified by the site Health and Safety Plan (HASP)
- paper plate
- latex/nitrile protective gloves
- hammer drill with ¾-inch coring machine bit
- generator with ground fault and electrical cords
- shop vacuum
- tape measurer
- equipment decontamination supplies
- sample bottles appropriate for the parameters to be sampled for laboratory analysis
- transport containers with ice (sample storage and shipping for laboratory analysis)
- documentation forms and notebooks, including excavation permit forms, chain-of-custody forms, sample labels and seals, and field logbook
- disposable aluminum pans

- sand bags
- orange safety cones
- spill kit
- fast-setting concrete mix
- white spray paint

IV. Cautions / Hazards

Task-specific Job Safety Analysis (JSAs) identifying site hazards and controls associated with the sampling activities must be reviewed by all field crew members prior to the start of work. Safe Performance Self-Assessment (SPSA) to be performed by employees before performing a new task.

The work will be performed in coordination with the Construction Manager (Parsons) to minimize disturbance to ongoing facility operations and to provide a clear sampling area.

Underground utilities will be cleared per the ARCADIS Utility Location Policy and Procedure and under the GE Project Excavation Permit utility clearance procedures.

V. Health and Safety Considerations

Sample collection will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task specific JSA forms, copies of which will be present on site during such activities.

VI. Sample Collection Procedure

1. Proper personal safety equipment shall be worn in accordance with the site HASP.
2. Coordinate sampling work with the Construction Manager and other contractors working in the area.
3. Obtain an approved Excavation Permit from the Construction Manager for each work area prior to starting sampling efforts.
4. Conduct final utility review of the sampling area, available drawings, and the approved Excavation Permit prior to sampling.

5. Set up orange safety cones (or other appropriate materials) around sampling area. Where possible, arrange the field vehicle in a manner to act as a barrier from adjacent traffic areas.

6. Prior to sample collection, confirm that the surface has been decontaminated (as applicable) in accordance with the approved plan.

7. If needed, place sand bags or other appropriate materials around the sampling area to prevent water from entering the area.

8. Make sure the surface is free of standing water at time of sampling.

9. Decontaminate drill bits in accordance with the protocols presented in Field Equipment Decontamination SOP.

10. Put on clean latex/nitrile protective gloves for each new sample collection.

11. Concrete/asphalt core samples will be collected as follows:

    a. Place disposable paper plate with center hole cut out over determined sample location. If sampling vertical surfaces, position a paper plate or aluminum tray under the sample collection point to collect the concrete/asphalt drill cuttings.

    b. Using a hammer drill with a pre-cleaned ¾-inch drill bit, drill the concrete/asphalt surface to the prescribed sample depth.

    c. Concrete and asphalt drill cuttings should be uplifted and deposited on the disposable paper template.

    d. Pour the collected sample material into an aluminum pan.

    e. Drill multiple holes at the sample locations as needed to obtain the required sample volume. A minimum sample size of 100 grams will be collected for locations where sample size is limited (about 25 grams is necessary for the PCB and moisture analysis). This minimum sample size allows reanalysis by the laboratory if necessary.

    f. Homogenize the sample in the aluminum pan and place into a pre-labeled laboratory-supplied sample container.

    g. Close, cap, seal, and label the sample container.

    h. Remove and dispose of protective gloves in appropriate waste container.

i. Repeat sample procedure for total number of samples required.

12. A shop vacuum will be used to remove residual dust and debris generated by the sample collection activities.

13. Complete chain-of-custody forms, prepare shipping containers, and send samples to the laboratory for analysis.

14. Where designated, collect subsurface construction media samples in accordance with the Subsurface Construction Media Sampling Using Manual Methods SOP.

15. Fill the core holes within the concrete/asphalt surface using a fast-setting concrete mix.

16. Mark the coring location with orange safety cones to allow for curing.

17. Sample observations will be recorded in a dedicated field log book and photographs will be taken.

18. Mark the sample locations and IDs using white spray paint, or other appropriate means.

19. Mark the sampling locations on a site figure/drawing.

20. Obtain locational coordinate information for each sample location using a hand-held global positioning satellite (GPS) unit. For locations inside buildings, obtain tie-off field measurements of the sample location. For sample locations that are accessible at the completion of the sampling program, survey the sample location using either a survey-grade GPS system or by conventional survey methods.

VII. Waste Management

All investigation-derived waste (IDW) generated by the sampling activities (e.g., core cuttings, sampling supplies, decontamination rinsate, PPE, etc.) will be transferred to appropriate DOT-approved waste containers for management and treatment/disposal by others.

Waste containers must be a sealed and labeled at the time of generation. Labels will indicate date, sample locations, site name, city, state, and description of the matrix (e.g., media, PPE).

VIII. Data Recording and Management

Field documentation entries and chain-of-custody records will be documented in accordance with the Field Sampling Plan. Chain-of-custody forms will be electronically transmitted each day. The field team leader will retain all site documentation while in the field and add to the project files when the field mobilization is complete.

IX. Quality Assurance

Quality assurance samples (duplicates and MS/MSDs) will be collected at the frequency specified in the Field Sampling Plan (FSP). Reusable sampling equipment will be cleaned prior to use following field equipment decontamination SOP. Field equipment blanks will be used to confirm that decontamination procedures are sufficient and samples are representative of site conditions. Any deviations from the SOP will be discussed with the project manager prior to changing any field procedures.

Attachment C –

Standard Operating Procedure:

Subsurface Media Sampling Using Manual Methods

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Standard Operating Procedure:
Subsurface Media Sampling Using Manual Methods

Hudson River Sediment Processing Facility
Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 3

Rev Date: September 10, 2015

I. Introduction

This document describes procedures for collection of subsurface media samples (e.g., Type F run-of-crusher sub-base stone and Type D washed sand) using hand tools.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading media sampling activities should have a minimum of 5 years of previous environmental sampling experience. ARCADIS personnel providing assistance to media sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences.

III. Equipment / Materials List

The following materials will be available, as required, during media sampling activities:

- personal protective equipment (PPE), as specified by the site Health and Safety Plan (HASP)
- disposable aluminum pans
- disposable wooden tongue depressors
- metal landscaping bar
- stainless steel hand augers
- indelible ink pens
- ruler
- sealable plastic bags (e.g., Ziploc®)
- electric concrete core drill machine with stand
- dry-bit concrete/asphalt core barrels
- garden sprayer
- generator with ground fault and electrical cords

- shop vacuum
- equipment decontamination supplies
- sample bottles appropriate for the parameters to be sampled for laboratory analysis
- transport containers with ice (sample storage and shipping for laboratory analysis)
- documentation forms and notebooks to have on hand include: excavation permit forms, chain-of-custody forms, sample labels and seals, field logbook

IV. Cautions / Hazards

Task-specific Job Safety Analysis (JSAs) identifying site hazards and controls associated with the sampling activities must be reviewed by all field crew members prior to the start of work. Safe Performance Self-Assessment (SPSA) to be performed by employees before performing a new task.

The work will be performed in coordination with the Construction Manager (Parsons) to minimize disturbance to ongoing facility operations and to provide a clear sampling area.

Underground utilities will be cleared per the ARCADIS Utility Location Policy and Procedure and the GE Project Excavation Permit Utility Clearance Procedures.

V. Health and Safety Considerations

Media sample collection will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task specific JSA forms, copies of which will be present on site during such activities.

VI. Sample Collection Procedures

Media samples will be collected at intervals from below the asphalt/concrete surface cover to designated depths to sample underlying materials above the flexible membrane liner (e.g., Type F run-of-crusher stone and Type D washed sand).

1. Prior to sample collection, review the Facility site construction record drawings to verify the types of materials and thicknesses for each subsurface layer to be sampled. If needed, adjust the sampling procedures accordingly.

2. Proper personal safety equipment shall be worn in accordance with the site HASP.

3. Clean sample gloves will be worn during all sample collection/handling procedures and gloves will be changed out between all samples.

4. Reusable sample equipment that will come in contact with the media sample will be cleaned in accordance with the field equipment decontamination SOP.

5. Where concrete or asphalt is present at the surface, use a core drill machine with dry bit cores to remove a 3-inch or 4-inch diameter plug of the asphalt or concrete construction surface. Remove generated dust and debris using a shop vacuum.

6. If necessary, to keep the dry core bit cool, periodically spray the bit with water using a garden sprayer. Remove generated water using a shop vacuum.

7. Samples of the “Type F” fill subbase material will be manually collected beneath the concrete/asphalt cover using a hand auger. A metal landscaping bar will be used to break-up the “Type F” fill subbase material to allow for sample collection using a hand auger.

8. A sharpened inner rod will be used to puncture the geotextile fabric under the subbase material.

9. Samples of the “Type D” washed sand will then be manually collected using a hand auger. Profile depths of each sample location will be measured with a ruler to obtain depth measurements of the sample intervals.

10. Record field collection information in the field log book.

11. Respective sample depth intervals will be emptied into separate disposable aluminum sampling pans for visual characterization prior to filling sample jars.

12. Sample observations will be recorded in a dedicated field log book and photographs will be taken.

13. After sample collection is completed, the resulting core holes will be filled using a fast-setting concrete mix and sealed at the surface with a fast-drying epoxy coating.

14. Coring locations will be marked with orange cones for a minimum of 8 hours to allow for curing.

VII. Sample Characterization and Processing Procedures

1. Collected media samples will be checked for sample IDs and documentation accuracy.

2. The sample media will be visually observed and logged by the field representatives according to general soil classifications. Other observations, including staining, sheens, odors, or moisture will be documented, if observed.

3. Samples will be sectioned into the following discrete sample intervals: Type F Run-of-Crusher Stone and Type D washed sand. The sample materials will be placed into disposable aluminum pans for homogenization using disposable wooden tongue depressors.

4. The homogenized samples will be placed in laboratory-supplied sample containers. The sample containers will be labeled with the sample identification number, sample date, and time of collection; and stored on ice.

5. Field personnel will collect and document the appropriate quality control samples in accordance with the Field Sampling Plan (FSP).

6. Complete chain-of-custody forms, prepare shipping containers, and send samples to the laboratory for analysis.

VIII. Waste Management

All investigation-derived waste (IDW) generated by the sampling activities (e.g., core cuttings, sampling supplies, decontamination rinsate, PPE, etc.) will be transferred to appropriate DOT-approved waste containers for management and treatment/disposal by others.

Waste containers must be a sealed and labeled at the time of generation. Labels will indicate date, sample locations, site name, city, state, and description of the matrix (e.g., media, PPE).

IX. Data Recording and Management

Field documentation entries and chain-of-custody records will be documented in accordance with the Field Sampling Plan. Chain-of-custody forms will be electronically transmitted each day. The field team leader will retain all site documentation while in the field and add to the project files when the field mobilization is complete.

X. Quality Assurance

Quality assurance samples (equipment blanks, duplicates, and MS/MSDs) will be collected at the frequency specified in the FSP and depending on the project quality objectives. Reusable media sampling equipment will be cleaned prior to use following field equipment decontamination SOP. Field equipment blanks will be used to confirm that decontamination procedures are sufficient and samples are

representative of site conditions. Any deviations from the SOP will be discussed with the project manager prior to changing any field procedures.

Attachment D –

Standard Operating Procedure:

Soil Boring Installation and Soil Sampling

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Standard Operating Procedure:

Soil Boring Installation and Soil Sampling

Hudson River Sediment Processing Facility

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 1

Rev Date: February 20, 2015

I. Introduction

This standard operating procedure (SOP) describes the field sampling procedures for installation of soil borings and collection of soil samples.

Subsurface soil samples will be collected from soil borings that will be advanced using direct-push technology (DPT) sampling methods (e.g., Geoprobe®-type rig). The method employed will be generally consistent with ASTM D-6282/D-6282M - *Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations*.

Surface soil samples will be collected using a hand auger sampling device that is manually advanced.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading sampling activities should have a minimum of 5 years of previous environmental sampling experience. ARCADIS personnel providing assistance to sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences.

III. Equipment / Materials List

The following equipment/materials, as required, shall be available during soil sampling:

- personal protective equipment (PPE), as specified by the site *Health and Safety Plan* (HASP)
- equipment decontamination supplies, as described in the Field Equipment Decontamination SOP
- drilling equipment required by ASTM D-6282/D-6282M
- appropriate sample containers and forms
- coolers with ice or “blue” ice
- photoionization detector (PID)
- sealable plastic bags (e.g., Ziploc®)
- field notebook
- stainless steel hand auger

IV. Cautions / Hazards

The work will be performed in coordination with the Construction Manager (Parsons) to minimize disturbance to ongoing facility operations and to provide a clear sampling area.

Underground utilities will be cleared per the ARCADIS Utility Location Policy and Procedure and under the GE Project Excavation Permit utility clearance procedures.

V. Health and Safety Considerations

Media sample collection will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task specific JSA forms, copies of which will be present on site during such activities.

VI. Sample Collection Procedures

Subsurface Soil Sampling

1. Proper personal safety equipment shall be worn in accordance with the site HASP.
2. Clean sample gloves will be worn during all sample collection/handling procedures and gloves will be changed out between all samples.
3. Reusable sample equipment that will come in contact with the media sample will be cleaned in accordance with the field equipment decontamination SOP.
4. Locate boring location, and if necessary, core through asphalt or concrete surface as specified in the Subsurface Media Sampling SOP, to provide access to underlying soil.
5. Advance soil boring to depth specified in the work plan.
6. After the Macro-Core® disposable acetate liners are recovered from the direct-push rig, cut the liner to expose the soil core for visual characterization. Qualified personnel will describe each soil sample in the field notebook. Additional information regarding procedures to identify soil types may be found in ASTM D2488 – Standard Practice for Description and Identification of Soils (Visual – Manual Procedures). Soil descriptions will be entered in the field notebook or on a subsurface log for the following parameters:
   - soil type;
   - color;
   - percent recovery;
   - moisture content;
   - texture;
   - grain size and shape;

- consistency;
- blow counts, if collected; and
- miscellaneous observations.

A common soil sample description format will be utilized in the field notes, such as: color; primary constituent (underlined or capitalized); secondary constituent(s) designated by “and” (if approximately 50% of the sample, should only be utilized if a second primary constituent is identified), “some” (if approximately 30% to 50% of the sample), “little” (if approximately 10% to 30% of the sample), and/or “trace” (if less than 10% of the sample); description of consistency; moisture content; miscellaneous observations; and initial interpretations (capitalized in parentheses).

Example 1: Brown fine SAND, some Silt, little medium-coarse Sand, trace concrete and brick debris, loose, wet, trace black staining. (FILL).

Example 2: Olive-gray SILT and CLAY, trace fine Gravel, angular dense, moist. (GLACIAL TILL).

In addition, the boring logs must identify the specific depth of the fill/native soil interface (if present) and will provide a detailed description of any debris observed in the fill. Observations of staining, sheens, or other potential indicators of impacted soil should also be described in detail, including the starting and ending depths of such observations.

Each sampling interval will be recorded based on any spaces or gaps and the recovery of the core. For example, if a soil sampler is advanced from 4 to 8 feet below ground surface (bgs), but the soil recovery is only 2.5 feet, the log will indicate that the description only applies to 4 to 6.5 feet bgs (i.e., assume that lower portion of the soil sample was not recovered) unless reasons to infer otherwise are evident in the sample or adjacent samples. In the event that insufficient sample recovery (i.e., consistently less than 50%) is encountered, the sampling will continue with the 4-foot Macro-Core® device, but the sampling intervals will be reduced to 2 feet.

7. Collect representative samples of soil from the soil core at appropriate intervals identified in the work plan, and place in labeled laboratory sample containers.
8. In accordance with the work plan, obtain split samples for screening with the PID for VOCs using the procedures set forth in the PID Field Screening SOP.
9. If PID screening indicates the presence of volatile compounds, collect a representative sample from the corresponding soil core interval and submit for additional analysis in accordance with the Field Sampling Plan (FSP).

10. Where applicable, backfill the boring location with a bentonite/cement grout and restore the concrete/asphalt surface with a fast-setting concrete mix. Mark with orange cones to allow for curing.
11. The samples will be placed in laboratory-supplied sample containers. The sample containers will be labeled with the sample identification number, sample date, and time of collection; and stored on ice.
12. Field personnel will collect and document the appropriate quality control samples in accordance with the FSP.
13. Complete chain-of-custody forms, prepare shipping containers, and send samples to the laboratory for analysis.

Surficial Soil Sampling

1. If the sample location is a grassed area or an area that exhibits overlying material (i.e., gravel, rocks, leaves, roots), the sod or overlying material should be removed and the underlying soil should be collected. The sod refers to the grass and dense root matter below the grass, including the soil within the dense root matter. Replace the sod following sample collection.
2. Using the stainless steel hand auger, secure a representative sample from the appropriate depth identified in the FSP.
3. Place a representative sample into a labeled laboratory-provided sample jar.
4. Record a soil sample description in the field notes, consistent with the description procedures identified in the subsurface sampling methods above.
5. Restore the surface soil sample location to conditions similar with the immediate surrounding ground surface.
6. The samples will be placed in laboratory-supplied sample containers. The sample containers will be labeled with the sample identification number, sample date, and time of collection; and stored on ice.
7. Field personnel will collect and document the appropriate quality control samples in accordance with the FSP.
8. Complete chain-of-custody forms, prepare shipping containers, and send samples to the laboratory for analysis.

VII. Field Cleaning

Cleaning of sampling equipment is to follow the procedures described in the Field Equipment Decontamination SOP. The sampling equipment is to be cleaned prior to the start of sampling activities, between samples, and following the completion of sampling activities. In addition, tools utilized in the handling and opening of sampling equipment, such as knives for cutting direct-push sample liners, are to be cleaned with

non-phosphate soap and water prior to the start of sampling activities, between boreholes, and following the completion of sampling activities, at a minimum.

VIII. Waste Management

All investigation-derived waste (IDW) generated by the sampling activities (e.g., core cuttings, sampling supplies, decontamination rinsate, PPE, etc.) will be transferred to appropriate DOT-approved waste containers for management and treatment/disposal by others.

Waste containers must be a sealed and labeled at the time of generation. Labels will indicate date, sample locations, site name, city, state, and description of the matrix (e.g., media, PPE).

IX. Data Recording and Management

Field documentation entries and chain-of-custody records will be documented in accordance with the Field Sampling Plan. Chain-of-custody forms will be electronically transmitted each day. The field team leader will retain all site documentation while in the field and add to the project files when the field mobilization is complete.

X. Quality Assurance

Quality assurance samples (duplicates and MS/MSDs) will be collected at the frequency specified in the Field Sampling Plan (FSP). Reusable sampling equipment will be cleaned prior to use following field equipment decontamination SOP. Field equipment blanks will be used to confirm that decontamination procedures are sufficient and samples are representative of site conditions. Any deviations from the SOP will be discussed with the project manager prior to changing any field procedures.

Field duplicates will be prepared by homogenizing soil collected at the same time and depth and then filling two sets of sample jars. For VOCs, the samples will be collected as close as practical to the original VOC sample depth and will not be homogenized prior to placement in the sample jars. The duplicate sample will be labeled in such a way that the sample designations will not indicate the duplicate nature of the samples. Information concerning the source of sample duplicates should be documented in the field notebook and on the version of the COC form that is retained by the sampling team. This information should NOT be provided in the copy of the COC form that is submitted to the laboratory.

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Attachment E –

Standard Operating Procedure:

Monitoring Well Development

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Standard Operating Procedure:

Monitoring Well Development

Hudson River Sediment Processing Facility

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 1

Rev Date: February 20, 2015

I. Introduction

Standard operating procedures (SOPs) for developing overburden groundwater monitoring wells are presented in this Appendix. Monitoring wells that yield water will be developed (i.e., cleared of fine-grained materials and sediments) so that the well screen is transmitting groundwater representative of the surrounding formation groundwater. Development will be accomplished by surging (using a surge block, where possible) and evacuating well water by either pumping or bailing. Acceptable pumping methods include the use of the following (presented in order of preferred use): electric submersible pump; peristaltic pump; and surface inertial pump (Waterra™ pump).

After the wells have been developed, the wells will be allowed to stabilize for a minimum of one week prior to groundwater sampling.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading sampling activities should have a minimum of 5 years of previous environmental sampling experience. ARCADIS personnel providing assistance to sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences.

III. Equipment / Materials List

Equipment and materials for monitoring well development include:

- personal protective equipment (PPE), as specified by the site Health and Safety Plan (HASP)
- cleaning equipment, as described in the Field Equipment Decontamination SOP
- polyethylene tubing (discarded between well locations) (for development using a pump)
- power source (generator or battery) (for development using a pump)
- pump (electric submersible pump; peristaltic pump; and/or surface inertial pump [Waterra™ pump]) (for development using a pump)
- bottom-loading bailer (for development using a bailer)

- polypropylene rope (for development using a bailer)
- field notebook
- graduated pails
- water level probe
- water quality (turbidity/temperature/pH/specific conductivity) meter
- appropriate containers
- monitoring well keys

IV. Cautions / Hazards

Task-specific Job Safety Analysis (JSAs) identifying site hazards and controls must be reviewed by all field crew members prior to the start of work. Safe Performance Self-Assessment (SPSA) to be performed by employees before performing a new task.

V. Health and Safety Considerations

Procedures will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task specific JSA forms, copies of which will be present on site during such activities.

VI. Monitoring Well Development Procedures

The procedures for monitoring well development using a pump are described below:

1. Don appropriate PPE, as required by the HASP.

2. Clean equipment entering each monitoring well (i.e., water level probe) as described in the Field Equipment Decontamination SOP. New tubing will be used for each well.

3. Measure the depth to water and total depth of well using a water level probe. Calculate the volume of water in the well (in gallons) by using the length of the water column (in feet), multiplying by 0.163 for a 2-inch well, or by 0.653 for a 4-inch well. For other well diameters, use the formula:

Volume (in gallons) = π TIMES well radius (in feet) squared TIMES length of water column (in feet) TIMES 7.481 (gallons per cubic foot)

4. A surge block will be lowered into the screened portion of the well on a rigid pipe or high density tubing and cycled up and down to force water in and out of the screen slots and formation. After surging along the entire length of the well screen, formation water will be removed by pumping or bailing along the entire length of screen interval. Surging and pumping will be performed until a minimum of three well volumes of water have been removed. Contain all water in appropriate containers.

5. If well runs dry, shut off pump and allow well to recover.

6. Measure field parameters (i.e., temperature, pH, specific conductivity, and turbidity) after removal of each well volume, or approximately every 15 minutes and record in field notebook. Development is considered complete after removal of a minimum of three well volumes and if the purge water is relatively clear of silt (i.e., turbidity less than 50 NTU). If the turbidity levels do not decrease to below 50 NTU after surging and pumping five well volumes, development will be considered complete if the other field indicator parameters have stabilized to within 10% over three consecutive measurement intervals.

7. When complete, collect a final water level and well depth measurement and secure the lid back on the well.

8. Place tubing in plastic bags for appropriate disposal, and clean pump as described in the Field Equipment Decontamination SOP.

The procedure for developing a well using the bailer method is outlined below:

1. Don appropriate PPE, as required by the HASP.

2. Stainless steel bailers will be cleaned as described in the Field Equipment Decontamination SOP.

3. Measure the depth to water and total depth of well. Calculate the volume of water in the well (in gallons) by using the length of the water column (in feet), multiplying by 0.163 for a 2-inch well, or by 0.653 for a 4-inch well. For other well diameters, use the formula:

   Volume (in gallons) = π TIMES well radius (in feet) squared TIMES length of water column (in feet) TIMES 7.481 (gallons per cubic foot)

4. Measure a length of rope at least 10 feet greater than the total depth of the well. Secure one end of the rope to the well casing, secure the other end of the rope to the bailer. Test the knots and make sure the rope will not loosen. Check bailers to be sure all parts are intact and will not be lost in the well.

5. Lower bailer into well until bailer reaches the bottom of the well.

6. Surge/purge by raising and lowering the bailer at 2-foot intervals, at least 10 times.

7. Remove approximately one well volume of water from the well. Contain all water in appropriate containers. Measure field parameters (i.e., temperature, pH, specific conductivity, and turbidity) and record in field notebook.

8. Lower bailer back into the well and repeat surging/purging at an interval 2 feet above the previous interval. Repeat Step 7 and Step 8 until entire screen has been surged. If well is bailed dry, allow well to recover.

9. Continue to remove groundwater until a minimum of three well volumes of water have been removed from the well. Measure field parameters (i.e., temperature, pH, specific conductivity, and turbidity) after removal of each well volume, or approximately every 15 minutes and record in field notebook. Development is considered complete after removal of a minimum of three well volumes and if the purge water is relatively clear of silt (i.e., turbidity less than 50 NTU). If the turbidity levels do not decrease to below 50 NTU after surging and bailing five well volumes, development will be considered complete if the other field indicator parameters have stabilized to within 10% over three consecutive measurement intervals.

10. Upon completion of development of the well, remove bailer and remove the rope from the bailer and the well. Collect a final water level and well depth measurement.

11. Secure lid on well.

12. Place polypropylene rope in plastic bags for appropriate disposal and clean bailer as described in the Field Equipment Decontamination SOP.

VII. Waste Management

All investigation-derived waste (IDW) generated (e.g., purge water, tubing, PPE, etc.) will be transferred to appropriate DOT-approved waste containers for management and treatment/disposal by others.

Waste containers must be a sealed and labeled at the time of generation. Labels will

indicate date, sample locations, site name, city, state, and description of the matrix (e.g., media, PPE).

VIII. Data Recording and Management

Field documentation entries will be documented in accordance with the Field Sampling Plan. The field team leader will retain all site documentation while in the field and add to the project files when the field mobilization is complete.

Attachment F –

Standard Operating Procedure:

Water Level Measurement Procedures

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Standard Operating Procedure:
Water Level Measurement Procedures

Hudson River Sediment Processing Facility
Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 1

Rev Date: February 20, 2015

I. Introduction

Standard operating procedures (SOPs) for determining water levels in monitoring wells are presented in this Appendix.

II. Equipment / Materials

- health and safety equipment, as required by the site Health and Safety Plan (HASP)
- cleaning equipment, as described in the Field Equipment Decontamination SOP
- nitrile gloves
- water level probe
- watch (to record time and day)
- field notebook
- appropriate log forms
- monitoring well keys
- paper towels or wipes

III. Cautions / Hazards

Task-specific Job Safety Analysis (JSAs) identifying site hazards and controls associated with the sampling activities must be reviewed by all field crew members prior to the start of work. Safe Performance Self-Assessment (SPSA) to be performed by employees before performing a new task.

IV. Health and Safety Considerations

Procedures will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task specific JSA forms, copies of which will be present on site during such activities.

V. Procedures

1. Review the operating and maintenance instruction manual for the probe prior to commencement of work.

2. Identify site and well number on Water Level Monitoring Field Log and/or field notebook, along with other appropriate information collected during water level measurement.

3. Don personal protective equipment (PPE), as required by the HASP.

4. Clean the water level probe and cable in accordance with the cleaning procedures described in the Field Equipment Decontamination SOP.

5. Remove well cap. Locate a measuring reference point on the well casing. If one is not found, initiate a reference point by notching the inner casing with a hacksaw or by using a waterproof marker. All down-hole measurements will be taken from the reference point. The acronym TOC will designate the top of casing. If a well has both inner and outer casings, use the top of the inner casing as the reference point.

   Note: The following steps describe the procedures for water level measurement. For wells subject to routine monitoring (e.g., monthly monitoring locations), determination of the total depth of the well will be performed initially and at a maximum interval of annually thereafter:

6. Lower the water level probe into the well. Record the depth of the air/water interface to the nearest hundredth of a foot.

7. Remove probe from the well.

8. Between wells, when obtaining water level measurements at more than one location, clean the instrument in accordance with the Field Equipment Decontamination SOP.

9. Close the well when all activities are completed.

10. Collect all PPE and other wastes generated for disposal.

VI. Waste Management

All investigation-derived waste (IDW) will be transferred to appropriate DOT-approved waste containers for management and treatment/disposal by others.

Waste containers must be a sealed and labeled at the time of generation. Labels will indicate date, sample locations, site name, city, state, and description of the matrix (e.g., media, PPE).

VII. Data Recording and Management

Field documentation entries will be documented in accordance with the Field Sampling Plan. The field team leader will retain all site documentation while in the field and add to the project files when the field mobilization is complete.

Attachment G –

Standard Operating Procedure:

Groundwater Purging and Sampling

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Standard Operating Procedure:

Groundwater Purging and Sampling

Hudson River Sediment Processing Facility

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 1

Rev Date: February 20, 2015

I. Introduction

Groundwater samples will be collected from monitoring wells to evaluate groundwater quality. The protocol presented in this standard operating procedure (SOP) describes the procedures to be used to purge monitoring wells and collect groundwater samples. Wells will not be sampled until well development has been performed, unless that well has been sampled or developed within the prior one-year time period. Groundwater samples will not be collected within a one-week time period following well development.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading sampling activities should have a minimum of 5 years of previous environmental sampling experience. ARCADIS personnel providing assistance to sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences.

III. Equipment / Materials List

Equipment and materials for groundwater sampling include:

- Site plan, well construction records, prior groundwater sampling records (if available)

- Sampling pump capable of maintaining a minimum pumping rate of 0.1 liter per minute (L/min), which may consist of one or more of the following:

  - Submersible pump (e.g., Grundfos Redi-Flo 2);
  - Peristaltic pump (e.g., Geopump); and/or
  - Bladder pump (e.g., Marschalk System 1).

- Polyethylene tubing of an appropriate size for the pump being utilized. For peristaltic pumps, dedicated silicone tubing (or other type as specified by the manufacturer) will also be utilized through the pump apparatus

- Water level probe (e.g., Solinst Model 101)

- Water quality meter (temperature, pH, specific conductivity, oxidation reduction potential [ORP], turbidity, dissolved oxygen [DO]) and flow-through measurement cell. Several brands may be utilized, including:

- YSI 6-Series Multi-Parameter Instrument;
- Hydrolab Series 3 or Series 4a Multiprobe and Display; and/or
- Horiba U-10, U-22, or U-52 Water Quality Monitoring System.

- Supplemental turbidity meter (e.g., Lamotte 2020 or Hach 2100P). Turbidity measurements collected with multi-parameter meters have been shown to sometimes be unreliable due to fouling of the optic lens of the turbidity meter within the flow-through cell. A supplemental turbidity meter will be utilized to verify turbidity data during purging if such fouling is suspected.

- Appropriate water sample containers (supplied by the laboratory)

- Appropriate blanks (trip blank supplied by the laboratory)

- Cleaning equipment, as described in the Field Equipment Decontamination SOP

- Health and safety equipment, as required in the site *Health and Safety Plan* (HASP).

The specific make/model of the equipment utilized during a sampling event will be recorded on a Groundwater Sampling Log.

IV. Cautions / Hazards

Task-specific Job Safety Analysis (JSAs) identifying site hazards and controls must be reviewed by all field crew members prior to the start of work. Safe Performance Self-Assessment (SPSA) to be performed by employees before performing a new task.

V. Health and Safety Considerations

Procedures will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task specific JSA forms, copies of which will be present on site during such activities.

VI. Procedures

Groundwater will be purged from the wells using an appropriate pump. Peristaltic pumps may be utilized if the depth to water is within the sampling range of a peristaltic pump (approximately 25 feet). Otherwise, submersible pumps or bladder pumps will be utilized provided the well is constructed with a casing diameter greater than or equal to 2 inches (the minimum well diameter capable of accommodating such

pumps). For smaller diameter wells where the depth to water is below the sampling range of a peristaltic pump, alternative sampling methods (i.e., bailing) will be utilized to purge and sample the groundwater. Purge water will be collected and containerized.

1. Perform calibration of field instruments.

2. Measure initial depth to groundwater prior to placement of pumps. If a submersible or bladder pump is being utilized, slowly lower pump, safety cable, tubing, and electrical lines into the well to a depth corresponding to the approximate center of the saturated screen section of the well. If a peristaltic pump is being utilized, slowly lower the sampling tubing into the well to a depth corresponding to the approximate center of the saturated screen section of the well. The pump intake or sampling tube must be kept at least 2 feet above the bottom of the well to prevent mobilization of any sediment present in the bottom of the well.

3. Measure the water level again with the pump in the well before starting the pump. Start pumping the well at 200 to 500 milliliters per minute (mL/min). The pump rate should be adjusted to cause little or no water level drawdown in the well (less than 0.3 feet below the initial static depth to water measurement, if possible) and the water level should stabilize. The water level should be monitored every 3 to 5 minutes (or as appropriate) during pumping if the well diameter is of sufficient size to allow such monitoring. Care should be taken not to break pump suction or cause entrainment of air in the sample. Record pumping rate adjustments and depths to water. If necessary, pumping rates should be reduced to the minimum capabilities of the pump to avoid pumping the well dry and/or to ensure stabilization of indicator parameters. A steady flow rate should be maintained to the extent practicable. Groundwater sampling records from previous sampling events (if available) should be examined to provide an estimate of the optimum pumping rate and anticipated drawdown for the well in order to more efficiently reach a stabilized pumping condition. If the recharge rate of the well is very low, the well should be pumped dry and sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection of samples.

4. During purging, monitor the field indicator parameters (e.g., turbidity, temperature, specific conductance, pH, dissolve oxygen, ORP) every 5 minutes (or as appropriate). Field indicator parameters will be measured using a flow-through analytical cell, although turbidity data may be measured by a separate turbidity meter located outside the flow-through cell if necessary. Record field indicator parameters on the Groundwater Sampling Log. The well is considered stabilized and ready for sample collection when turbidity values remain within 10% (or within 1 NTU if the turbidity reading is less than 10 NTU), the dissolved oxygen level remains within 10% (or within 0.1 milligrams per liter [mg/l] if the dissolved oxygen

level is less than 1.0 mg/l), the specific conductance and temperature values remain within 3%, ORP remains within 10 millivolts, and pH remains within 0.1 units for three consecutive readings collected at 5-minute intervals. If the field indicator parameters do not stabilize within one hour of the start of purging, but the groundwater turbidity is below the goal of 50 NTU and the values for all other parameters are within 10%, the well can be sampled. If the parameters have stabilized but the turbidity is not in the range of the 50 NTU goal, the pump flow rate should be decreased to a minimum rate of 100 mL/min to reduce turbidity levels as low as possible. During extreme weather conditions, stabilization of field indicator parameters may be difficult to obtain. Modifications to the sampling procedures to alleviate these conditions will be documented in the field notes. If other field conditions exist which preclude stabilization of certain parameters, an explanation of why the parameters did not stabilize will also be documented in the field notebook.

5. Complete the sample label and cover the label with clear packing tape to secure the label onto the container.

6. After the indicator parameters have stabilized, collect groundwater sample by diverting flow out of the unfiltered discharge tubing into the appropriate labeled sample container. If a flow-through analytical cell is being used to measure field parameters, the flow-through cell should be disconnected after stabilization of the field indicator parameters and prior to groundwater sample collection. Under no circumstances should analytical samples be collected from the discharge of the flow-through cell. When the container is full, tightly screw on the cap. Samples should be collected in the following order: volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), metals and polychlorinated biphenyls (PCBs), and others. If sampling for total and filtered metals and/or PCBs, a filtered and unfiltered sample will be collected. If the sample cannot be transferred to the laboratory for filtering, sample filtration for the filtered sample will be performed in the field utilizing an in-line filtration system. Install an in-line, disposable 0.45-micron particle filter on the discharge tubing after the appropriate unfiltered groundwater sample has been collected. Continue to run the pump until an initial volume of “flush” water has been run through the filter in accordance with the manufacturer’s directions (generally 100-300 mL). Collect filtered groundwater sample by diverting flow out of the filter into the appropriate labeled sample container. When the container is full, tightly screw on the cap.

7. Secure with packing material and store at 4°C in an insulated transport container provided by the laboratory.

8. Record on the Groundwater Sampling Log or bound field notebook the time sampling procedures were completed, any pertinent observations of the sample

(e.g., physical appearance; the presence of, or lack of, odors, sheens, etc.), and the values of the stabilized field indicator parameters, as measured during the final reading during purging.

9. Remove pump and tubing from well, secure well, properly dispose of personal protective equipment (PPE) and disposable equipment.

10. Complete the procedures for handling, packing, and shipping, with associated chain-of-custody.

11. Complete cleaning procedures for flow-through analytical cell and submersible pump, as appropriate (see Section VI - Equipment Cleaning).

12. At end of day, perform calibration check of field instruments.

If it is not technically feasible to utilize the low-flow sampling method, purging and sampling of monitoring wells may be conducted using the volume-based purging and sampling method as outlined below:

1. Don appropriate PPE, as required by the HASP.

2. Place plastic sheeting around the well.

3. Clean the sampling equipment with the procedures described in the Field Equipment Decontamination SOP.

4. Measure the depth to water and determine depth of well through examination of drilling log data or by direct measurement. Calculate the volume of water in the well (in gallons) by using the length of the water column (in feet), multiplying by 0.163 for a 2-inch well, or by 0.653 for a 4-inch well. For other well diameters, use the formula: *Volume (in gallons) = π TIMES well radius (in feet) squared TIMES length of water column (in feet) TIMES 7.481 (gallons per cubic foot)*

5. If a bailer is to be utilized, measure a length of rope at least 10 feet greater than the total depth of the well. Secure one end of the rope to the well casing, secure the other end of the rope to the bailer. Test the knots and make sure the rope will not loosen. Check bailers to be sure all parts are intact and will not be lost in the well.

6. Lower bailer, submersible pump, or peristaltic pump tubing (whichever is applicable) into well and remove one well volume of water. Contain all water in appropriate containers.

7. Monitor the field indicator parameters (e.g., turbidity, temperature, specific conductance, pH, etc.). Field indicator parameters will be measured using a clean container such as a glass beaker or sampling cups provided with the instrument. A flow-through analytical cell should be utilized if a pump is used for purging and sampling to enable collection of dissolved oxygen and ORP data; however, turbidity measurements may be conducted using a separate turbidity meter located outside the flow-through cell. Record field indicator parameters on the Groundwater Sampling Log.

8. Repeat Step 6 and Step 7 until three or four well volumes have been removed. Examine the field indicator parameter data to determine if the parameters have stabilized. The well is considered stabilized and ready for sample collection when turbidity values remain within 10% (or within 1 NTU if the turbidity reading is less than 10 NTU), the dissolved oxygen level remains within 10% (or within 0.1 mg/l if the dissolved oxygen level is less than 1.0 mg/l), the specific conductance and temperature values remain within 3%, ORP remains within 10 millivolts, and pH remains within 0.1 units for three consecutive readings collected once per well volume removed. Since accurate collection of dissolved oxygen and ORP data is not possible without the use of a flow-through cell (which cannot be used with bailed groundwater), the stabilization criteria for dissolved oxygen and ORP is only applicable to situations where a flow-through cell is utilized during well purging.

9. If the field indicator parameters have not stabilized, remove a maximum of five well volumes prior to sample collection.

10. If the recharge rate of the well is very low, wells may be bailed dry and sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection of samples. Field indicator parameter will be recorded again at the time of sample collection. For extremely low recharge wells, where sampling attempts over several days are necessary to collect the required sample volume, field indicator parameters will be recorded each day that samples are collected, provided the well contains sufficient volume to conduct those measurements.

11. Slowly lower the bailer into the screened portion of the well and carefully retrieve a filled bailer from the well causing minimal disturbance to the water and any sediments in the well.

12. The sample collection order (as appropriate) will be as follows: VOCs; SVOCs; Metals and PCBs; and others.

13. Samples will be collected directly into labeled laboratory-provided sample containers.

14. Secure with packing material and store at 4°C in an insulated transport container provided by the laboratory.

15. Remove bailer from well, secure well, and properly dispose of PPE and disposable equipment.

16. If a bailer is to be dedicated to a well, it should be secured inside the well above the water table, if possible. Dedicated bailers should be tied to the well cap so that inadvertent loss of the bailer will not occur when the well is opened.

17. Complete the procedures for handling, packing, and shipping with associated chain-of-custody.

VII. Equipment Cleaning

All groundwater sampling equipment should be cleaned prior to use in the first well and after each subsequent well using procedures described in the Field Equipment Decontamination SOP.

VIII. Waste Management

All investigation-derived waste (IDW) generated by the sampling activities (e.g., purge water sampling supplies, decontamination rinsate, PPE, etc.) will be transferred to appropriate DOT-approved waste containers for management and treatment/disposal by others.

Waste containers must be a sealed and labeled at the time of generation. Labels will indicate date, sample locations, site name, city, state, and description of the matrix (e.g., media, PPE).

IX. Data Recording and Management

Field documentation entries and chain-of-custody records will be documented in accordance with the Field Sampling Plan. Chain-of-custody forms will be electronically transmitted each day. The field team leader will retain all site documentation while in the field and add to the project files when the field mobilization is complete.

X. Quality Assurance

Quality assurance samples (duplicates and MS/MSDs) will be collected at the frequency specified in the Field Sampling Plan (FSP). Reusable sampling equipment will be cleaned prior to use following field equipment decontamination SOP. Any deviations from the SOP will be discussed with the project manager prior to changing any field procedures.

Attachment H –

Standard Operating Procedure:

Sediment Sampling

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Standard Operating Procedure:
Sediment Sampling

Hudson River Sediment Processing Facility
Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 1

Rev Date: February 20, 2015

I. Introduction

General procedures utilized to obtain sediment samples from waterbodies are outlined below. Lexan® tubing will be the primary method used to collect sediment cores. The core will be inserted with a straight, vertical entry into the sediments so as to secure a reliably representative cross-section sample.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading sampling activities should have a minimum of 5 years of previous environmental sampling experience. ARCADIS personnel providing assistance to sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences.

III. Equipment / Materials List

The following equipment/materials will be available, as required, during sediment sampling activities.

- health and safety equipment, as required by the HASP
- cleaning equipment, as described in the Field Equipment Decontamination SOP
- boat
- chip or hip waders
- stainless steel tray
- duct tape
- Lexan® tubing with end caps
- 6-foot rule or survey rod
- transport container with ice or “blue” ice;
- appropriate sample containers and forms

- field notebook

IV. Cautions / Hazards

Task-specific Job Safety Analysis (JSAs) identifying site hazards and controls associated with the sampling activities must be reviewed by all field crew members prior to the start of work. Safe Performance Self-Assessment (SPSA) to be performed by employees before performing a new task.

V. Health and Safety Considerations

Sample collection will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task specific JSA forms, copies of which will be present on site during such activities.

VI. Sediment Sampling Procedures

1. Identify the proposed sample location on a sampling log sheet and/or field notebook, along with other appropriate information collected during sediment sampling activities.

2. Don PPE, as required by the HASP.

3. At each sample location, lower a section of Lexan® tube until it just reaches the top of sediment. Measure the depth of water.

4. Push the Lexan® tube into the sediment by hand until refusal. Measure the depth of sediment. If procedure is being performed to determine sediment depth, a calibrated rod may be used in place of the Lexan® tube. If procedure is being performed to collect samples for laboratory analysis, continue with Step 5.

5. Drive the tube several more inches using a stainless steel core driver block and measure the distance. This procedure is performed to obtain a “plug” at the bottom of the core and prevent the loose sediment from escaping. Place an end cap on top end of the tube.

6. Slowly pull the tube from the sediment, twisting it slightly as it is removed (if necessary).

7. Before the tube is fully removed from the water, place a cap on the bottom end of the tube while it is still submerged.

8. Keeping the tube upright, wipe the bottom end dry, seal the caps with duct tape if needed, and label. Measure the length of sediment recovered and evaluate the

integrity of the core. If the core is not suitably intact, repeat coring procedure within 5 to 10 feet of the first location attempted.

9. Transport the core sample to the shore.

10. Decant water from tube, and extrude sediment core from the Lexan® tubing onto a stainless steel tray. Describe and record sample description.

11. If sampling for VOCs, the core section will be placed immediately into the sample jar (without compositing) following extrusion from the Lexan® tubing.

12. Label all sample containers with: 1) site, 2) project number, 3) location number, 4) sample interval, 5) date, 6) time of core collection, and 7) names of sampling personnel.

13. Record all appropriate information in the field notebook and sampling log form(s).

14. Survey the sampling locations using standard instrument survey techniques.

VII. Waste Management

All investigation-derived waste (IDW) generated by the sampling activities (e.g., excess soil/sediment, sampling supplies, decontamination rinsate, PPE, etc.) will be transferred to appropriate DOT-approved waste containers for management and treatment/disposal by others. Waste containers must be a sealed and labeled at the time of generation. Labels will indicate date, sample locations, site name, city, state, and description of the matrix (e.g., media, PPE).

VIII. Data Recording and Management

Field documentation entries and chain-of-custody records will be documented in accordance with the Field Sampling Plan. Chain-of-custody forms will be electronically transmitted each day. The field team leader will retain all site documentation while in the field and add to the project files when the field mobilization is complete.

IX. Quality Assurance

Quality assurance samples (duplicates and MS/MSDs) will be collected at the frequency specified in the Field Sampling Plan (FSP). Reusable sampling equipment will be cleaned prior to use following field equipment decontamination SOP. Field equipment blanks will be used to confirm that decontamination procedures are sufficient and samples are representative of site conditions. Any deviations from the SOP will be discussed with the project manager prior to changing any field procedures.

Field duplicates will be prepared by compositing sediment collected from directly adjacent (within 6 inches) locations at the same time and depth and then transferring this material into two sets of sample jars. For VOCs, the samples will not be composited prior to placement in the sample jars. Because of this, the VOC samples will not be truly duplicate samples. The samples will be labeled in such a way that the designations will not indicate the duplicate nature of the samples.

Attachment I –

Standard Operating Procedure:

Surface Water Sampling

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Standard Operating Procedure: Surface Water Sampling

Hudson River Sediment Processing Facility

Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 1

Rev Date: February 20, 2015

I. Introduction

This appendix specifies procedures for collecting surface water samples for subsequent chemical analysis. Surface water sampling will not take place during precipitation events (unless so specified in the project-specific work plan), and samples will be obtained beginning with the most downstream location and proceeding upstream.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading sampling activities should have a minimum of 5 years of previous environmental sampling experience. ARCADIS personnel providing assistance to sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences.

III. Equipment / Materials Lists

The following equipment/materials will be available, as required, during surface water sampling.

- health and safety equipment, as required by the site *Health and Safety Plan* (HASP)
- cleaning equipment, as described in the Field Equipment Decontamination SOP
- boat, as needed
- chest or hip waders
- rope
- surveyor's rod and/or 6-foot rule
- duct tape
- peristaltic pump
- medical-grade silicone tubing

- polyethylene tubing
- field notebook
- multi-parameter water quality meter
- appropriate blanks (trip), if necessary
- appropriate sampling containers and forms
- appropriate preservatives (as required)
- coolers with ice or “blue” ice
- appropriate water sampler as field conditions dictate, which may include the following:
  - surface water grab sampler consisting of a 1,000-milliliter (mL) beaker, adjustable clamp, and two-or three-piece telescoping aluminum tube or an equivalent acceptable sampling device; or
  - peristaltic pump with a short piece of medical-grade silicone tubing and polyethylene tubing; or
  - kemmerer stainless steel bottle sampler.

IV. Cautions / Hazards

Task-specific Job Safety Analysis (JSAs) identifying site hazards and controls associated with the sampling activities must be reviewed by all field crew members prior to the start of work. Safe Performance Self-Assessment (SPSA) to be performed by employees before performing a new task.

Caution will be used not to disturb the sediment within the sampling area at the time of water sample collection. Approach the sampling from the shore or by boat first with the last option to enter the stream or creek starting downstream and walking upstream towards the location.

V. Health and Safety Considerations

Sample collection will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task-specific JSA forms, copies of which will be present on site during such activities.

VI. Sampling Procedures

The following procedures will be used to obtain surface water grab samples:

1. Identify surface water sampling location on appropriate sampling log sheet and/or field notebook, along with other appropriate information.

2. Don health and safety equipment, as required by the HASP.

3. Clean the sampling equipment in accordance with the procedures described in the Field Equipment Decontamination SOP.

4. Assemble the water grab sampler. Make sure that the sampling beaker and the nuts and bolts that secure the clamp to the pole are tightened properly.

5. Obtain sample by slowly submerging the beaker with minimal surface disturbance (if sampling a stream, the beaker opening will be upstream) to a depth that is 0.5 times the total water depth, unless otherwise specified in the project-specific work plan.

6. Retrieve the water sampler from the surface water with minimal disturbance.

7. Empty the sample slowly into laboratory-provided sample containers.

8. The sample collection order (as appropriate) will be as follows: volatile organic compounds (VOCs); Semi-volatile organic compounds (SVOCs); metals; and polychlorinated biphenyls (PCBs); and others.

9. If sampling for total and filtered metals, a filtered and unfiltered sample will be collected. Sample filtration for the filtered sample will be performed in the field utilizing a peristaltic pump. Install new medical-grade silicone tubing in the pump head. Place new polyethylene tubing into the sample collection container and attach to the intake side of pump tubing. Attach (clamp) a new 0.45-micron filter to the discharge side of the pump tubing (noting the correct filter flow direction). Turn the pump on and dispense the filtered liquid directly into the laboratory sample bottles.

10. If sampling for total and filtered PCBs, two samples must be collected, one of which will be filtered by the laboratory prior to analysis.

11. Secure the sample jar cap(s) tightly.

12. Label all sample containers as appropriate.

13. After sample containers have been filled, measure the pH, turbidity, temperature, and conductivity at the surface water location.

14. Record required information on the appropriate forms and/or field notebook.

15. Complete chain-of-custody forms, prepare shipping containers, and send to the analytical laboratory for analysis.

16. Survey the sampling locations using standard instrument survey techniques.

VII. Waste Management

Investigation-derived waste (IDW) generated by the sampling activities (e.g., sampling supplies, decontamination rinsate, PPE, etc.) will be transferred to appropriate DOT-approved waste containers for management and treatment/disposal by others. Waste containers must be a sealed and labeled at the time of generation. Labels will indicate date, sample locations, site name, city, state, and description of the matrix (e.g., media, PPE).

VIII. Data Recording and Management

Field documentation entries and chain-of-custody records will be documented in accordance with the Field Sampling Plan. Chain-of-custody forms will be electronically transmitted each day. The field team leader will retain all site documentation while in the field and add to the project files when the field mobilization is complete.

IX. Quality Assurance

Quality assurance samples (duplicates and MS/MSDs) will be collected at the frequency specified in the Field Sampling Plan (FSP). Reusable sampling equipment will be cleaned prior to use following field equipment decontamination SOP. Field equipment blanks will be used to confirm that decontamination procedures are sufficient and samples are representative of site conditions. Any deviations from the SOP will be discussed with the project manager prior to changing any field procedures.

Collection of duplicates involves the collection of two independent samples. The sample collection procedures are repeated at the same location and sample depth to the extent possible. The sample device (e.g., Kemmerer bottle) is sent down to a specific depth, retrieves the sample, is brought to the surface, and the sample is transferred to the duplicate sample container. The duplicate sample will be labeled in such a way that the sample descriptions will not indicate the duplicate nature of the samples.

Attachment J –

Standard Operating Procedure:

Field Parameter Measurement

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Standard Operating Procedure:
Field Parameter Measurement

Hudson River Sediment Processing Facility
Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 1

Rev Date: February 20, 2015

I. Introduction

Hydrochemical parameters such as specific conductivity, pH, temperature, turbidity, oxidation/reduction potential (ORP), and dissolved oxygen (DO) of groundwater or surface water are measured in the field. The pH and conductivity of the ground/surface water will be recorded using a portable meter with temperature compensating pH and conductivity electrodes. The meters will be calibrated twice-daily (at a minimum) in the field. Calibration will occur before use and at the end of the day, and according to the manufacturer's instructions and the procedures specified herein (which include EPA analytical methods). Additional calibration may be performed if conditions and/or manufacturer's specifications dictate. All calibration data should be recorded and filed with the project field records.

II. Equipment / Materials List

The following equipment/materials (or equivalent) shall be available, as required, during measurement of hydrochemical parameters:

- Two water quality (temperature/pH/specific conductivity/ORP/turbidity/DO) meters (one for back up) and flow-through measurement cells. Several brands may be utilized, including:
  - YSI 6-Series Multi-Parameter Instrument;
  - Hydrolab Series 3 or Series 4a Multiprobe and Display; and/or
  - Horiba U-22/U-52/U-53 Water Quality Monitoring System.
- Supplemental turbidity meter (Lamotte 2020) if necessary
- Appropriate calibration standards
- Field sample container
- Cleaning equipment, as described in the Field Equipment Decontamination SOP
- Fine screwdriver
- Extra batteries
- Appropriate log forms

- Field notebook

III. Procedures

Calibration

The detailed procedure for the calibration of field instruments used to measure water quality is outlined below. This guidance document was prepared primarily to address calibration of multi-probe water quality monitoring instruments, but is also applicable to the calibration of most single-parameter monitoring instruments.

- *Temperature*: Perform annual accuracy check according to procedures outlined in the equipment manual, as needed.

- *pH*: Perform calibration according to procedures outlined in the equipment manual. Note that if pH values observed during field activities are outside the initial calibration range, re-calibration will be required.

- *Dissolved Oxygen*: Perform a saturated air calibration according to procedures outlined in the equipment manual. The DO probe membrane and electrolyte solution should be replaced prior to the sampling period if the instrument has been inactive for an extended time period or as an initial response if erratic measurements are observed.

- *Conductivity/Specific Conductivity*: Perform calibration according to procedures outlined in the equipment manual.

- *Oxidation/Reduction Potential*: Perform calibration according to procedures outlined in the equipment manual. If possible, plot values of millivolt versus temperature for the calibration standard on graph paper to aid in interpolation of temperature-corrected millivolt values. These values are usually found on the label of the calibration standard and may vary between solutions. Therefore, the values should be checked for each bottle of calibration solution utilized and new interpolation graphs should be prepared if necessary.

- *Turbidity*: Perform calibration according to procedures outlined in the equipment manual. If erratic readings are observed, clean detector according to manufacturer’s instructions as an initial response.

Field Measurement

The detailed procedure for obtaining the temperature, turbidity, specific conductivity, pH, ORP, and DO of a single water sample utilizing a multi-probe water quality monitoring instrument and flow-through cell is presented in Groundwater Purging and Sampling Procedures for Monitoring Wells SOP. The detailed procedure for obtaining the temperature, turbidity, conductivity, pH, and DO of a single water sample is outlined below.

1. Obtain a small quantity of the water sample, place it in field sample container, agitate, then discard. Refill the container. Rinse the pH, specific conductivity, DO, turbidity, and temperature probes with distilled water. Submerse probes into the container containing the water. Allow approximately one minute for readings to stabilize, then record the measurements on the appropriate forms.

2. Clean the probe and cable with a non-phosphate soap and water wash followed by a distilled/deionized water rinse. Store in a clean container.

IV. Data Recording and Management

Field documentation entries will be documented in accordance with the Field Sampling Plan. The field team leader will retain all site documentation while in the field and add to the project files when the field mobilization is complete.

Attachment K –

Standard Operating Procedure:

Photoionization Detector Field Screening

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Standard Operating Procedure:
Photoionization Detector Field Screening

Hudson River Sediment Processing Facility
Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 1

Rev Date: February 20, 2015

I. Introduction

Field screening with a photoionization detector (PID), such as an HNu™, Photovac™, MicroTIP™, or MiniRAE™, is a procedure to measure relative concentrations of volatile organic compounds (VOCs) and other compounds. Field screening may be conducted in the headspace of soil samples to assess the relative concentration of volatile organics in the sample.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading sampling activities should have a minimum of 5 years of previous environmental sampling experience. ARCADIS personnel providing assistance to sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences.

III. Equipment / Materials List

The following materials, as required, shall be available while performing PID field screening:

- personal protective equipment (PPE), as required by the site Health and Safety Plan (HASP)
- PID and operating manual
- PID extra battery pack and battery charger
- calibration canisters for the PID
- Ziploc-type bags
- field notebook

IV. Cautions

PIDs are sensitive to moisture and may not function under high humidity. PIDs cannot be used to indicate oxygen deficiency or combustible gases.

V. Health and Safety Considerations

Sample collection will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task specific JSA forms, copies of which will be present on site during such activities.

VI. Procedures

<u>PID Calibration</u>

PID field instruments will be calibrated and operated to yield “total organic vapor” in parts per million (ppm) (v/v) relative to benzene or isobutylene (or equivalent). Operation, maintenance, and calibration shall be performed in accordance with the manufacturer’s instructions.

1. Don PPE, as required by the HASP.

2. Perform a BATTERY CHECK. If battery is low, the battery must be charged before calibration.

3. Listen for the fan operation to verify fan function.

4. Calibrate the PID using a two point calibration. First calibrate to zero using ambient air.

5. Next, calibrate to a known concentration using calibration gas (isobutylene) provided with the PID. Perform the SPAN calibration.

6. If so equipped, set the alarm at desired level.

<u>Field Screening Procedures</u>

Soil samples will be field screened upon collection with the PID for a relative measure of the total volatile organic concentration. The following steps define the PID field screening procedures.

1. Half-fill a sealable freezer bag with the sample to be analyzed. Quickly seal the top of the bag.

2. Allow headspace development for at least 10 minutes. Vigorously shake bag for 15 seconds at both the beginning and end of the headspace development period.

Where ambient temperatures are below 32°F (0°C), headspace development should be within a heated building.

3. Carefully open a corner of the bag and insert the sampling probe to a point about one-half of the headspace depth. Exercise care to avoid contact with water droplets or soil particulates.

4. Following probe insertion, record the highest meter response for each sample as the headspace concentration. Erratic meter response may occur at high organic vapor concentrations or conditions of elevated headspace moisture; in which case, headspace data should be recorded and erratic meter response noted.

5. PID field instruments will be operated and calibrated to yield "total organic vapors" in ppm (v/v). PID instruments must be operated with at least a 10.0 eV(+) lamp source. Operation, maintenance, and calibration will be performed in accordance with the manufacturer's specifications.

6. Instrumentation with digital (LED/LCD) displays may not be able to discern maximum headspace response unless equipped with a "maximum hold" feature or strip-chart recorder.

VII. Waste Management

Investigation-derived waste (IDW) generated by the sampling activities (e.g., core cuttings, sampling supplies, PPE, etc.) will be transferred to appropriate DOT-approved waste containers for management and treatment/disposal by others. Waste containers must be a sealed and labeled at the time of generation. Labels will indicate date, sample locations, site name, city, state, and description of the matrix (e.g., media, PPE).

Do not dispose of canisters of compressed gas if there is still compressed gas in the canister. While standing outdoors and upwind of the canister, discharge gas in canister by opening the valve until the pressure in the gauge is zero. DO NOT PUNCTURE CANISTER. When empty, mark "EMPTY" on canister and discard the canister in trash.

VIII. Data Recording and Management

Measurements will be recorded in the field notebook or boring logs at the time of measurement with notation of date, time, location, depth, and item monitored. If a data memory is available, readings will be downloaded from the unit upon access to a computer with software to retrieve the data.

IX. Quality Assurance

After each use, the readout unit should be wiped down with a clean cloth or paper towel.

For a HNu, the UV light source window and ionization chamber should be cleaned once a month in the following manner:

1. With the PID off, disconnect the sensor/probe from the unit.

2. Remove the exhaust screw, grasp the end cap in one hand and the probe shell in the other, and pull apart.

3. Loosen the screws on top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing.

4. Tilt the lamp housing with one hand over the opening so that the lamp slides out into your hand.

5. Clean the lamp with lens paper and HNu cleaning compound (except 11.7 eV). For the 11.7 eV lamp, use a chlorinated organic solvent.

6. Clean the ion chamber using methanol on a Q-tip and then dry gently at 50ºC to 60ºC for 30 minutes.

7. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Place the ion chamber on top of the housing, making sure the contacts are properly aligned.

8. Place the end cap on top of the ion chamber and replace the two screws (tighten the screws only enough to seal the o-ring).

9. Line up the pins on the base of the lamp housing with pins inside the probe shell and slide the housing assembly into the shell.

Attachment L –

Standard Operating Procedure:

Field Equipment Decontamination

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Standard Operating Procedure:
Field Equipment Decontamination

Hudson River Sediment Processing Facility
Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Rev. #: 2

Rev Date: February 20, 2015

I. Introduction

Equipment decontamination is performed to ensure that sampling equipment that contacts a sample is free from analytes of interest and/or constituents that would interfere with laboratory analysis for analytes of interest. Equipment must be cleaned prior to use for sampling or contact with environmental media to be sampled, and prior to shipment or storage.

Equipment that may require decontamination includes: sampling tools/devices; testing instruments; and other sampling equipment. Non-disposable equipment will be cleaned before collecting each sample, between sampling events, and prior to leaving the site. The effectiveness of the decontamination procedure will be monitored by collecting equipment blank samples as specified in the Field Sampling Plan (FSP). Dedicated and/or disposable (not to be re-used) sampling equipment will not require decontamination.

II. Personnel Qualifications

ARCADIS field sampling personnel will have current health and safety training, including 40-hour HAZWOPER training, site supervisor training, and site-specific training. In addition, ARCADIS field sampling personnel will be versed in the relevant Standard Operating Procedures (SOP) and possess the skills and experience necessary to successfully complete the project field work. The project Health and Safety Plan (HASP) and other documents will identify any other training requirements such as site-specific safety training or access control requirements.

III. Equipment / Materials List

- health and safety equipment, as required in the site HASP
- non-phosphate detergent, such as Alconox (or equivalent)
- tap water
- hexane
- distilled/deionized water
- rinseate collection plastic containers

- DOT-approved waste container(s) (if decontamination waste is to be shipped for disposal)
- scrub brushes
- large heavy-duty garbage bags
- spray bottles
- hexane
- Ziploc-type bags
- plastic sheeting
- lint-free towels

IV. Cautions / Hazards

Task-specific Job Safety Analysis (JSAs) identifying site hazards and controls associated with the decontamination activities must be reviewed by all field crew members prior to the start of work. Safe Performance Self-Assessment (SPSA) to be performed by employees before performing a new task

Rinse equipment thoroughly and allow the equipment to dry before re-use or storage to prevent introducing solvent into sample medium. If manual drying of equipment is required, use clean lint-free material to wipe the equipment dry.

Store decontaminated equipment in a clean, dry environment. Do not store near combustion engine exhausts.

If equipment is damaged to the extent that decontamination is uncertain due to cracks or dents, the equipment should not be used and should be discarded or submitted for repair prior to use for sample collection.

V. Health and Safety Considerations

Sample collection will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task specific JSA forms, copies of which will be present on site during such activities.

Review the material safety data sheets (MSDSs) for the cleaning materials used in decontamination. If solvent is used during decontamination, work in a well-ventilated area and stand upwind while applying solvent to equipment. Apply solvent in a manner that minimizes potential for exposure to workers. Follow health and safety procedures outlined in the HASP.

VI. Procedure

As practicable, a designated area will be established to clean sampling equipment in the field prior to and following sample collection. Equipment cleaning areas will be set up within or adjacent to the specific work area, but not at a location exposed to combustion engine exhaust. Detergent solutions will be prepared in clean containers for use in equipment decontamination.

Cleaning Sampling Equipment

1. Wash the equipment with potable water.

2. Wash the equipment with a detergent solution and scrub brush (Alconox or equivalent) to remove all visible solids.

3. Rinse the equipment with potable water.

4. Rinse the equipment with hexane.

5. Rinse the equipment with distilled/deionized water.

6. Allow the equipment to dry before re-use or storage. If manual drying of equipment is required, use clean lint-free material to wipe the equipment dry.

7. Wrap dry equipment in aluminum foil or other appropriate material until the equipment is used.

VII. Waste Management

All investigation-derived waste (IDW) generated by the sampling activities (e.g., core cuttings, sampling supplies, decontamination rinsate, personal protective equipment [PPE], etc.) will be transferred to appropriate DOT-approved waste containers for management and treatment/disposal by others.

Waste containers must be a sealed and labeled at the time of generation. Labels will indicate date, sample locations, site name, city, state, and description of the matrix (e.g., media, PPE).

VIII. Data Recording and Management

Field equipment cleaning and decontamination procedures will be documented in accordance with the FSP. The field team leader will retain all site documentation while in the field and add to project files when the field mobilization is complete.

Any unusual field conditions should be noted if there is potential to impact the efficiency of the decontamination or subsequent sample collection.

All containers with decontamination fluids will be properly labeled prior to use.

IX. Quality Assurance

Equipment blanks will be collected at the frequency specified in the FSP and depending on the project quality objectives. Equipment blanks will verify that the decontamination procedures are effective in minimizing potential for cross contamination. Equipment blanks will be analyzed for the same set of parameters that are performed on the field samples collected with the equipment that was cleaned. Equipment blanks are collected per equipment set, which represents all of the tools needed to collect a specific sample.

Attachment M –

Sample ID Nomenclature

Sample ID Nomenclature

Hudson River Sediment Processing Facility
Phase 2 Sediment Processing Facility Demobilization and Restoration Field Sampling Plan

Location ID =

DCM-[LocType]-[yyyymmdd]-[nnn]

Where,

DCM = Field program code (Sediment Processing Facility Decommissioning)

LocType = Abbreviations for the different areas of the Sediment Processing Facility including,

CMS = Coarse Material Staging Area (exclusion zone)
CSE = Coarse Material Staging Area - Expansion Area (exclusion zone)
DCA = Decontamination Area (exclusion zone)
DWA = Dewatering Area (exclusion zone)
FBN = Filter Cake Building-North (exclusion zone)
FBS = Filter Cake Building-South (exclusion zone)
FPB = Filter Press Building (exclusion zone)
NSB = North Stormwater Basin (exclusion zone)
REZ = Site Roads-Exclusion Zone (exclusion zone)
RLP = Rail Loading Platform (Track 7) (exclusion zone)
SSA = Size Separation Area (exclusion zone)
SSB = South Stormwater Basin (exclusion zone)
ULW = Unloading Wharf (exclusion zone)
WSB = Waterfront Stormwater Basin (exclusion zone)
WTP = Water Treatment Plant Building (exclusion zone)
OTE = Other (exclusion zone)
BND = Bond Creek (outside exclusion zone)
CHC = Champlain Canal (outside exclusion zone)
LDC = Lock Diversion Canal (outside exclusion zone)
SPT = Support Zone (outside exclusion zone)
TRB = Unnamed Tributary
OTS = (outside exclusion zone)

nnn = sample location numeric incrementer including
001 = sample location #1
002 = sample location #2
003 = sample location #3
etc.

Sample ID =

[loc id]-[matrix]-[character]

Where,

loc id = The Location ID defined above

matrix = Sample matrix code including,

ASP = Asphalt
SNC = Sand (Construction Material)
CRT = Concrete
DEB = Debris
GRV = Gravel/Crushed Stone
GW = Groundwater
NPW = Non-Painted Surface (Wipe Sample)
PSW = Painted Surface (Wipe Sample)
SED = Sediment
SO = Soil
SW = Surface Water
PC = Paint Chip
OTH = Other

character = character incrementer including,
A = first sample collected as a given location
B = second sample collected at a given location
C = third sample collected at a given location
D = fourth sample collected at a given location; etc.
DDD = duplicate sample
EB = Equipment Blank
MS/MSD = matrix spike/matrix spike duplicate

* The sampling ID nomenclature for equipment items will also include the distinct tracking IDs to be generated for each item.

Attachment N –

Monitoring Well Construction and Gauging Summary

General Electric Company
New York State Canal Corporation Characterization Baseline Data Summary Report

Table 2-2 - Monitoring Well Construction and Gauging Summary

Well ID	Northing	Easting	Ground Surface Elevation	Top of Casing Elevation	Total Depth (feet bgs)	Screened Interval (feet bgs)	Depth to Water (bgs)	Depth to Water (Below TOC)	Water Table Elevation
MW-01	1621538.3	741774.0	137.40	140.30	20.95	7.86 to 17.86	10.38	13.28	127.02
MW-01D	1621546.1	741774.0	137.60	140.33	50.70	36.90 to 46.90	3.58	6.31	134.02
MW-02	1621542.8	740677.5	132.60	135.43	17.80	3.62 to 13.62	5.53	8.36	127.07
MW-02D	1621544.9	740684.3	132.60	135.17	74.10	60.95 to 71.65	5.52	8.09	127.08
MW-03	1620087.8	740945.6	130.80	133.10	16.46	3.57 to 13.57	6.20	8.50	124.60
MW-03D	1620071.3	740957.3	130.60	133.45	39.14	25.67 to 35.67	5.65	8.50	124.95
MW-04	1619325.6	739865.3	131.20	133.88	16.85	3.78 to 13.78	5.50	8.18	125.70
MW-04D	1619328.4	739857.4	131.20	134.26	75.30	61.71 to 71.71	32.46	35.52	98.74
MW-05	1619950.5	739155.3	134.70	137.66	17.30	4.50 to 14.50	8.19	11.15	126.51
MW-05D	1619956.3	739158.6	134.80	137.77	77.65	64.19 to 74.19	36.11	39.08	98.69
MW-06	1620696.7	739766.7	133.60	136.39	19.53	6.83 to 16.83	7.01	9.80	126.59
MW-06D	1620700.9	739770.6	133.70	136.27	76.30	64.09 to 74.09	50.23	52.80	83.47
MW-07	1620331.9	739456.2	134.00	137.02	16.85	3.46 to 13.46	7.47	10.49	126.53
MW-08	1619358.6	738634.5	137.90	140.61	21.02	7.73 to 17.73	11.54	14.25	126.36
MW-09	1619104.4	738770.1	134.10	137.14	18.50	5.24 to 15.24	8.05	11.09	126.05
MW-10	1619158.9	739353.6	130.40	133.09	18.38	5.40 to 15.40	4.71	7.40	125.69
MW-11	1618999.1	740261.3	132.20	132.20	14.51	4.51 to 14.51	6.32	6.32	125.88
MW-12	1619799.0	740302.5	130.20	132.74	17.28	4.45 to 14.45	4.29	6.83	125.91
MW-13	1620280.5	740556.0	132.80	135.68	16.90	3.18 to 13.18	6.62	9.50	126.18
MW-14	1620623.9	741368.3	133.15	133.15	14.15	4.15 to 14.15	5.45	5.45	127.70
MW-15	1620824.3	740961.8	129.90	132.79	17.70	3.89 to 13.89	3.64	6.53	126.26
MW-16	1621167.8	741828.9	131.56	131.56	14.57	4.57 to 14.57	4.15	4.15	127.41
MW-17	1621653.9	741439.0	132.20	135.06	22.97	4.48 to 19.48	4.95	7.81	127.25
MW-18	1621213.7	740069.4	140.00	142.70	27.44	15.00 to 25.00	13.07	15.77	126.93

Notes:
1. The coordinates obtained based upon the North American Datum of 1983 New York State Plane East Zone.
2. Elevations are provided in feet above mean sea level relative to the North American Vertical Datum of 1988.
3. TOC - Top of Casing.
4. bgs - Below Ground Surface.

Attachment O –
Analytical Approach and Procedures –
Facility Decontamination-Related Samples

ATTACHMENT O

ANALYTICAL PROGRAM
APPROACH AND PROCEDURES
FACILITY DECONTAMINATION-RELATED SAMPLES

Prepared for

General Electric
Albany, New York

Prepared by
Environmental Standards, Inc.
Valley Forge, Pennsylvania

Revised September 2015

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Appendix

Appendix O2-1 SOP for the Soxhlet Extraction of Wipe Samples for PCB Analysis (S-NY-O-088-rev.10)

LIST OF ACRONYMS AND ABBREVIATIONS

%R	Percent Recovery
COC	Chain-of-Custody
DQI	Data Quality Indicators
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
EPA	United States Environmental Protection Agency
FSP	Field Sampling Plan
GC/ECD	Gas Chromatograph/Electron Capture Detector
GE	General Electric Company
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MDL	Method Detection Limit
mg/kg	milligrams per kilogram
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NELAP	National Environmental Accreditation Program
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
PASRCC	Precision, Accuracy/Bias, Sensitivity, Representativeness, Comparability and Completeness
PCB	Polychlorinated Biphenyl
PE	Performance Evaluation
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QC	Quality Control
RAM QAPP	Remedial Action Monitoring Quality Assurance Project Plan
RL	Reporting Limit
RPD	Relative Percent Difference
SOP	Standard Operating Procedure
SPF	Sediment Processing Facility
TAT	Turn-around Time
VTSR	Verified Time of Sample Receipt

1 INTRODUCTION

1.1 Analytical Program Summary

Table O1-1 lists the analyses that will be performed by the laboratory for the Facility decontamination-related samples collected as part of the Sediment Processing Facility (SPF) Demobilization. The responsibilities and duties of the analytical laboratory will be consistent with those described in the Phase 2 Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP, Anchor QEA and ESI 2012) including the October 2014 revision to the Analytical Approach and Procedures (Revised Attachment A, ESI 2014) and subsequent updates. The analyte lists and method detection limits (MDLs) and reporting limits (RLs) for each analyte and method are listed in Table O1-2. Quality assurance/quality control (QA/QC) samples originating in the field are described in Section 5 of the SPF Demobilization and Restoration Field Sampling Plan (FSP). Laboratory QA/QC samples are described in the standard operating procedures (SOPs) provided in the Phase 2 RAM QAPP or appended to this attachment as noted below.

The turnaround times (TATs) for the Facility decontamination-related samples will begin at the verified time of sample receipt [VTSR] at the laboratory and will be specified on the Chain-of Custody record. The samples will be submitted for expedited TATs (anywhere from 24 hours to 7 days for the electronic data deliverable [EDD]) on an as needed basis or standard TAT (10 business days for the EDD). Hard copy data packages will be submitted within 20 business days. Laboratory analytical data for this project will be reported in both an EDD and an analytical data package. Analytical data packages will be prepared by the laboratories according to the procedures described in the SOP “Data Package Deliverable,” which is included in Appendix A1-1 of Revised Attachment A of the Phase 2 RAM QAPP. Full data packages (Level B) will be provided for all sample analyses. Data packages will be provided by the laboratory in an Adobe® Acrobat® .pdf electronic format.

1.2 Special Training/Certification

The laboratory will have sufficient personnel with the necessary education, training, technical knowledge, and experience for their assigned functions as described in Revised Attachment A. The laboratory (NELAP) participating in this project will be accredited through New York State’s Environmental Laboratory Accreditation Program (ELAP) and the National Environmental Laboratory Accreditation Program (NELAP) for the analyses being performed. Table O1-1 lists the certification status or if certification is not available for that analyte.

2 FACILITY DECONTAMINATION-RELATED SAMPLES ANALYTICAL PROCEDURES

The SPF Demobilization will involve the analysis of samples collected from equipment, structures, and construction materials in connection with Facility decontamination efforts. It is anticipated that the Facility decontamination-related samples will include:

- wipe samples from painted and non-painted surfaces;
- pulverized core samples from asphalt and concrete materials; and

- bulk material samples from construction materials (*e.g.*, sand and sub-base gravel).

PCB and moisture content analyses of Facility decontamination-related samples will be performed in accordance with Method GEHR8082 (Appendix A4-1, Revised Attachment A), and the associated extraction SOPs (Appendices A4-3 through A4-5, Revised Attachment A), with the following exceptions:

- Performance Evaluation samples will not be prepared and analyzed as discussed in Section 11.2.1 of the Phase 2 RAM QAPP. Instead, matrix spike/matrix spike duplicate (MS/MSD) samples spiked with only Aroclor 1242 will be prepared and analyzed at a frequency of five percent (*i.e.*, one for every 20 samples) of each matrix, with the exception of wipe samples.
- Laboratory control sample (LCS) will consist of only Aroclor 1242 instead of a combination of Aroclor 1221 and Aroclor 1242 at a ratio of 3:1 as discussed in Phase 2 RAM QAPP Revised Attachment A, Section 4. A laboratory control sample duplicate (LCSD) will also be prepared for wipe samples since it is not possible to collect triplicate volume for wipe samples in order to perform MS/MSD analysis.
- Wipe samples will be extracted in accordance with the procedures specified in Appendix O2-1 (Pace SOP S-NY-O-088-rev.10). In addition, the percent moisture determination does not apply to wipe samples. The PCB results for wipe samples will be reported in units of µg/100 cm^2 instead of mg/kg. The PCB analysis will be performed in accordance with Method GEHR8082 (Appendix A4-1, Revised Attachment A) but the analytical method will be referenced as "GEHR8082WI" instead of "GEHR8082" in the analytical database due to these differences.

Measurement performance criteria for precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity have been established for the analytical procedures and are summarized in Tables O2-1a-b.

3 REFERENCES

Anchor QEA and ESI, 2012. *Phase 2 Remedial Action Monitoring Quality Assurance Project Plan. Hudson River PCBs Superfund Site.* Prepared for General Electric Company, Albany, New York. May 2012.

ESI, 2014. *Analytical Program Approach and Procedures*. Revised Attachment A to the Phase 2 RAM QAPP (Anchor QEA and ESI, 2012). Prepared for General Electric Company, Albany, New York. October 2014.

TABLES

Table O1-1

Laboratory Analyses and Certifications for the Sediment Processing Facility Decontamination-Related Samples

Matrix	Category	Laboratory	Analyte Name	CAS Number	Analytical Method	Certification
Facility Decontamination-Related Solids and Wipes	PCBs	Pace	Aroclor 1016	12674-11-2	GEHR8082/GEHR8082WI	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1221	11104-28-2	GEHR8082/GEHR8082WI	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1232	11141-16-5	GEHR8082/GEHR8082WI	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1242	53469-21-9	GEHR8082/GEHR8082WI	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1248	12672-29-6	GEHR8082/GEHR8082WI	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1254	11097-69-1	GEHR8082/GEHR8082WI	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1260	11096-82-5	GEHR8082/GEHR8082WI	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Total PCBs (sum of Aroclors)	1336-36-3	GEHR8082/GEHR8082WI	No Certification Available
Facility Decontamination-Related Solids	Other	Pace	Percent Moisture	WC002	ASTM D2216-98	No Certification Available

Notes:

Pace - Pace Analytical Services, Inc. of Schenectady, New York

Table O1-2
Reference Limit and Evaluation for the Sediment Processing Facility Decontamination-Related Samples

Matrix	Category	Analyte Name	CAS number(s)	Analytical Method	Units	Laboratory Method Detection Limits[1]	Laboratory Reporting Limits[1]
Facility Decontamination-Related Solids	PCBs (Aroclors) - Microwave	Aroclor 1016	12674-11-2	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1221	11104-28-2	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1232	11141-16-5	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1242	53469-21-9	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1248	12672-29-6	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1254	11097-69-1	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1260	11096-82-5	GEHR8082	mg/kg	0.019	0.050
		Total PCBs (sum of Aroclors)	1336-36-3	GEHR8082	mg/kg	0.019	0.20
	PCBs (Aroclors) - ASE	Aroclor 1016	12674-11-2	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1221	11104-28-2	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1232	11141-16-5	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1242	53469-21-9	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1248	12672-29-6	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1254	11097-69-1	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1260	11096-82-5	GEHR8082	mg/kg	0.029	0.050
		Total PCBs (sum of Aroclors)	1336-36-3	GEHR8082	mg/kg	0.029	0.20
	Other	Percent Moisture	WC002	ASTM D2216-98	%	NA	0.020
Wipes	PCBs (Aroclors)	Aroclor 1016	12674-11-2	GEHR8082WI	μg/100 cm^2	0.269	0.50
		Aroclor 1221	11104-28-2	GEHR8082WI	μg/100 cm^2	0.269	0.50
		Aroclor 1232	11141-16-5	GEHR8082WI	μg/100 cm^2	0.269	0.50
		Aroclor 1242	53469-21-9	GEHR8082WI	μg/100 cm^2	0.269	0.50
		Aroclor 1248	12672-29-6	GEHR8082WI	μg/100 cm^2	0.269	0.50
		Aroclor 1254	11097-69-1	GEHR8082WI	μg/100 cm^2	0.269	0.50
		Aroclor 1260	11096-82-5	GEHR8082WI	μg/100 cm^2	0.269	0.50
		Total PCBs (sum of Aroclors)	1336-36-3	GEHR8082WI	μg/100 cm^2	0.269	0.50

Notes

[1] The MDLs and RLs will be adjusted for sample specific factors such as % solids, weights/volumes and dilutions that vary from the standard procedure. Sample-specific MDLs and RLs are highly matrix dependent. The MDLs and RLs reported for the Wipe Matrix are based on a surface of 100 cm^2. Data will be evaluated against sample-specific MDLs and RLs. Non-detects, or values detected at a level below the sample specific MDL, will be reported as the sample specific MDL and U flagged (with the exception of analytes where MDL is NA). Values detected above the sample-specific MDL and below the sample-specific RL will be reported and flagged as estimated ("J").

NA - Not Applicable. Method detection limit (MDL) reporting will not be used for this anlayte. The analyte will be reported to the Reporting Limit (RL).

Table O2-1a
Measurement Performance Criteria for the Sediment Processing Facility Decontamination-Related Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory or Equipment Blank	S&A
	50-150 %R	Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD, Wipes only) (spiked with Aroclor 1242)	A
	50-150 %R	Matrix Spike (MS) and Matrix Spike Duplicate (MSD) (Not Applicable to Wipes) (spiked with Aroclor 1242)	S&A
	60-140 %R	Surrogates (TCMX and DCB)	A
Precision	The RPD for field duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Field Duplicates (Co-Located Samples for Wipes)	S&A
	RPD should be ≤ 40%	MS/MSD (Not Applicable to Wipes)	S&A
	RPD should be ≤ 40%	LCS/LCSD (Wipes)	A
Sensitivity	See Table O1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Facility Decontamination-Related Solids and Wipes
Analytical Parameter: Total PCBs as Aroclors
Concentration Level: Low to High
Method: GEHR8082/GEHR8082WI (Phase 2 RAM QAPP Revised Attachment A Appendix A4-1)

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] *Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability).*

Table O2-1b

Measurement Performance Criteria for the Sediment Processing Facility Decontamination-Related Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Precision	The RPD for field duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	The RPD for lab duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Laboratory Duplicates	A
Sensitivity	See Table O1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Facility Decontamination-Related Solids
Analytical Parameter: Percent Moisture
Concentration Level: Low to High
Method: Per Extraction SOPs (Phase 2 RAM QAPP Revised Attachment A Appendices A4-3 through A4-5)

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] *Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability).*

APPENDIX O2-1
SOP FOR THE SOXHLET EXTRACTION OF WIPE SAMPLES FOR PCB ANALYSIS BY SW-846 METHOD 3540C (S-NY-O-088-REV.10)

STANDARD OPERATING PROCEDURE

SOXHLET EXTRACTION OF WIPE SAMPLES FOR PCB ANALYSIS

Reference Methods: EPA Method 3540C

SOP Number:	S-NY-O-088-rev.10
Effective Date:	07/08/15
Supersedes:	S-NY-O-088-rev.09

APPROVALS

Assistant General Manager — Date: 07/08/15

Quality Manager — Date: 07/08/15

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date

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S-NY-O-088-rev.10

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1. Purpose/Identification of Method

1.1. This is a Standard Operating Procedure (SOP) for the extraction of wipe samples for Polychlorinated Biphenyl (PCB) analysis using the Soxhlet extraction technique by SW-846 Method 3540C for subsequent analysis by SW-846 Method 8082A (Aroclor Specific Analysis).

2. Summary of Method

2.1. Using metal tweezers, a wipe is placed into a Soxhlet apparatus that contains 200mL of hexane.

2.2. The samples are spiked and surrogated directly onto the wipe.

2.3. After an 18 +/- 2 hour extraction the derived solvent is concentrated..

2.4. The extract may be put through cleanup processes (see separate SOPs) and is then properly diluted and submitted for GC analysis.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the Soxhlet extraction of wipe samples

3.2. **Parameters**: See analytical method SOPs for PCB analysis.

4. Applicable Matrices

4.1. This test method is appropriate for the extraction and cleanup of wipe samples. Projects performed for compliance with the US-EPA Toxic Substances Control Act (40 CFR 761) require Soxhlet Extraction. Extract cleanup steps employed may vary from sample to sample, matrix to matrix. The chemist must have an understanding of the methods and requirements of USEPA-SW- 846 "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition. Methods 3545A and 3500C. The chemist must also be certified to perform the procedure by an approved instructor. The chemist must have performed an acceptable demonstration of precision and accuracy before performing this procedure without supervision.

5. Limits of Detection and Quantitation

5.1. Please see determinative method (Lab SOP S-NY-O-314) for details.

6. Interferences

6.1. Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of certain plastics. Phthalate esters respond on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Samples and extracts should not be exposed to plastics such as gloves, tubing, coating on clamps, and pipette bulbs, etc.

7. Sample Collection, Preservation, Shipment and Storage

7.1. All wipes samples should be collected in 4 oz jars with Teflon lined lids.

7.2. All wipes should be stored in the walk in refrigerator at 0-6°C before extraction.

7.3. For EPA Method 8082A the hold time for extraction is up to one year from date of collection.

7.4. The extracted solvents must be analyzed within 40 days of their extraction date.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. **Surrogate Standard Solution:** The chemical composition and chromatography of surrogates are similar to the analytes of interest. They are usually not found in environmental samples. These compounds are spiked into all samples, blanks, and matrix spike samples prior to extraction. Percent recoveries are calculated for each surrogate.

8.3. **Laboratory Method Blank:** A laboratory derived sample consisting of a sterile gauze pad that is carried through all extraction and cleanup steps. The laboratory method blank is used to define the level of laboratory analyte background or other interferences that exist in the laboratory environment, the reagents, or extraction apparatus.

8.4. **Laboratory Control Sample (LCS):** Also known as the Quality Control (QC) Check Standard or Quality Control (QC) Check Sample. The LCS consists of a sterile gauze pad to which known quantities of the method analytes are added. The LCS is extracted and cleaned up exactly like a field sample, and its purpose is to determine whether the analysis is in control and whether the laboratory is capable of making accurate and precise measurements.

8.5. **Lab Control Sample Duplicate:** An exact copy of the Lab Control Sample to further assess analyte recovery efficiency.

8.6. **Matrix Spike (MS):** An aliquot of a field sample that is fortified with known quantities of the method analytes and is carried through all the extraction and cleanup steps. Its purpose is to assess the appropriateness of the method for the matrix by measuring the recovery of the method analytes.

8.7. **Sample Matrix Spike Duplicate (MSD):** An exact copy of the Matrix Spike. This is an aliquot of a field sample which is fortified with known quantities of the method analytes and is subject to the entire procedure. Its purpose is to assess the appropriateness of the method for the matrix by measuring the recovery of the method analytes.

8.8. **QC-Quality Control:** A set of measures for each sample within an analysis methodology to assure that the process is in control.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Water Cooled Condenser: Pyrex 45/50 #3840-MCO (or equivalent).

9.2. 250mL Round Bottom Flask: Pyrex #4100 (or equivalent).

9.3. Heating Mantle: Type "VF" laboratory heating mantle #HM0250VF1 (or equivalent).

9.4. Soxhlet Repetitive Flushing (reflux) Unit: 45/50 Pyrex. 45/50 – 24/40 joints (CG-13608-08) (or equivalent).

9.5. Heating Mantle Controller: Glass-Col #PL3122 Minitwin (or equivalent) regulates temperature control of the mantle.

9.6. Cellulose Extraction Thimble: Advantec Thimble Filters (cat. #NO8433 x 80MM) (or equivalent).

9.7. Chiller: Pump driven water circulating cooling system cool flow #75 NESLABS Instruments, Inc. (or equivalent).

9.8. Analytical Balance: Mettler PL-303 (or equivalent) used to determine sample mass.

9.9. Sterile Gauze Pad: Used for Blank and LCS.

9.10. Turbo Vap Evaporator: Zymark #61392/3 (or equivalent).

9.11. **PowerVap Evaporator:** Fluid Management Systems (or equivalent).

9.12. TurboVap Evaporator concentrator tubes: Zymark 250mL, 0.5mL endpoint.

9.13. Vials: glass, 40mL vial and 4 dram (with Polyseal sealed cap), for sample extracts.

9.14. Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.

9.15. Centrifuge: International Equipment Co., Model CL (or equivalent).

9.16. Wrist Shaker: Burrell wrist action shaker, Model 75 and 88 (or equivalent).

9.17. Pipettes: S/P Disposable Serological Borosilicate Pipettes:

9.17.1. 1mL X 1/10.

9.17.2. 5mL X 1/10.

9.17.3. 10mL X 1/10.

9.17.4. Fisher Brand Borosilicate glass pipette (or equivalent).

9.18. Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL, used for liquid containment and pipette storage.

10. Reagents and Standards

10.1. Sodium Sulfate: J.T.Baker, #3375-05 Anhydrous, Granular (12-60 Mesh) (or equivalent). Used for the laboratory method blank and laboratory control sample.

10.2. Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208 (or equivalent).

10.3. Acetone: P/N CS011-200 Honeywell Burdick & Jackson. (or equivalent)

10.4. 1:1 Hexane/Acetone: 50%:50% by volume solvent mixture, prepared in the lab.

10.5. Spike standard solution: PCB Aroclor at 12.5ug/mL in acetone:

10.5.1. To make a 12.5ug/mL Aroclor spike: Allow the Stock Standard Solution (SOP S-NY-O-148) warm up to room temperature. Using a gastight syringe, add 1.25mL of the Stock Standard Solution to a 100mL volumetric flask and fill with acetone. All information is recorded in the Standards logbook.

10.6. Surrogates Standards: Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) TCMX/DCBP at 0.5/5.0ug/mL in acetone:

10.6.1. To make this standard an ampule of Custom Standard CUS-4911 (Ultra Scientific) is brought to room temperature and shaken on a wrist action shaker for at least 30 minutes. Once the standard is room temperature 1mL is added to 1L of acetone. All information is recorded in the Standards logbook.

11. Calibration and Standardization

11.1. Please see determinative method (Lab SOP S-NY-O-314) for details.

12. Procedure

12.1. **Sample Preparation**:

12.1.1. Throughout the entire process it should be noted that if the chemist encounters any problems or difficulties with any samples or steps involved, all work should **STOP**! Any problems should be brought to the attention of the supervisor and documented in the Laboratory Information Management System (LIMS).

12.1.2. Before any steps are taken, the chemist should first review the sample job folder. The chemist should also verify the sample IDs on the bottle against the chain of custody. If there is a discrepancy on either the sample label or the chain of custody, this should be documented in LIMS and brought to the attention of the sample receiving supervisor.

12.2. **Sample Extraction**:

12.2.1. Rinse enough cellulose extraction thimbles for one per sample and QC sample with hexane. Allow them to dry out in a 4 oz. jar in a fume hood. Label the 4 oz. jars with the sample ID.

12.2.2. Obtain enough 250mL round bottoms and Soxhlets for each sample. Inspect the glassware for cracks or chips that will allow solvent to leak out. Add several boiling chips and approximately 200mL of 1:1 Hexane/Acetone to each round bottom. Place a Soxhlet extractor on top of the round bottom and label each round bottom with a sample number. Record the round bottom and Soxhlet used for each sample in LIMS.

12.2.3. Using metal tweezers, place each sample and QC wipe directly into the Soxhlet using tweezers. Rinse the sample jar with hexane and pour the hexane into the Soxhlet. **NOTE:** Rinse the tweezers thoroughly between samples to avoid cross contamination. Use a sterile gauze pad for the method blank, lab control spike, and lab control spike duplicate.

12.2.4. Add surrogate to all samples and spike to the appropriate QC samples. The surrogate and spike should be added directly to the wipe sample in the soxhlet. Spiking must be done with a witness and recorded in LIMS.

12.2.5. Using hexane, rinse the inside and the outer connecting joint of the condenser units to be used. Turn on the chiller units that will be used to cool the condensers.

12.2.6. Place the Soxhlet extractors into heating mantles and attach the condensers. Turn on the corresponding control units to a setting of 5.5. Double check the Soxhlet and round bottoms at this time for cracks or chips.

12.2.7. Once the solvent begins to boil, a flushing action of 4-6 flushes per hour should be reached. Allow the extraction to proceed for 18 hours ± 2 hours (usually overnight).

12.2.8. After the extraction time has finished, turn off the heating mantles and allow the samples to cool to room temperature. Rinse the inside of the condenser with several pipette volumes of hexane. Disengage the Soxhlet extractor from the condenser, rinse the connecting joint into the Soxhlet, and remove to a fume hood.

12.2.9. Tip the extractor to flush the solvent remaining in the Soxhlet into the round bottom. Using a pair of long-handled tweezers, pull the thimble to the top of the Soxhlet and allow them to drip-dry by balancing them on the edge of the Soxhlet. Rinse tweezers between each sample. Once the thimbles are dry, remove them to a sheet of aluminum foil in the hood for total evaporation. When completely dry, fold them in the foil and dispose of them.

12.2.10. Rinse the Soxhlet with several pipette volumes of hexane and tip to drain into the round bottom.

12.2.11. Procure the same number of Turbo Tubes as there are samples. Pre-rinse turbo tubes with hexane and allow to dry. Using an individual Turbo Tube stand, label a TurboTube with the corresponding sample ID number and place in the holder.

12.2.12. Add a suitable amount of sodium sulfate to round bottom flask and swirl around to remove any residual water. Pour, decanting off the sodium sulfate, the contents of the round bottom into the TurboTube, using a pipette and hexane to rinse the last drops out of the mouth of the round bottom. Rinse the round bottom with several pipette volumes of hexane, swirl gently, and decant into same TurboTube. Repeat this step twice for same sample then repeat all preceding steps for all other samples.

12.2.13. All glassware must be rinsed with acetone and dried in the hood before other cleaning steps.

12.3. **Solvent Reduction- TurboVap/PowerVap:**

12.3.1. The TurboVap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The TurboVap uses a heated water bath and positive pressure nitrogen flow/vortex action. The PowerVap uses a heated aluminum block as well as positive pressure nitrogen flow/vortex action. Both units maintain a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract, which allows fractional reduction of the solvents without loss of higher boiling point analytes.

12.3.2. Turn the TurboVap on and allow it to heat up to 40 ± 2°C. If using the PowerVap, turn on and allow it to heat up to 60 ± 2°C.

12.3.3. As a precaution both systems regulators should be checked to assure that no residual gas pressure remains within the system and that gas pressure regulators is off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty TurboTube into the water bath and close the lid. Ensure that the nitrogen gas pressure regulator is turned off. Bleed any residual gas until the regulator output pressure gauge reads "0" psi.

12.3.4. Rinse each tip and wipe down all surfaces of the TurboVap/Powervap with solvent. Close the lid and turn the pressure up to blow the lines clean. Turn off the pressure and bleed the system of any residual gas.

12.3.5. Place the TurboTubes containing the samples into the TurboVap/PowerVap and close the lid. Begin slowly turning the pressure regulator on. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the reduction.

12.3.6. The process for solvent reduction takes approximately 30-45 minutes. Do not leave the unit unattended as extracts may be blown to dryness and PCB loss may occur.

12.3.7. Concentrate the solvent to approximately 10.0mL. Remove the samples from the TurboVap and place in the rack. **NOTE:** Not all samples will evaporate at the same rate; sample extracts containing large amounts of petroleum or other non-volatile liquids may stop reducing before the 10.0mL point is achieved. Samples which stop reducing should be removed as soon as possible.

12.3.8. Quantitatively transfer the sample extract with a Pasteur pipette into an appropriate volumetric flask (25mL is the default set volume for solid extracts). Rinse the TurboTube with 3 Pasteur pipettes of hexane, and then transfer the Hexane rinse to the volumetric. Repeat the hexane rinse two more times for a total of three hexane rinses of the TurboTube. After the sample has been transferred, rinse the Pasteur pipette with 0.5mL of hexane into the volumetric flask. Add hexane to bring the solvent level up to exactly the meniscus mark on the volumetric. Stopper and invert the volumetric flask to mix. Decant the contents into a pre-labeled 40mL vial.

12.3.9. All used glassware must be rinsed with tech-acetone or a "For Rinsing-Only" labeled solvent and dried in the fume hood before being washed.

12.4. **Sample Extract Cleanup:**

12.4.1. Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection, contain co-extracted interfering substances which must be removed before accurate chromatographic analysis can be performed.

12.4.2. See separate cleanup SOPs for details (S-NY-O-337, S-NY-O-338, S-NY-O-339 and S-NY-O-340, as applicable).

12.5. **Extract Screening and Dilution:**

12.5.1. PCB extracts are generally screened by GC initially to determine the approximate concentration before final analysis. Prior site history and client supplied estimates of sample concentration may be used to determine what, if any, extract dilution is necessary. Extracts of unknown concentration are generally screened at a 10 to 100 fold dilution.

12.5.2. The supervising chemist is responsible for determining initial screening dilutions. Extract dilutions are prepared by transferring an aliquot of the original sample extract into a vial containing the correct amount of "make up" volume of hexane. Dilutions must be recorded in LIMS.

12.5.3. Perform the dilution using appropriate class "A" disposable volumetric pipettes to transfer the extract and to add the make-up volume of Hexane. Make sure that the vial is properly labeled. Cap and invert the vial at least three times to thoroughly mix the extract with the solvent.

12.5.4. Transfer 1mL of the extract to a labeled 1.5mL GC autosampler vial. Record the sample data in LIMS and submit with the sample extracts to the GC analyst.

13. Quality Control

13.1. The extraction chemist should have completed an acceptable demonstration of precision and accuracy before performing the method without supervision. The addition of spiking material to a sample or blank must be witnessed by another extraction chemist and signed in the extraction logbook. All surrogates and matrix spikes must meet acceptable QC limits.

13.2. A method blank sample, lab control sample, and lab control sample duplicate must be prepared per each extraction batch of up to 20 samples. Spike default for LCS,and LCSD is 1.0mL of A1242 at 12.5ug/mL in acetone. Client and/or project specifications may dictate alternate amount or Aroclor.

13.3. PCB Surrogates TCMX and DCBP are added to each sample prior to extraction to measure extraction/cleanup efficiency. Default surrogate is: 0.5mL of 0.5ug/mL TCMX / 5.0ug/mL DCBP in acetone. Client and/or project specifications may dictate alternate amount.

14. Data Analysis and Calculations

14.1. Please see determinative method (Lab SOP S-NY-O-314) for details.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Please see determinative method (Lab SOP S-NY-O-314) for details.

16. Corrective Actions for Out-of-Control Data

16.1. Please see determinative method (Lab SOP S-NY-O-314) for details.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Please see determinative method (Lab SOP S-NY-O-314) for details.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. Please see determinative method (Lab SOP S-NY-O-314) for details.

19. Method Modifications

19.1. Use of a drying column containing sodium sulfate to dry the extract in method 3540C differs from the laboratory's use of lose sodium sulfate.

19.2. Determinative method 8082A requires a matrix spike/duplicate or matrix spike/matrix spike duplicate pair for each batch of up to 20 samples. Due to the nature of wipe samples, the laboratory does not typically receive the additional sample volume needed to meet this requirement. Instead, a lab control sample duplicate is extracted with every batch of up to 20 samples to allow for calculation of method precision.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Polychlorinated biphenyls should be treated with extreme caution; as a class of chemical compounds they possess both toxic and suspected carcinogenic properties. Refer to SDS for further details.

22.2. The extraction technician should have received in-house safety training and should know the location of the first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment.

22.3. Safety glasses, a lab coat and gloves must be worn when handling glassware and samples.

23. Waste Management

23.1. See SOP S-NY-W-054.

24. Pollution Prevention

24.1. See SOP S-NY-S-168.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

25.5. **"**Guide to Environmental Analytical Methods", Third Edition, Genium Publishing Corporation, 1996.

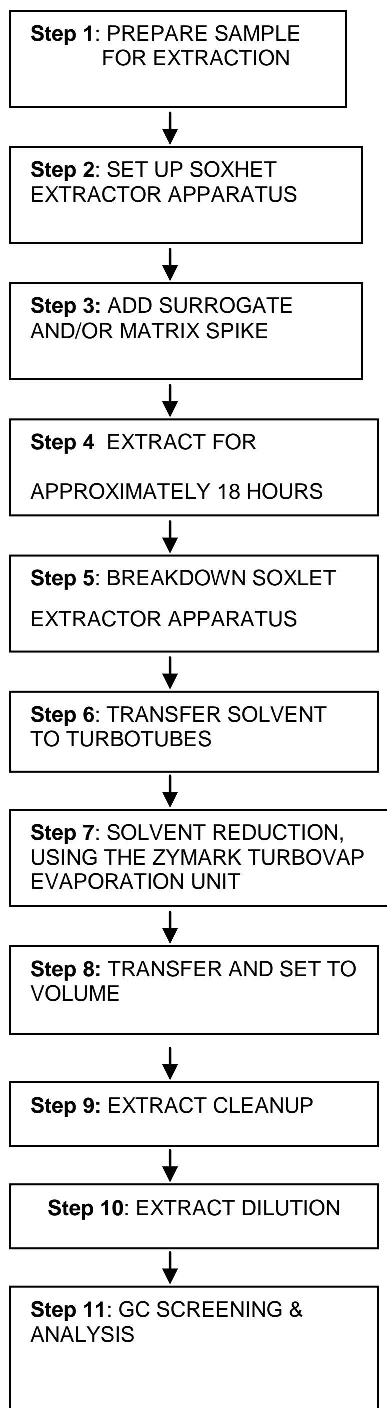
26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Flowchart for the Extraction and Clean-up of Wipes for PCB Analysis.

27. Revisions

Document Number	Reason for Change	Date
S-NY-O-088-rev.09	General: converted to new format. Section 10: removed reagents involved in cleanup procedures. Section 12.4: removed cleanup procedures and added references for cleanup SOPs. Sections 25.1-25.3: added standard Pace references.	27Jan2015
S-NY-O-088-rev.09	Sections 9.11, 12.3.2, 12.3.5: added PowerVap system Section 10.3: added Acetone Sections 10.4, 12.2.2: changed to use of 1:1 Acetone/Hexane Sections12.2.3 and 13.2: added requirement for LCSD with every batch Section 19.1: documented use of lose Sodium Sulfate as a modification Section 19.2: documented use of LCSD in place of MS/DUP as a modification	08July2015

Attachment I: Flowchart for the Extraction and Cleanup of Wipes for PCB Analysis



Attachment P –

Analytical Approach and Procedures –

Environmental Media Samples

ATTACHMENT P

ANALYTICAL PROGRAM
APPROACH AND PROCEDURES
ENVIRONMENTAL MEDIA SAMPLES

Prepared for

General Electric
Albany, New York

Prepared by
Environmental Standards, Inc.
Valley Forge, Pennsylvania

Revised September 2015

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Appendix P3-5	SOP for the Determination of Polychlorinated Biphenyls (PCBs) Aroclors in Aqueous Samples by SW-846 Method 8082A (S-NY-O-314-rev.03)
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Appendix P3-7	SOP for the Preparation and Analysis of Mercury by Manual Cold Vapor Technique by SW-846 Method 7470A (S-LI-M-002-rev.01)
Appendix P3-8	SOP for Total Recoverable Oil and Grease Analysis in Waters n-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetery by EPA 1664A (S-LI-I-016-rev.02)

LIST OF ACRONYMS AND ABBREVIATIONS

%R	Percent Recovery
µg/L	micrograms per liter
COC	Chain-of-Custody
DQI	Data Quality Indicators
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
EPA	United States Environmental Protection Agency
FSP	Field Sampling Plan
GC/ECD	Gas Chromatograph/Electron Capture Detector
GE	General Electric Company
L	liter
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MDL	Method Detection Limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mL	milliliter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NELAP	National Environmental Accreditation Program
ng/L	nanograms per liter
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
PASRCC	Precision, Accuracy/Bias, Sensitivity, Representativeness, Comparability and Completeness
PCB	Polychlorinated Biphenyl
PE	Performance Evaluation
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QC	Quality Control
RAM QAPP	Remedial Action Monitoring Quality Assurance Project Plan
RL	Reporting Limit
RPD	Relative Percent Difference
SOP	Standard Operating Procedure
SPF	Sediment Processing Facility
STARS	Spill Technology and Remediation Series
TAL	Target Analyte List
TAT	Turn-around Time
TCL	Target Compound List
TOC	Total Organic Carbon
VTSR	Verified Time of Sample Receipt

1 INTRODUCTION

1.1 Analytical Program Summary

Table P1-1 lists the analyses that will be performed by each laboratory for the environmental media samples collected as part of the Sediment Processing Facility (SPF) Demobilization. The responsibilities and duties of the analytical laboratories will be consistent with those described in the Phase 2 Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP, Anchor QEA and ESI 2012) including the October 2014 revision to the Analytical Approach and Procedures (Revised Attachment A, ESI 2014) and subsequent updates. The analyte lists and method detection limits (MDLs) and reporting limits (RLs) for each analyte and method are listed in Table P1-2. Quality assurance/quality control (QA/QC) samples originating in the field are described in Section 5 of the SPF Demobilization Field Sampling Plan (FSP). Laboratory QA/QC samples are described in the standard operating procedures (SOPs) provided in the Phase 2 RAM QAPP or appended to this attachment as noted below.

All samples will be submitted with standard turnaround times (10 business days for the electronic data deliverable (EDD) and 20 business days for the hard copy data package). Laboratory analytical data for this project will be reported in both an EDD and an analytical data package. Analytical data packages will be prepared by the laboratories according to the procedures described in the SOP "Data Package Deliverable," which is included in Appendix P1-1. Full data packages (Level B) will be provided for all sample analyses. Data packages will be provided by the laboratory in an Adobe® Acrobat® .pdf electronic format.

1.2 Special Training/Certification

The laboratory will have sufficient personnel with the necessary education, training, technical knowledge, and experience for their assigned functions as described in Revised Attachment A. The laboratories participating in this project will be accredited through New York State's Environmental Laboratory Accreditation Program (ELAP) and the National Environmental Laboratory Accreditation Program (NELAP) for the analyses being performed. Table P1-1 lists the certification status or if certification is not available for that analyte.

2 SOIL AND SEDIMENT SAMPLE ANALYTICAL PROCEDURES

The SPF Demobilization will involve the analysis of soil and sediment samples. Soil and sediment samples will be collectively analyzed for Aroclor and Total PCBs, moisture content, total organic carbon (TOC), New York State Department of Environmental Conservation (NYSDEC) Spills Technology and Remediation Series (STARS; Tables 2-3 in Final Commissioner Policy CP-51 Soil Cleanup Guidance, NYSDEC 2010) volatile organic compounds (VOCs), STARS and target compound list (TCL) semivolatile organic compounds (SVOCs), gasoline range organics (GRO), and diesel range organics (DRO). Analyses to be performed for specific samples are specified in the SPF Demobilization FSP.

PCB and moisture content analyses of soil and sediment samples will be performed in accordance with Method GEHR8082 (Appendix A4-1, Revised Attachment A), and the associated extraction SOPs (Appendices A4-3 through A4-5, Revised Attachment A), with the following exceptions:

- Performance Evaluation samples will not be prepared and analyzed as discussed in Section 11.2.1 of the Phase 2 RAM QAPP. Instead, matrix spike/matrix spike duplicate (MS/MSD) samples spiked with only Aroclor 1242 will be prepared and analyzed at a frequency of of five percent (i.e., one for every 20 samples) of each matrix.
- Laboratory control spike (LCS) will consist of only Aroclor 1242 instead of a combination of Aroclor 1221 and Aroclor 1242 at a ratio of 3:1 as discussed in Phase 2 RAM QAPP Attachment A, Section 4.

Select soil samples will collectively include analysis for VOCs, SVOCs, GRO, and DRO according to the following SOPs:

- Appendix P2-1 Pace Long Island SOP S-LI-O-012-rev.00 – Sample Preparation for Volatile Organics by SW-846 Method 5035A
- Appendix P2-2 Pace Long Island SOP S-LI-O-011-rev.00 – Determination of Volatile Organics by GC/MS by SW-846 Method 8260C
- Appendix P2-3 Pace Long Island SOP S-LI-O-020-rev.00 – Pressurized Fluid Extraction by SW-846 Method 3545A
- Appendix P2-4 Pace Long Island SOP S-LI-O-014-rev.00 – Determination of Semi-volatile Organics by GC/MS by SW-846 Method 8270D
- Appendix P2-5 Pace Long Island SOP S-LI-O-007-rev.01 – Determination of Gasoline Range and Diesel Range Organics by GC/FID by SW-846 Method 8015D

Select sediment samples will also include analysis for TOC by the Lloyd Kahn method (Appendix A6-1, Revised Attachment A).

Measurement performance criteria for precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity have been established for the analytical procedures and are summarized in the Tables P2-1a-f.

3 AQUEOUS SAMPLE ANALYTICAL PROCEDURES

The SPF Demobilization will involve the analysis of groundwater and surface water samples. Groundwater and surface water samples will be collectively analyzed for PCBs, TCL VOCs, TCL SVOCs, TCL pesticides, target analyte list (TAL) metals (including mercury), and oil and grease. Analyses to be performed for specific samples are specified in the SPF Demobilization FSP. The analyses will be performed according to the following SOPs (To be modified upon selection of an analytical laboratory):

- Appendix P3-1 Pace Long Island SOP S-LI-O-013-rev.00 – Sample Preparation for Volatile Organics by SW-846 Method 5030C
- Appendix P2-2 Pace Long Island SOP S-LI-O-011-rev.00 – Determination of Volatile Organics by GC/MS by SW-846 Method 8260C
- Appendix P3-2 Pace Long Island SOP S-LI-O-004-rev.00 – Separatory Funnel Extraction by SW-846 Method 3510C
- Appendix P2-4 Pace Long Island SOP S-LI-I-014-rev.00 – Determination of Semi-volatile Organics by GC/MS by SW-846 Method 8270D

- Appendix P3-3 Pace Long Island SOP 8081B (Revision 3) – Sample Preparation and Analysis of Chlorinated Pesticides by SW-846 Method 8081B
- Appendix P3-4 Pace Schenectady SOP S-NY-O-218-rev.08 - Extraction of Aqueous Samples by Solid Phase Extraction by SW-846 Method 3535A (prior to PCB analysis by SW-846 Method 8082A)
- Appendix P3-5 Pace Schenectady SOP S-NY-O-314-rev.03 – Determination of Polychlorinated Biphenyls (PCBs) Aroclors by SW-846 Method 8082A
- Appendix P3-6 Pace long Island SOP S-LI-M-001-rev.00 – Preparation and Analysis of Trace Metals by ICP-AES by SW-846 Methods 3005A and 6010C
- Appendix P3-7 Pace Long Island SOP S-LI-M-002-rev.01 – Preparation and Analysis of Mercury by Manual Cold Vapor Technique by SW-846 Method 7470A
- Appendix P3-8 Pace Long Island SOP S-LI-I-016-rev.02 - Total Recoverable Oil and Grease Analysis in Waters n-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetery by EPA 1664A

Measurement performance criteria for precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity have been established for the analytical procedures and are summarized in Tables P3-1a-g.

4 REFERENCES

Anchor QEA and ESI, 2012. *Phase 2 Remedial Action Monitoring Quality Assurance Project Plan. Hudson River PCBs Superfund Site.* Prepared for General Electric Company, Albany, New York. May 2012.

ESI, 2014. *Analytical Program Approach and Procedures*. Revised Attachment A to the Phase 2 RAM QAPP (Anchor QEA and ESI, 2012). Prepared for General Electric Company, Albany, New York. October 2014.

NYSDEC, 2010. Final Commissioner Policy CP-51 Soil Cleanup Guidance, Issued 10/21/2010; Effective 12/03/2010

TABLES

Table P1-1
Laboratory analyses and certifications for the SPF Demobilization Environmental Media Samples

Matrix	Category	Laboratory	Analyte Name	CAS Number	Analytical Method	Certification
Soil and Sediment	PCBs (Aroclors)	Pace Schenectady	Aroclor 1016	12674-11-2	GEHR8082	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1221	11104-28-2	GEHR8082	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1232	11141-16-5	GEHR8082	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1242	53469-21-9	GEHR8082	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1248	12672-29-6	GEHR8082	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1254	11097-69-1	GEHR8082	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Aroclor 1260	11096-82-5	GEHR8082	NYSDOH Solid and Hazardous Waste - SW846 8082A
			Total PCBs (sum of Aroclors)	1336-36-3	GEHR8082	No Certification Available
	Other	Pace Schenectady	Total Organic Carbon	OC002	Lloyd Kahn	No Certification Available
			Percent Moisture	WC002	ASTM D2216-98	No Certification Available
	STARS Volatile Organic Compounds	Pace Long Island	1,2,4-Trimethylbenzene	95-63-6	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			1,3,5-Trimethylbenzene	108-67-8	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			1.4-Dioxane	123-91-1	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			Benzene	71-43-2	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			Ethylbenzene	100-41-4	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			Isopropylbenzene	98-82-8	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			Methyl-Tert-Butyl-Ether	1634-04-4	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			Naphthalene	91-20-3	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			n-Butylbenzene	104-51-8	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			n-Propylbenzene	103-65-1	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			p-Isopropyltoluene	99-87-6	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			sec-Butylbenzene	135-98-8	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			Tert-Butylbenzene	98-06-6	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			Toluene	108-88-3	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
			Xylene (Mixed)	1330-20-7	SW-846 8260C	NYSDOH Solid and Hazardous Waste - SW846 8260C
	TCL Semivolatile Organic Compounds	Pace Long Island	1,1'-Biphenyl	92-52-4	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			1,2,4,5-Tetrachlorobenzene	95-94-3	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2,2'-Oxybis(1-choloropropane)	108-60-1	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2,3,4,6-Tetrachlorophenol	58-90-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2,4,5-Trichlorophenol	95-95-4	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2,4,6-Trichlorophenol	88-06-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2,4-Dichlorophenol	120-83-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2,4-Dimethylphenol	105-67-9	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2,4-Dinitrophenol	51-28-5	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2,4-Dinitrotoluene	121-14-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2,6-Dinitrotoluene	606-20-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2-Chloronaphthalene	91-58-7	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2-Chlorophenol	95-57-8	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2-Methylnaphthalene	91-57-6	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2-Methylphenol	95-48-7	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D

Table P1-1
Laboratory analyses and certifications for the SPF Demobilization Environmental Media Samples

Matrix	Category	Laboratory	Analyte Name	CAS Number	Analytical Method	Certification
Soil and Sediment (Continued)	TCL Semivolatile Organic Compounds (Continued)	Pace Long Island	2-Nitroaniline	88-74-4	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			2-Nitrophenol	88-75-5	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			3,3'-Dicholorobenzidine	91-94-1	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			3-Methylphenol	108-39-4	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			3-Nitroaniline	99-09-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			4,6-Dinitro-2-methylphenol	534-52-1	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			4-Bromophenyl-phenylether	101-55-3	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			4-Chloro-3-methylphenol	59-50-7	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			4-Chloroaniline	106-47-8	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			4-Chlorophenyl-phenyl ether	7005-72-3	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			4-Methylphenol	106-44-5	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			4-Nitroaniline	100-01-6	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			4-Nitrophenol	100-02-7	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Acenaphthene	83-32-9	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Acenaphthylene	208-96-8	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Acetophenone	98-86-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Anthracene	120-12-7	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Atrazine	1912-24-9	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Benzo(a) pyrene	50-32-8	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Benzo(a)anthracene	56-55-3	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Benzo(b) fluoranthene	205-99-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Benzo(g,h,i) perylene	191-24-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Benzo(k) fluoranthene	207-08-9	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Bis(2-chloroethoxy) methane	111-91-1	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Bis(2-chloroethyl) ether	111-44-4	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Bis(2-ethylhexyl) phthalate	117-81-7	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Butylbenzylphthalate	85-68-7	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Caprolactam	105-60-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Carbazole	86-74-8	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Chrysene	218-01-9	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Dibenzo(a,h) anthracene	53-70-3	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Dibenzofuran	132-64-9	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Diethylphthalate	84-66-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Dimethylphthalate	131-11-3	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Di-n-butylphthalate	84-74-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Di-n-octylphthalate	117-84-0	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Fluoranthene	206-44-0	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Fluorene	86-73-7	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Hexachlorobenzene	118-74-1	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Hexachlorobutadiene	87-68-3	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Hexachlorocyclopentadiene	77-47-4	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D

Table P1-1
Laboratory analyses and certifications for the SPF Demobilization Environmental Media Samples

Matrix	Category	Laboratory	Analyte Name	CAS Number	Analytical Method	Certification
Soil and Sediment (Continued)	TCL Semivolatile Organic Compounds (Continued)	Pace Long Island	Hexachloroethane	67-72-1	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Indeno(1,2,3,-cd) pyrene	193-39-5	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Isophorone	78-59-1	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Naphthalene	91-20-3	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Nitrobenzene	98-95-3	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			N-Nitroso-di-n propylamine	621-64-7	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			N-Nitrosodiphenylamine	86-30-6	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Pentachlorophenol	87-86-5	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Phenanthrene	85-01-8	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Phenol	108-95-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Pyrene	129-00-0	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
	STARS Semivolatile Organic Compounds	Pace Long Island	Acenaphthylene	208-96-8	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Anthracene	120-12-7	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Benz(a)Anthracene	56-55-3	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Benzo(a)Pyrene	50-32-8	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Benzo(b)Fluoranthene	205-99-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Benzo(g,h,i)Perylene	191-24-2	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Benzo(k)Fluoranthene	207-08-9	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Chrysene	218-01-9	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Dibenzo(a,h)Anthracene	53-70-3	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Fluoranthene	206-44-0	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Fluorene	86-73-7	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Indeno(1,2,3-cd)Pyrene	193-39-5	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Phenanthrene	85-01-8	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
			Pyrene	129-00-0	SW-846 8270D	NYSDOH Solid and Hazardous Waste - SW846 8270D
	Gasoline Range Organics	Pace Long Island	Gasoline Range Organics		SW-846 8015D	NYSDOH Solid and Hazardous Waste - SW846 8015D
	Diesel Range Organics		Diesel Range Organics		SW-846 8015D	NYSDOH Solid and Hazardous Waste - SW846 8015D
Aqueous	TCL Volatile Organic Compounds	Pace Long Island	1,1,1-Trichloroethane	71–55–6	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,1,2,2-Tetrachloroethane	79–34–5	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,1,2-Trichloro-1,2,2-trifluoroethane	76–13–1	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,1,2-Trichloroethane	79–00–5	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,1-Dichloroethane	75–34–3	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,1-Dichloroethene	75–35–4	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,2,3-Trichlorobenzene	87–61–6	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,2,4-Trichlorobenzene	120–82–1	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,2-Dibromo-3-chloropropane	96–12–8	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,2-Dibromoethane	106–93–4	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,2-Dichlorobenzene	95–50–1	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,2-Dichloroethane	107–06–2	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C

Table P1-1
Laboratory analyses and certifications for the SPF Demobilization Environmental Media Samples

Matrix	Category	Laboratory	Analyte Name	CAS Number	Analytical Method	Certification
Aqueous (Continued)	TCL Volatile Organic Compounds (Continued)	Pace Long Island	1,2-Dichloropropane	78-87-5	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,3-Dichlorobenzene	541-73-1	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1,4-Dichlorobenzene	106-46-7	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			1.4-Dioxane	123-91-1	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			2-Butanone	78-93-3	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			2-Hexanone	591-78-6	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			4-Methyl-2-pentanone	108-10-1	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Acetone	67-64-1	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Benzene	71-43-2	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Bromochloromethane	74-97-5	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Bromodichloromethane	75-27-4	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Bromoform	75-25-2	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Bromomethane	74-83-9	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Carbon disulfide	75-15-0	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Carbon tetrachloride	56-23-5	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Chlorobenzene	108-90-7	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Chloroethane	75-00-3	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Chloroform	67-66-3	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Chloromethane	74-87-3	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			cis-1,2-Dichloroethene	156-59-2	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			cis-1,3-Dichloropropene	10061-01-5	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Cyclohexane	110-82-7	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Dibromochloromethane	124-48-1	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Dichlorodifluoromethane	75-71-8	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Ethylbenzene	100-41-4	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Isopropylbenzene	98-82-8	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			m,p-Xylene	179601-23-1	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Methyl acetate	79-20-9	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Methyl tert-butyl ether	1634-04-4	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Methylcyclohexane	108-87-2	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Methylene chloride	75-09-2	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			o-Xylene	95-47-6	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Styrene	100-42-5	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Tetrachloroethene	127-18-4	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Toluene	108-88-3	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			trans-1,2-Dichloroethene	156-60-5	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			trans-1,3-Dichloropropene	10061-02-6	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Trichloroethene	79-01-6	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Trichlorofluoromethane	75-69-4	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C
			Vinyl chloride	75-01-4	SW-846 8260C	NYSDOH Non-Potable Water - SW-846 8260C

Table P1-1
Laboratory analyses and certifications for the SPF Demobilization Environmental Media Samples

Matrix	Category	Laboratory	Analyte Name	CAS Number	Analytical Method	Certification
Aqueous (Continued)	TCL Semivolatile Organic Compounds	Pace Long Island	1,1'-Biphenyl	92-52-4	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			1,2,4,5-Tetrachlorobenzene	95-94-3	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2,2'-Oxybis(1-choloropropane)	108-60-1	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2,3,4,6-Tetrachlorophenol	58-90-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2,4,5-Trichlorophenol	95-95-4	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2,4,6-Trichlorophenol	88-06-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2,4-Dichlorophenol	120-83-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2,4-Dimethylphenol	105-67-9	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2,4-Dinitrophenol	51-28-5	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2,4-Dinitrotoluene	121-14-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2,6-Dinitrotoluene	606-20-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2-Chloronaphthalene	91-58-7	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2-Chlorophenol	95-57-8	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2-Methylnaphthalene	91-57-6	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2-Methylphenol	95-48-7	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2-Nitroaniline	88-74-4	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			2-Nitrophenol	88-75-5	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			3,3'-Dicholorobenzidine	91-94-1	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			3-Methylphenol	108-39-4	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			3-Nitroaniline	99-09-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			4,6-Dinitro-2-methylphenol	534-52-1	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			4-Bromophenyl-phenylether	101-55-3	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			4-Chloro-3-methylphenol	59-50-7	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			4-Chloroaniline	106-47-8	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			4-Chlorophenyl-phenyl ether	7005-72-3	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			4-Methylphenol	106-44-5	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			4-Nitroaniline	100-01-6	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			4-Nitrophenol	100-02-7	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Acenaphthene	83-32-9	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Acenaphthylene	208-96-8	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Acetophenone	98-86-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Anthracene	120-12-7	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Atrazine	1912-24-9	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Benzaldehyde	100-52-7	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Benzo(a) pyrene	50-32-8	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Benzo(a)anthracene	56-55-3	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Benzo(b) fluoranthene	205-99-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Benzo(g,h,i) perylene	191-24-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Benzo(k) fluoranthene	207-08-9	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Bis(2-chloroethoxy) methane	111-91-1	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Bis(2-chloroethyl) ether	111-44-4	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D

Table P1-1
Laboratory analyses and certifications for the SPF Demobilization Environmental Media Samples

Matrix	Category	Laboratory	Analyte Name	CAS Number	Analytical Method	Certification
Aqueous (Continued)	TCL Semivolatile Organic Compounds (Continued)	Pace Long Island	Bis(2-ethylhexyl) phthalate	117-81-7	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Butylbenzylphthalate	85-68-7	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Caprolactam	105-60-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Carbazole	86-74-8	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Chrysene	218-01-9	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Dibenzo(a,h) anthracene	53-70-3	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Dibenzofuran	132-64-9	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Diethylphthalate	84-66-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Dimethylphthalate	131-11-3	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Di-n-butylphthalate	84-74-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Di-n-octylphthalate	117-84-0	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Fluoranthene	206-44-0	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Fluorene	86-73-7	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Hexachlorobenzene	118-74-1	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Hexachlorobutadiene	87-68-3	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Hexachlorocyclopentadiene	77-47-4	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Hexachloroethane	67-72-1	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Indeno(1,2,3,-cd) pyrene	193-39-5	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Isophorone	78-59-1	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Naphthalene	91-20-3	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Nitrobenzene	98-95-3	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			N-Nitroso-di-n propylamine	621-64-7	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			N-Nitrosodiphenylamine	86-30-6	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Pentachlorophenol	87-86-5	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Phenanthrene	85-01-8	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Phenol	108-95-2	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
			Pyrene	129-00-0	SW-846 8270D	NYSDOH Non-Potable Water - SW-846 8270D
	Pesticides	Pace Long Island	4,4'-DDD	72-54-8	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			4,4'-DDE	72-55-9	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			4,4'-DDT	50-29-3	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			Aldrin	309-00-2	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			alpha-BHC	319-84-6	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			beta-BHC	319-85-7	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			cis-Chlordane	5103-71-9	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			delta-BHC	319-86-8	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			Dieldrin	60-57-1	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			Endosulfan I	959-98-8	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			Endosulfan II	33213-65-9	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			Endosulfan sulfate	1031-07-8	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			Endrin	72-20-8	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			Endrin aldehyde	7421-93-4	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B

Table P1-1
Laboratory analyses and certifications for the SPF Demobilization Environmental Media Samples

Matrix	Category	Laboratory	Analyte Name	CAS Number	Analytical Method	Certification
Aqueous (Continued)	Pesticides (Continued)	Pace Long Island	Endrin ketone	53494-70-5	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			gamma-BHC (Lindane)	58-89-9	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			Heptachlor	76-44-8	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			Heptachlor epoxide	1024-57-3	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			Methoxychlor	72-43-5	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			Toxaphene	8001-35-2	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
			trans-Chlordane	5103-74-2	SW-846 8081B	NYSDOH Non-Potable Water - SW-846 8081B
	PCBs (Aroclors)	Pace Schenectady	Aroclor 1016	12674-11-2	SW-846 8082A	NYSDOH Non-Potable Water - SW-846 8082A
			Aroclor 1221	11104-28-2	SW-846 8082A	NYSDOH Non-Potable Water - SW-846 8082A
			Aroclor 1232	11141-16-5	SW-846 8082A	NYSDOH Non-Potable Water - SW-846 8082A
			Aroclor 1242	53469-21-9	SW-846 8082A	NYSDOH Non-Potable Water - SW-846 8082A
			Aroclor 1248	12672-29-6	SW-846 8082A	NYSDOH Non-Potable Water - SW-846 8082A
			Aroclor 1254	11097-69-1	SW-846 8082A	NYSDOH Non-Potable Water - SW-846 8082A
			Aroclor 1260	11096-82-5	SW-846 8082A	NYSDOH Non-Potable Water - SW-846 8082A
			Total PCBs (sum of Aroclors)	1336-36-3	SW-846 8082A	No Certification Available
	TAL Metals (except Mercury)	Pace Long Island	Aluminum	7429-90-5	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Antimony	7440-36-0	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Arsenic	7440-38-2	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Barium	7440-39-3	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Beryllium	7440-41-7	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Cadmium	7440-43-9	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Calcium	7440-70-2	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Chromium	7440-47-3	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Cobalt	7440-48-4	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Copper	7440-50-8	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Iron	7439-89-6	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Lead	7439-92-1	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Magnesium	7439-95-4	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Manganese	7439-96-5	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Nickel	7440-02-0	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Potassium	7440-09-7	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Selenium	7782-49-2	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Silver	7440-22-4	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Sodium	7440-23-5	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Thallium	7440-28-0	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Vanadium	7440-62-2	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
			Zinc	7440-66-6	SW-846 6010C	NYSDOH Non-Potable Water - SW-846 6010C
	Mercury	Pace Long Island	Mercury	7439-97-6	SW-846 7470A	NYSDOH Non-Potable Water - SW-846 7470A

Table P1-1

Laboratory analyses and certifications for the SPF Demobilization Environmental Media Samples

Matrix	Category	Laboratory	Analyte Name	CAS Number	Analytical Method	Certification
Aqueous (Continued)	Oil and Grease	Pace Long Island	Oil and Grease	Q2240	EPA 1664A	NYSDOH Non-Potable Water - EPA 1664A

Notes:
Pace - Pace Analytical Services, Inc.

Table P1-2
Reference Limit and Evaluation for the SPF Demobilization Environmental Media Samples

Matrix	Category	Analyte Name	CAS number(s)	Analytical Method	Units	Laboratory Method Detection Limits[1]	Laboratory Reporting Limits[1]
Soil and Sediment	PCBs (Aroclors) - Microwave	Aroclor 1016	12674-11-2	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1221	11104-28-2	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1232	11141-16-5	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1242	53469-21-9	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1248	12672-29-6	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1254	11097-69-1	GEHR8082	mg/kg	0.019	0.050
		Aroclor 1260	11096-82-5	GEHR8082	mg/kg	0.019	0.050
		Total PCBs (sum of Aroclors)	1336-36-3	GEHR8082	mg/kg	0.019	0.20
	PCBs (Aroclors) - ASE	Aroclor 1016	12674-11-2	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1221	11104-28-2	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1232	11141-16-5	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1242	53469-21-9	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1248	12672-29-6	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1254	11097-69-1	GEHR8082	mg/kg	0.029	0.050
		Aroclor 1260	11096-82-5	GEHR8082	mg/kg	0.029	0.050
		Total PCBs (sum of Aroclors)	1336-36-3	GEHR8082	mg/kg	0.029	0.20
	Other	TOC	OC002	Lloyd Kahn	mg/Kg	196	400
		Percent Moisture	WC002	ASTM D2216-98	%	NA	0.020
	STARS Volatile Organic Compounds	1,2,4-Trimethylbenzene	95-63-6	SW-846 8260C	µg/kg	0.71	10
		1,3,5-Trimethylbenzene	108-67-8	SW-846 8260C	µg/kg	1.1	10
		1.4-Dioxane	123-91-1	SW-846 8260C	µg/kg	26.6	100
		Benzene	71-43-2	SW-846 8260C	µg/kg	0.50	10
		Ethylbenzene	100-41-4	SW-846 8260C	µg/kg	0.62	10
		Isopropylbenzene	98-82-8	SW-846 8260C	µg/kg	0.59	10
		Methyl-Tert-Butyl-Ether	1634-04-4	SW-846 8260C	µg/kg	0.46	10
		Naphthalene	91-20-3	SW-846 8260C	µg/kg	0.88	10
		n-Butylbenzene	104-51-8	SW-846 8260C	µg/kg	0.62	10
		n-Propylbenzene	103-65-1	SW-846 8260C	µg/kg	0.58	10
		p-Isopropyltoluene	99-87-6	SW-846 8260C	µg/kg	0.57	10
		sec-Butylbenzene	135-98-8	SW-846 8260C	µg/kg	0.64	10
		Tert-Butylbenzene	98-06-6	SW-846 8260C	µg/kg	0.59	10
		Toluene	108-88-3	SW-846 8260C	µg/kg	0.46	10
		Xylene (Mixed)	1330-20-7	SW-846 8260C	µg/kg	1.8	10
		1,1'-Biphenyl	92-52-4	SW-846 8270D	µg/kg		
		1,2,4,5-Tetrachlorobenzene	95-94-3	SW-846 8270D	µg/kg	9.5	330
		2,2'-Oxybis(1-choloropropane)	108-60-1	SW-846 8270D	µg/kg	5.8	330
		2,3,4,6-Tetrachlorophenol	58-90-2	SW-846 8270D	µg/kg	12.8	330
		2,4,5-Trichlorophenol	95-95-4	SW-846 8270D	µg/kg	14.2	330
		2,4,6-Trichlorophenol	88-06-2	SW-846 8270D	µg/kg	12.5	330
		2,4-Dichlorophenol	120-83-2	SW-846 8270D	µg/kg	8.5	330
		2,4-Dimethylphenol	105-67-9	SW-846 8270D	µg/kg	9.9	330
		2,4-Dinitrophenol	51-28-5	SW-846 8270D	µg/kg		
		2,4-Dinitrotoluene	121-14-2	SW-846 8270D	µg/kg	10.9	330
		2,6-Dinitrotoluene	606-20-2	SW-846 8270D	µg/kg	6.3	330
		2-Chloronaphthalene	91-58-7	SW-846 8270D	µg/kg	7.2	330
		2-Chlorophenol	95-57-8	SW-846 8270D	µg/kg	9.5	330
		2-Methylnaphthalene	91-57-6	SW-846 8270D	µg/kg	10.8	330
		2-Methylphenol	95-48-7	SW-846 8270D	µg/kg	9.1	330
		2-Nitroaniline	88-74-4	SW-846 8270D	µg/kg	8.1	330
		2-Nitrophenol	88-75-5	SW-846 8270D	µg/kg	6.0	330
		3,3'-Dicholorobenzidine	91-94-1	SW-846 8270D	µg/kg	12.9	330
		3-Methylphenol/4-Methylphenol	108-39-4/ 106-44-5	SW-846 8270D	µg/kg	10.6	330
		3-Nitroaniline	99-09-2	SW-846 8270D	µg/kg	10.6	330
		4,6-Dinitro-2-methylphenol	534-52-1	SW-846 8270D	µg/kg		
		4-Bromophenyl-phenylether	101-55-3	SW-846 8270D	µg/kg	10.9	330
		4-Chloro-3-methylphenol	59-50-7	SW-846 8270D	µg/kg	8.3	330
		4-Chloroaniline	106-47-8	SW-846 8270D	µg/kg	9.4	330
		4-Chlorophenyl-phenyl ether	7005-72-3	SW-846 8270D	µg/kg	9.8	330
		4-Nitroaniline	100-01-6	SW-846 8270D	µg/kg	9.4	330
		4-Nitrophenol	100-02-7	SW-846 8270D	µg/kg		
		Acenaphthene	83-32-9	SW-846 8270D	µg/kg	10.5	330
		Acenaphthylene	208-96-8	SW-846 8270D	µg/kg	5.9	330
		Acetophenone	98-86-2	SW-846 8270D	µg/kg	8.0	330
		Anthracene	120-12-7	SW-846 8270D	µg/kg	8.1	330
		Atrazine	1912-24-9	SW-846 8270D	µg/kg		
		Benzo(a) pyrene	50-32-8	SW-846 8270D	µg/kg	9.5	330
		Benzo(a)anthracene	56-55-3	SW-846 8270D	µg/kg	11.8	330
		Benzo(b) fluoranthene	205-99-2	SW-846 8270D	µg/kg	9.2	330
		Benzo(g,h,i) perylene	191-24-2	SW-846 8270D	µg/kg	11.2	330

Table P1-2
Reference Limit and Evaluation for the SPF Demobilization Environmental Media Samples

Matrix	Category	Analyte Name	CAS number(s)	Analytical Method	Units	Laboratory Method Detection Limits[1]	Laboratory Reporting Limits[1]
Soil and Sediment (Continued)	TCL Semivolatile Organic Compounds (Continued)	Benzo(k) fluoranthene	207-08-9	SW-846 8270D	µg/kg	14.0	330
		Bis(2-chloroethoxy) methane	111-91-1	SW-846 8270D	µg/kg	8.6	330
		Bis(2-chloroethyl) ether	111-44-4	SW-846 8270D	µg/kg	6.6	330
		Bis(2-ethylhexyl) phthalate	117-81-7	SW-846 8270D	µg/kg	11.9	330
		Butylbenzylphthalate	85-68-7	SW-846 8270D	µg/kg	9.6	330
		Caprolactam	105-60-2	SW-846 8270D	µg/kg	17.5	330
		Carbazole	86-74-8	SW-846 8270D	µg/kg		
		Chrysene	218-01-9	SW-846 8270D	µg/kg	10.5	330
		Dibenzo(a,h) anthracene	53-70-3	SW-846 8270D	µg/kg	12.0	330
		Dibenzofuran	132-64-9	SW-846 8270D	µg/kg	4.6	330
		Diethylphthalate	84-66-2	SW-846 8270D	µg/kg	5.5	330
		Dimethylphthalate	131-11-3	SW-846 8270D	µg/kg	7.5	330
		Di-n-butylphthalate	84-74-2	SW-846 8270D	µg/kg	7.8	330
		Di-n-octylphthalate	117-84-0	SW-846 8270D	µg/kg	6.3	330
		Fluoranthene	206-44-0	SW-846 8270D	µg/kg	7.5	330
		Fluorene	86-73-7	SW-846 8270D	µg/kg	11.5	330
		Hexachlorobenzene	118-74-1	SW-846 8270D	µg/kg	9.9	330
		Hexachlorobutadiene	87-68-3	SW-846 8270D	µg/kg	9.8	330
		Hexachlorocyclopentadiene	77-47-4	SW-846 8270D	µg/kg	7.9	330
		Hexachloroethane	67-72-1	SW-846 8270D	µg/kg	10.0	330
		Indeno(1,2,3,-cd) pyrene	193-39-5	SW-846 8270D	µg/kg	9.6	330
		Isophorone	78-59-1	SW-846 8270D	µg/kg	5.8	330
		Naphthalene	91-20-3	SW-846 8270D	µg/kg	10.7	330
		Nitrobenzene	98-95-3	SW-846 8270D	µg/kg	7.4	330
		N-Nitroso-di-n propylamine	621-64-7	SW-846 8270D	µg/kg	8.7	330
		N-Nitrosodiphenylamine	86-30-6	SW-846 8270D	µg/kg	8.9	330
		Pentachlorophenol	87-86-5	SW-846 8270D	µg/kg		
		Phenanthrene	85-01-8	SW-846 8270D	µg/kg	7.6	330
		Phenol	108-95-2	SW-846 8270D	µg/kg	6.7	330
		Pyrene	129-00-0	SW-846 8270D	µg/kg	8.9	330
	STARS Semivolatile Organic Compounds	Acenaphthylene	208-96-8	SW-846 8270D	µg/kg	5.9	330
		Anthracene	120-12-7	SW-846 8270D	µg/kg	8.1	330
		Benz(a)Anthracene	56-55-3	SW-846 8270D	µg/kg	11.8	330
		Benzo(a)Pyrene	50-32-8	SW-846 8270D	µg/kg	9.5	330
		Benzo(b)Fluoranthene	205-99-2	SW-846 8270D	µg/kg	9.2	330
		Benzo(g,h,i)Perylene	191-24-2	SW-846 8270D	µg/kg	11.2	330
		Benzo(k)Fluoranthene	207-08-9	SW-846 8270D	µg/kg	14.0	330
		Chrysene	218-01-9	SW-846 8270D	µg/kg	10.5	330
		Dibenzo(a,h)Anthracene	53-70-3	SW-846 8270D	µg/kg	12.0	330
		Fluoranthene	206-44-0	SW-846 8270D	µg/kg	7.5	330
		Fluorene	86-73-7	SW-846 8270D	µg/kg	11.5	330
		Indeno(1,2,3-cd)Pyrene	193-39-5	SW-846 8270D	µg/kg	9.6	330
		Phenanthrene	85-01-8	SW-846 8270D	µg/kg	7.6	330
		Pyrene	129-00-0	SW-846 8270D	µg/kg	8.9	330
	Gasoline Range Organics	Gasoline Range Organics		SW-846 8015D	µg/kg	19.9	100
	Diesel Range Organics	Diesel Range Organics		SW-846 8015D	mg/kg	0.65	7.0
Aqueous	TCL Volatile Organic Compounds	1,1,1-Trichloroethane	71-55-6	SW-846 8260C	µg/L	0.47	1.0
		1,1,2,2-Tetrachloroethane	79-34-5	SW-846 8260C	µg/L	0.43	1.0
		1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	SW-846 8260C	µg/L		
		1,1,2-Trichloroethane	79-00-5	SW-846 8260C	µg/L	0.37	1.0
		1,1-Dichloroethane	75-34-3	SW-846 8260C	µg/L	0.62	1.0
		1,1-Dichloroethene	75-35-4	SW-846 8260C	µg/L	0.49	1.0
		1,2,3-Trichlorobenzene	87-61-6	SW-846 8260C	µg/L	0.29	1.0
		1,2,4-Trichlorobenzene	120-82-1	SW-846 8260C	µg/L	0.32	1.0
		1,2-Dibromo-3-chloropropane	96-12-8	SW-846 8260C	µg/L		
		1,2-Dibromoethane	106-93-4	SW-846 8260C	µg/L		
		1,2-Dichlorobenzene	95-50-1	SW-846 8260C	µg/L	0.41	1.0
		1,2-Dichloroethane	107-06-2	SW-846 8260C	µg/L	0.61	1.0
		1,2-Dichloropropane	78-87-5	SW-846 8260C	µg/L	0.53	1.0
		1,3-Dichlorobenzene	541-73-1	SW-846 8260C	µg/L	0.37	1.0
		1,4-Dichlorobenzene	106-46-7	SW-846 8260C	µg/L	0.40	1.0
		1.4-Dioxane	123-91-1	SW-846 8260C	µg/L		
		2-Butanone	78-93-3	SW-846 8260C	µg/L	0.50	1.0
		2-Hexanone	591-78-6	SW-846 8260C	µg/L	0.61	1.0
		4-Methyl-2-pentanone	108-10-1	SW-846 8260C	µg/L	0.60	1.0
		Acetone	67-64-1	SW-846 8260C	µg/L	0.59	1.0
		Benzene	71-43-2	SW-846 8260C	µg/L	0.45	1.0
		Bromochloromethane	74-97-5	SW-846 8260C	µg/L		
		Bromodichloromethane	75-27-4	SW-846 8260C	µg/L	0.37	1.0

Table P1-2
Reference Limit and Evaluation for the SPF Demobilization Environmental Media Samples

Matrix	Category	Analyte Name	CAS number(s)	Analytical Method	Units	Laboratory Method Detection Limits[1]	Laboratory Reporting Limits[1]
Aqueous (Continued)	TCL Volatile Organic Compounds (Continued)	Bromoform	75-25-2	SW-846 8260C	µg/L	0.16	1.0
		Bromomethane	74-83-9	SW-846 8260C	µg/L	0.46	1.0
		Carbon disulfide	75-15-0	SW-846 8260C	µg/L	0.42	1.0
		Carbon tetrachloride	56-23-5	SW-846 8260C	µg/L	0.40	1.0
		Chlorobenzene	108-90-7	SW-846 8260C	µg/L	0.35	1.0
		Chloroethane	75-00-3	SW-846 8260C	µg/L	0.55	1.0
		Chloroform	67-66-3	SW-846 8260C	µg/L	0.53	1.0
		Chloromethane	74-87-3	SW-846 8260C	µg/L	0.81	1.0
		cis-1,2-Dichloroethene	156-59-2	SW-846 8260C	µg/L	0.50	1.0
		cis-1,3-Dichloropropene	10061-01-5	SW-846 8260C	µg/L	0.42	1.0
		Cyclohexane	110-82-7	SW-846 8260C	µg/L		
		Dibromochloromethane	124-48-1	SW-846 8260C	µg/L	0.25	1.0
		Dichlorodifluoromethane	75-71-8	SW-846 8260C	µg/L	0.39	1.0
		Ethylbenzene	100-41-4	SW-846 8260C	µg/L	0.42	1.0
		Isopropylbenzene	98-82-8	SW-846 8260C	µg/L	0.47	1.0
		m,p-Xylene	179601-23-1	SW-846 8260C	µg/L	0.88	1.0
		Methyl acetate	79-20-9	SW-846 8260C	µg/L		
		Methyl tert-butyl ether	1634-04-4	SW-846 8260C	µg/L	0.50	1.0
		Methylcyclohexane	108-87-2	SW-846 8260C	µg/L		
		Methylene chloride	75-09-2	SW-846 8260C	µg/L	0.69	1.0
		o-Xylene	95-47-6	SW-846 8260C	µg/L	0.40	1.0
		Styrene	100-42-5	SW-846 8260C	µg/L	0.32	1.0
		Tetrachloroethene	127-18-4	SW-846 8260C	µg/L	0.40	1.0
		Toluene	108-88-3	SW-846 8260C	µg/L	0.39	1.0
		trans-1,2-Dichloroethene	156-60-5	SW-846 8260C	µg/L	0.50	1.0
		trans-1,3-Dichloropropene	10061-02-6	SW-846 8260C	µg/L	0.39	1.0
		Trichloroethene	79-01-6	SW-846 8260C	µg/L	0.64	1.0
		Trichlorofluoromethane	75-69-4	SW-846 8260C	µg/L	0.44	1.0
		Vinyl chloride	75-01-4	SW-846 8260C	µg/L	0.54	1.0
	TCL Semivolatile Organic Compounds	1,1'-Biphenyl	92-52-4	SW-846 8270D	µg/L	0.36	1.0
		1,2,4,5-Tetrachlorobenzene	95-94-3	SW-846 8270D	µg/L	0.34	1.0
		2,2'-Oxybis(1-choloropropane)	108-60-1	SW-846 8270D	µg/L	0.52	1.0
		2,3,4,6-Tetrachlorophenol	58-90-2	SW-846 8270D	µg/L	0.18	1.0
		2,4,5-Trichlorophenol	95-95-4	SW-846 8270D	µg/L	0.23	1.0
		2,4,6-Trichlorophenol	88-06-2	SW-846 8270D	µg/L	0.23	1.0
		2,4-Dichlorophenol	120-83-2	SW-846 8270D	µg/L	0.30	1.0
		2,4-Dimethylphenol	105-67-9	SW-846 8270D	µg/L	0.76	5.0
		2,4-Dinitrophenol	51-28-5	SW-846 8270D	µg/L	0.49	5.0
		2,4-Dinitrotoluene	121-14-2	SW-846 8270D	µg/L	0.31	1.0
		2,6-Dinitrotoluene	606-20-2	SW-846 8270D	µg/L	0.30	1.0
		2-Chloronaphthalene	91-58-7	SW-846 8270D	µg/L	0.28	1.0
		2-Chlorophenol	95-57-8	SW-846 8270D	µg/L	0.29	1.0
		2-Methylnaphthalene	91-57-6	SW-846 8270D	µg/L	0.34	1.0
		2-Methylphenol	95-48-7	SW-846 8270D	µg/L	0.21	1.0
		2-Nitroaniline	88-74-4	SW-846 8270D	µg/L	0.38	1.0
		2-Nitrophenol	88-75-5	SW-846 8270D	µg/L	0.42	1.0
		3,3'-Dicholorobenzidine	91-94-1	SW-846 8270D	µg/L	0.27	1.0
		3-Methylphenol/4-Methylphenol	108-39-4/ 106-44-5	SW-846 8270D	µg/L	0.30	1.0
		3-Nitroaniline	99-09-2	SW-846 8270D	µg/L	0.32	1.0
		4,6-Dinitro-2-methylphenol	534-52-1	SW-846 8270D	µg/L	0.36	5.0
		4-Bromophenyl-phenylether	101-55-3	SW-846 8270D	µg/L	0.38	1.0
		4-Chloro-3-methylphenol	59-50-7	SW-846 8270D	µg/L	0.22	1.0
		4-Chloroaniline	106-47-8	SW-846 8270D	µg/L	0.35	1.0
		4-Chlorophenyl-phenyl ether	7005-72-3	SW-846 8270D	µg/L	0.39	1.0
		4-Nitroaniline	100-01-6	SW-846 8270D	µg/L	0.30	1.0
		4-Nitrophenol	100-02-7	SW-846 8270D	µg/L	0.45	5.0
		Acenaphthene	83-32-9	SW-846 8270D	µg/L	0.34	1.0
		Acenaphthylene	208-96-8	SW-846 8270D	µg/L	0.32	1.0
		Acetophenone	98-86-2	SW-846 8270D	µg/L	0.68	1.0
		Anthracene	120-12-7	SW-846 8270D	µg/L	0.28	1.0
		Atrazine	1912-24-9	SW-846 8270D	µg/L	0.37	1.0
		Benzaldehyde	100-52-7	SW-846 8270D	µg/L	0.73	1.0
		Benzo(a) pyrene	50-32-8	SW-846 8270D	µg/L	0.14	1.0
		Benzo(a)anthracene	56-55-3	SW-846 8270D	µg/L	0.14	1.0
		Benzo(b) fluoranthene	205-99-2	SW-846 8270D	µg/L	0.24	1.0
		Benzo(g,h,i) perylene	191-24-2	SW-846 8270D	µg/L	0.17	1.0
		Benzo(k) fluoranthene	207-08-9	SW-846 8270D	µg/L	0.15	1.0
		Bis(2-chloroethoxy) methane	111-91-1	SW-846 8270D	µg/L	0.49	1.0
		Bis(2-chloroethyl) ether	111-44-4	SW-846 8270D	µg/L	0.29	1.0
		Bis(2-ethylhexyl) phthalate	117-81-7	SW-846 8270D	µg/L	0.35	1.0

Table P1-2
Reference Limit and Evaluation for the SPF Demobilization Environmental Media Samples

Matrix	Category	Analyte Name	CAS number(s)	Analytical Method	Units	Laboratory Method Detection Limits[1]	Laboratory Reporting Limits[1]
Aqueous (Continued)	TCL Semivolatile Organic Compounds (Continued)	Butylbenzylphthalate	85-68-7	SW-846 8270D	µg/L	0.17	1.0
		Caprolactam	105-60-2	SW-846 8270D	µg/L	0.72	5.0
		Carbazole	86-74-8	SW-846 8270D	µg/L	0.18	1.0
		Chrysene	218-01-9	SW-846 8270D	µg/L	0.21	1.0
		Dibenzo(a,h) anthracene	53-70-3	SW-846 8270D	µg/L	0.16	1.0
		Dibenzofuran	132-64-9	SW-846 8270D	µg/L	0.34	1.0
		Diethylphthalate	84-66-2	SW-846 8270D	µg/L	0.27	1.0
		Dimethylphthalate	131-11-3	SW-846 8270D	µg/L	0.34	1.0
		Di-n-butylphthalate	84-74-2	SW-846 8270D	µg/L	0.12	1.0
		Di-n-octylphthalate	117-84-0	SW-846 8270D	µg/L	0.18	1.0
		Fluoranthene	206-44-0	SW-846 8270D	µg/L	0.13	1.0
		Fluorene	86-73-7	SW-846 8270D	µg/L	0.33	1.0
		Hexachlorobenzene	118-74-1	SW-846 8270D	µg/L	0.32	1.0
		Hexachlorobutadiene	87-68-3	SW-846 8270D	µg/L	0.33	1.0
		Hexachlorocyclopentadiene	77-47-4	SW-846 8270D	µg/L	0.20	1.0
		Hexachloroethane	67-72-1	SW-846 8270D	µg/L	0.39	1.0
		Indeno(1,2,3,-cd) pyrene	193-39-5	SW-846 8270D	µg/L	0.19	1.0
		Isophorone	78-59-1	SW-846 8270D	µg/L	0.38	1.0
		Naphthalene	91-20-3	SW-846 8270D	µg/L	0.30	1.0
		Nitrobenzene	98-95-3	SW-846 8270D	µg/L	0.30	1.0
		N-Nitroso-di-n propylamine	621-64-7	SW-846 8270D	µg/L	0.47	1.0
		N-Nitrosodiphenylamine	86-30-6	SW-846 8270D	µg/L	0.31	1.0
		Pentachlorophenol	87-86-5	SW-846 8270D	µg/L	0.56	5.0
		Phenanthrene	85-01-8	SW-846 8270D	µg/L	0.32	1.0
		Phenol	108-95-2	SW-846 8270D	µg/L	0.12	1.0
		Pyrene	129-00-0	SW-846 8270D	µg/L	0.18	1.0
	Pesticides	4,4'-DDD	72-54-8	SW-846 8081B	µg/L	0.012	0.10
		4,4'-DDE	72-55-9	SW-846 8081B	µg/L	0.0079	0.10
		4,4'-DDT	50-29-3	SW-846 8081B	µg/L	0.0094	0.10
		Aldrin	309-00-2	SW-846 8081B	µg/L	0.012	0.050
		alpha-BHC	319-84-6	SW-846 8081B	µg/L	0.0073	0.050
		beta-BHC	319-85-7	SW-846 8081B	µg/L	0.018	0.050
		cis-Chlordane	5103-71-9	SW-846 8081B	µg/L	0.011	0.050
		delta-BHC	319-86-8	SW-846 8081B	µg/L	0.013	0.050
		Dieldrin	60-57-1	SW-846 8081B	µg/L	0.0083	0.10
		Endosulfan I	959-98-8	SW-846 8081B	µg/L	0.0082	0.050
		Endosulfan II	33213-65-9	SW-846 8081B	µg/L	0.0094	0.10
		Endosulfan sulfate	1031-07-8	SW-846 8081B	µg/L	0.032	0.10
		Endrin	72-20-8	SW-846 8081B	µg/L	0.0082	0.10
		Endrin aldehyde	7421-93-4	SW-846 8081B	µg/L	0.019	0.10
		Endrin ketone	53494-70-5	SW-846 8081B	µg/L	0.0081	0.10
		gamma-BHC (Lindane)	58-89-9	SW-846 8081B	µg/L	0.0078	0.050
		Heptachlor	76-44-8	SW-846 8081B	µg/L	0.010	0.050
		Heptachlor epoxide	1024-57-3	SW-846 8081B	µg/L	0.0090	0.050
		Methoxychlor	72-43-5	SW-846 8081B	µg/L	0.021	0.050
		Toxaphene	8001-35-2	SW-846 8081B	µg/L	0.087	5.0
		trans-Chlordane	5103-74-2	SW-846 8081B	µg/L	0.016	0.050
	PCBs (Aroclors)	Aroclor 1016	12674-11-2	SW-846 8082A	µg/L	0.016	0.050
		Aroclor 1221	11104-28-2	SW-846 8082A	µg/L	0.016	0.050
		Aroclor 1232	11141-16-5	SW-846 8082A	µg/L	0.016	0.050
		Aroclor 1242	53469-21-9	SW-846 8082A	µg/L	0.016	0.050
		Aroclor 1248	12672-29-6	SW-846 8082A	µg/L	0.016	0.050
		Aroclor 1254	11097-69-1	SW-846 8082A	µg/L	0.016	0.050
		Aroclor 1260	11096-82-5	SW-846 8082A	µg/L	0.016	0.050
		Total PCBs (sum of Aroclors)	1336-36-3	SW-846 8082A	µg/L	0.016	0.050
	TAL Metals (except Mercury)	Aluminum	7429-90-5	SW-846 6010C	µg/L	13.3	200
		Antimony	7440-36-0	SW-846 6010C	µg/L	3.3	60
		Arsenic	7440-38-2	SW-846 6010C	µg/L	1.6	10
		Barium	7440-39-3	SW-846 6010C	µg/L	5.6	200
		Beryllium	7440-41-7	SW-846 6010C	µg/L	0.61	5.0
		Cadmium	7440-43-9	SW-846 6010C	µg/L	0.38	2.5
		Calcium	7440-70-2	SW-846 6010C	µg/L	37.0	1000
		Chromium	7440-47-3	SW-846 6010C	µg/L	0.53	10
		Cobalt	7440-48-4	SW-846 6010C	µg/L	1.7	50
		Copper	7440-50-8	SW-846 6010C	µg/L	1.0	25
		Iron	7439-89-6	SW-846 6010C	µg/L	12.5	100
		Lead	7439-92-1	SW-846 6010C	µg/L	1.1	5.0
		Magnesium	7439-95-4	SW-846 6010C	µg/L	58.9	1000
		Manganese	7439-96-5	SW-846 6010C	µg/L	0.70	15
		Nickel	7440-02-0	SW-846 6010C	µg/L	1.7	40
		Potassium	7440-09-7	SW-846 6010C	µg/L	332	5000

Table P1-2
Reference Limit and Evaluation for the SPF Demobilization Environmental Media Samples

Matrix	Category	Analyte Name	CAS number(s)	Analytical Method	Units	Laboratory Method Detection Limits[1]	Laboratory Reporting Limits[1]
Aqueous (Continued)	TAL Metals (except Mercury) (Continued)	Selenium	7782-49-2	SW-846 6010C	µg/L	2.4	10
		Silver	7440-22-4	SW-846 6010C	µg/L	0.57	10
		Sodium	7440-23-5	SW-846 6010C	µg/L	152	5000
		Thallium	7440-28-0	SW-846 6010C	µg/L	4.4	10
		Vanadium	7440-62-2	SW-846 6010C	µg/L	1.6	50
		Zinc	7440-66-6	SW-846 6010C	µg/L	1.1	20
	Mercury	Mercury	7439-97-6	SW-846 7470A	µg/L	0.023	0.20
	Oil and Grease	Oil and Grease	Q2240	EPA 1664A	mg/L	0.95	5.0

Notes

[1] The MDLs and RLs will be adjusted for sample specific factors such as % solids, weights/volumes and dilutions that vary from the standard procedure. Sample-specific MDLs and RLs are highly matrix dependent. Data will be evaluated against sample-specific MDLs and RLs. Non-detects, or values detected at a level below the sample specific MDL, will be reported as the sample specific MDL and U flagged (with the exception of analytes where MDL is NA). Values detected above the sample-specific MDL and below the sample-specific RL will be reported and flagged as estimated ("J").

NA - Not Applicable. Method detection limit (MDL) reporting will not be used for this anlayte. The analyte will be reported to the Reporting Limit (RL).

Table P2-1a
Measurement Performance Criteria for the SPF Demobilization Soil and Sediment Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory or Equipment Blank	S&A
	50-150 %R	Laboratory Control Sample (LCS) (spiked with Aroclor 1242)	A
	50-150 %R	Matrix Spike (MS) and Matrix Spike Duplicate (MSD) (spiked with Aroclor 1242)	S&A
	60-140 %R	Surrogates (TCMX and DCB)	A
Precision	The RPD for field duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	RPD should be ≤ 40%	MS/MSD	S&A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Soil and Sediment
Analytical Parameter: Total PCBs as Aroclors
Concentration Level: Low to High
Method: GEHR8082 (Phase 2 RAM QAPP Revised Attachment A Appendix A4-1)

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] *Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability)*

Table P2-1b
Measurement Performance Criteria for the SPF Demobilization Soil and Sediment Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Precision	The RPD for field duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	The RPD for lab duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Laboratory Duplicates	A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Soil and Sediment
Analytical Parameter: Percent Moisture
Concentration Level: Low to High
Method: Per Extraction SOPs (Phase 2 RAM QAPP Revised Attachment A Appendices A4-3 through A4-5)

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] *Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability)*

Table P2-1c
Measurement Performance Criteria for the SPF Demobilization Soil and Sediment Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory Blank	S&A
	75-125 %R	Matrix Spike	S&A
	33.1-167%R	Laboratory Control Sample	A
Precision	The RPD for field duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	The RPD for lab duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Laboratory Duplicates	A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Field audits and laboratory audits. See QAPP Section 10.3.4	S&A
Completeness	95%	See QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Sediment
Analytical Parameter: TOC
Concentration Level: Low to High
Method: Lloyd Kahn (Phase 2 RAM QAPP Attachment A Appendix A4-2)

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] *Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability)*

Table P2-1d
Measurement Performance Criteria for the SPF Demobilization Soil and Sediment Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory, Trip, or Equipment Blank	S&A
	Laboratory-specified limits	Laboratory Control Sample (LCS)	A
	Laboratory-specified limits	Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	S&A
	Laboratory-specified limits	Surrogates	A
Precision	The RPD for field duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	RPD should be ≤ 40%	MS/MSD	S&A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Soil
Analytical Parameter: Volatile Organic Compounds
Concentration Level: Low to High
Method: SW-846 8260C

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] *Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability)*

Table P2-1e
Measurement Performance Criteria for the SPF Demobilization Soil and Sediment Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory or Equipment Blank	S&A
	Laboratory-specified limits	Laboratory Control Sample (LCS)	A
	Laboratory-specified limits	Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	S&A
	Laboratory-specified limits	Surrogates	A
Precision	The RPD for field duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	RPD should be ≤ 40%	MS/MSD	S&A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Soil
Analytical Parameter: Semivolatile Organic Compounds
Concentration Level: Low to High
Method: SW-846 8270D

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] *Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability)*

Table P2-1f
Measurement Performance Criteria for the SPF Demobilization Soil and Sediment Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory, Trip, or Equipment Blank	S&A
	Laboratory-specified limits	Laboratory Control Sample (LCS)	A
	Laboratory-specified limits	Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	S&A
	Laboratory-specified limits	Surrogates	A
Precision	The RPD for field duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	RPD should be ≤ 40%	MS/MSD	S&A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Soil
Analytical Parameter: Gasoline Range Organics
Concentration Level: Low to High
Method: SW-846 8015D

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] *Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability)*

Table P2-1g
Measurement Performance Criteria for the SPF Demobilization Soil and Sediment Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory or Equipment Blank	S&A
	Laboratory-specified limits	Laboratory Control Sample (LCS)	A
	Laboratory-specified limits	Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	S&A
	Laboratory-specified limits	Surrogates	A
Precision	The RPD for field duplicates should be ≤40% for results >5× the RL. The difference between results should be ≤ 2× the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	RPD should be ≤ 40%	MS/MSD	S&A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Soil
Analytical Parameter: Diesel Range Organics
Concentration Level: Low to High
Method: SW-846 8015D

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] *Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability)*

Table P3-1a
Measurement Performance Criteria for the SPF Demobilization Aqueous Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory or Equipment Blank	S&A
	Laboratory-specified limits	Laboratory Control Sample (LCS)	A
	Laboratory-specified limits	Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	S&A
	Laboratory-specified limits	Surrogates	A
Precision	The RPD for field duplicates should be ≤20% for results >5× the RL. The difference between results should be ≤ the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	RPD should be ≤ 20%	MS/MSD	S&A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Water
Analytical Parameter: Total PCBs as Aroclors
Concentration Level: Low to High
Method: SW-846 8082A

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] *Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability).*

Table P3-1b
Measurement Performance Criteria for the SPF Demobilization Aqueous Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory, Trip, or Equipment Blank	S&A
	Laboratory-specified limits	Laboratory Control Sample (LCS)	A
	Laboratory-specified limits	Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	S&A
	Laboratory-specified limits	Surrogates	A
Precision	The RPD for field duplicates should be ≤20% for results >5× the RL. The difference between results should be ≤ the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	RPD should be ≤ 20%	MS/MSD	S&A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Water
Analytical Parameter: Volatile Organic Compounds
Concentration Level: Low to High
Method: SW-846 8260C

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability).

Table P3-1c
Measurement Performance Criteria for the SPF Demobilization Aqueous Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory or Equipment Blank	S&A
	Laboratory-specified limits	Laboratory Control Sample (LCS)	A
	Laboratory-specified limits	Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	S&A
	Laboratory-specified limits	Surrogates	A
Precision	The RPD for field duplicates should be ≤20% for results >5× the RL. The difference between results should be ≤ the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	RPD should be ≤ 20%	MS/MSD	S&A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Water
Analytical Parameter: Semivolatile Organic Compounds
Concentration Level: Low to High
Method: SW-846 8270D

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] *Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability).*

Table P3-1d
Measurement Performance Criteria for the SPF Demobilization Aqueous Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory or Equipment Blank	S&A
	Laboratory-specified limits	Laboratory Control Sample (LCS)	A
	Laboratory-specified limits	Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	S&A
	Laboratory-specified limits	Surrogates	A
Precision	The RPD for field duplicates should be ≤20% for results >5× the RL. The difference between results should be ≤ the RL when at least one result is ≤5× the RL	Field Duplicates	S&A
	RPD should be ≤ 20%	MS/MSD	S&A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes:

Matrix: Water
Analytical Parameter: Semivolatile Organic Compounds
Concentration Level: Low to High
Method: SW-846 8081B

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1] Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability).

Table P3-1e
Measurement Performance Criteria for the SPF Demobilization Aqueous Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory or Equipment Blank	S&A
	85-115 %R	Laboratory Control Sample	A
	75-125 %R (NA if sample concentration is >4× spike added)	Matrix Spike	S&A
Precision	The RPD for water lab duplicates should be ≤20% for results >5x the RL. The difference between results should be ≤ the RL when at least one result is ≤5x the RL	Laboratory Duplicates	A
	The RPD for water field duplicates should be ≤20% for results >5x the RL. The difference between results should be ≤ the RL when at least one result is ≤5x the RL	Field Duplicates	S&A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Representativeness	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes

Matrix: Water
Analytical Parameter: Metals (except Mercury and Hexavalent Chromium)
Concentration Level: Low to High
Method: SW-846 6010C

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1]Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability).

Table P3-1f
Measurement Performance Criteria for the SPF Demobilization Aqueous Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory or Equipment Blank	S&A
	85-115 %R	Laboratory Control Sample	A
	75-125 %R (NA if sample concentration is >4× spike added)	Matrix Spike	S&A
Precision	The RPD for water lab duplicates should be ≤20% for results >5x the RL. The difference between results should be ≤ the RL when at least one result is ≤5x the RL	Laboratory Duplicates	A
	The RPD for water field duplicates should be ≤20% for results >5x the RL. The difference between results should be ≤ the RL when at least one result is ≤5x the RL	Field Duplicates	S&A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Representativeness	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes

Matrix: Water
Analytical Parameter: Mercury
Concentration Level: Low to High
Method: SW-846 7470A

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1]Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability).

Table P3-1g
Measurement Performance Criteria for the SPF Demobilization Aqueous Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or activity used to assess measurement performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory Blank	A
	Laboratory-specified limits	Laboratory Control Sample	A
	Laboratory-specified limits	Matrix Spike/Matrix Spike Duplicate	A
Precision	Laboratory-specified limits	Matrix Spike/Matrix Spike Duplicate	A
Sensitivity	See Table P1-2	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Internal field audits and laboratory audits. See Phase 2 RAM QAPP Sections 11.1.1 and 11.2.2	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes

Matrix: Water
Analytical Parameter: Oil and Grease
Concentration Level: Low to High
Method: USEPA 1664A

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1]Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability).

APPENDIX P1-1
SOP FOR DATA PACKAGE DELIVERABLES (DPDSOP)

OBJECTIVE

This Standard Operating Procedure (SOP) describes the requirements for the data packages that will be generated as part of the Sediment Process Facility (SPF) Demobilization. This SOP applies to the contractor(s) involved in analytical data generation and reporting. All data packages generated for the SPF Demobilization must be provided in an Adobe Acrobat (.pdf) file format.

SECTION A-9 DATA PACKAGE DELIVERABLES

The following sections describe in detail the types of data packages designed for the SPF Demobilization. These details are provided to allow several participating laboratories to produce data packages that are similar in format, order of presentation, and content. The data packages detailed in Section A-9.1 have been developed based on deliverables specified in the US EPA Contract Laboratory Program Statement of Work (CLP SOW). The CLP SOW has additional details concerning data packages that are specific to the CLP analyses. The most recent Statement of Work should be referenced for details concerning CLP-style data packages. Note: the summary forms provided in these data packages should be in similar format and content to the Contract Laboratory Program (CLP) forms listed (as references) next to the form title. These CLP forms references are only provided as guidance on content and format and should be modified by the laboratory to meet specific method requirements. Section A-9.2 provides details concerning specific contents of the data deliverables described in Section A-9.1.

The data package deliverables are as follows:

Level B - Fully documented data package.

The Level B package resembles the information required by the CLP SOW. This type of package includes a cover letter, sample delivery group (SDG) narrative, field Chain-of-Custody Records, analytical results summaries, a glossary of qualifier codes and summary forms for quality control procedures and all sample and quality control raw data to support the results reported.

A-9.1 Data Package Contents and Order of Presentation

The laboratory will be required to submit supporting documentation for the reported analytical results. The supporting documentation and the analytical results will be reported in a Level B, fully documented data package, as defined below. The laboratory need not include the documentation for any fraction not required for an SDG.

A-9.1.1 General Format for Level B Deliverables

The Level B Sample Data Package shall include data for analyses of all samples in one SDG, including field samples (including all field quality control [QC] samples), reanalyses, secondary dilutions, blanks, laboratory control samples, matrix spikes, matrix spike duplicates, and/or laboratory duplicates. The complete Sample Data Package is divided into the units as described below. Units for each analytical fraction have been detailed. If the analysis of that fraction was not required for samples in the SDG, the fraction-specific unit is not a required deliverable. The Sample Data Package must be complete before submission and must be consecutively paginated. The Sample Data Package should generally be arranged in the following order:

A) Cover Letter/Letter of Transmittal signed by the laboratory manager.

B) Title Page

C) Table of Contents

D) SDG Narrative

This document should be clearly labeled "SDG Narrative" and should contain: laboratory name; SDG number; GE sample identifications; laboratory sample numbers; and detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing (preparing and analyzing) the samples reported in the data package. A glossary of qualifier codes used in the SDG must also be provided.

The laboratory must also include any technical and administrative problems encountered, corrective actions taken and method of resolution, and an explanation of all flagged edits (i.e., exhibit edits) on quantitation reports.

Additionally, the SDG Narrative must be signed and dated by the laboratory manager.

E) Field Chain-of-Custody Records and Sample Receipt Documentation Log

Copies of field Chain-of-Custody Records for all samples within the SDG must be included in the deliverables. A description of the condition and temperature of the

samples upon laboratory receipt (*i.e.*, custody seal condition, container status) must be provided for each Chain-of-Custody Record/sample cooler.

F) GC/MS Volatile Organic Data.

1. Quality Control (QC) Summary.

a. Surrogate Percent Recovery Summary (modified CLP SOM01.2 Form II VOA).

b. Matrix Spike/Matrix Spike Duplicate Summary (modified CLP SOM01.2 Form III VOA).

c. Laboratory Control Sample Summary (modified CLP SOM01.2 Form III VOA).

d. Method Blank Summary (modified CLP SOM01.2 Form IV VOA) -- arranged in chronological order by date of analysis of the blank, by instrument.

e. GC/MS Tuning and Mass Calibration Summary (modified CLP SOM01.2 Form V VOA) -- arranged in chronological order, by instrument.

f. Internal Standard Area and Retention Time Summary (modified CLP SOM01.2 Form VIII VOA) -- arranged in chronological order, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for volatile samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Target Compound Results (modified CLP SOM01.2 Form I VOA).

b. Reconstructed total ion chromatogram (RIC) and quantitation reports.

c. Copies of raw spectra and copies of background-subtracted mass spectra of each target compound identified in the sample and corresponding background-subtracted target compound standard mass spectra.

d. Exhibit work sheet (including example calculations showing how sample results are calculated using the initial calibration and sample responses for at least one sample).

3. Standards Data

a. Initial Calibration Data (modified CLP SOM01.2 Form VI VOA and associated volatile standard RICs and quantitation reports) -- for all initial calibrations associated with analyses in the SDG, in

chronological order, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

b. Continuing Calibration Data (modified CLP SOM01.2 Form VII VOA and associated volatile standard RICs and quantitation reports) -- for all continuing calibrations associated with analyses in the SDG, in chronological order, by instrument.

4. Raw QC Data

a. For each GC/MS tuning and mass calibration (in chronological order, by instrument):

i. Bromofluorobenzene (BFB) bar graph spectrum.

ii. BFB mass listing.

b. Method/Storage Blank Data - in chronological order, by instrument:

i. Target Compound Results (modified CLP SOM01.2 Form I VOA).

ii. RIC and quantitation reports.

iii. Copies of raw spectra and copies of background-subtracted mass spectra of each target compounds identified in the blank

and corresponding background-subtracted target compound standard mass spectra.

c. Laboratory Control Sample Data:

i. Target Compound Results (modified CLP SOM01.2 Form I VOA).

ii. RIC and quantitation reports.

d. Matrix Spike Data:

i. Target Compound Results (modified CLP SOM01.2 Form I VOA).

ii. RIC and quantitation reports.

e. Matrix Spike Duplicate Data:

i. Target Compound Results (modified CLP SOM01.2 Form I VOA).

ii. RIC and quantitation reports.

G) GC/MS Semivolatile Organic Data

1. QC Summary

a. Surrogate Percent Recovery Summary (modified CLP SOM01.2 Form II SV).

b. Matrix Spike/Matrix Spike Duplicate Summary (modified CLP SOM01.2 Form III SV).

c. Laboratory Control Sample Summary (modified CLP SOM01.2 Form III SV).

d. Method Blank Summary (modified CLP SOM01.2 Form IV SV) -- arranged in chronological order by date of analysis of the blank, by instrument.

e. GC/MS Tuning and Mass Calibration Summary (modified CLP SOM01.2 Form V SV) -- arranged in chronological order, by instrument.

f. Internal Standard Area and Retention Time Summary (modified CLP SOM01.2 Form VIII SV) -- arranged in chronological order, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries, followed by the raw data for semivolatile samples. These

sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Target Compound Results (modified CLP SOM01.2 Form I).

b. RIC and quantitation report.

c. Copies of raw spectra and copies of background-subtracted mass spectra of each target compound identified in the sample and corresponding background-subtracted target compound standard mass spectra.

d. UV traces from Gel Permeation Chromatography (GPC) chromatograms cleanup (if performed).

e. Exhibit work sheet (including example calculations showing how sample results are calculated using the initial calibration and sample responses for at least one sample).

3. Standards Data

a. Initial Calibration Data (modified CLP SOM01.2 Form VI SV and associated semivolatile standard RICs and quantitation reports) -- for all initial calibrations associated with analyses in the SDG, in chronological order, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

b. Continuing Calibration Data (modified CLP SOM01.2 Form VII SV and associated semivolatile standard RICs and quantitation reports) -- for all continuing calibrations associated with analyses in the SDG, in chronological order, by instrument.

4. Raw QC Data

a. For each GC/MS tuning and mass calibration (in chronological order, by instrument):

i. Decafluorotriphenylphosphine (DFTPP) bar graph spectrum.

ii. DFTPP mass listing.

b. Blank Data -- in chronological order, by instrument:

i. Target Compound Results (modified CLP SOM01.2 Form I SV).

ii. RIC and quantitation reports.

iii. Copies of raw spectra and copies of background-subtracted mass spectra of each target compounds identified in the blank and corresponding background-subtracted target compound standard mass spectra.

c. Laboratory Control Sample Data:

i. Target Compound Results (modified CLP SOM01.2 Form I SV).

ii. RIC and quantitation reports.

d. Matrix Spike Data:

i. Target Compound Results (modified CLP SOM01.2 Form I SV).

ii. RIC and quantitation reports.

e. Matrix Spike Duplicate Data

i. Target Compound Results (modified CLP SOM01.2 Form I SV).

ii. RIC and quantitation reports.

H) GC Organochlorine Pesticide Data

1. QC Summary

a. Surrogate Percent Recovery Summary (modified CLP SOM01.2 Form II PEST).

b. Matrix Spike/Matrix Spike Duplicate Summary (modified CLP SOM01.2 Form III PEST).

c. Laboratory Control Sample Summary (modified CLP SOM01.2 Form III PEST).

d. Method Blank Summary (modified CLP SOM01.2 Form IV PEST) -- arranged in chronological order by date of analysis of the blank, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for organochlorine pesticide samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary (modified CLP SOM01.2 Form I PEST).

b. Copies of organochlorine pesticide chromatograms.

c. Copies of organochlorine pesticide chromatograms from second GC column confirmation (if performed).

d. GC integration reports or data system printouts.

e. Exhibit work sheet (including example calculation showing how sample results are calculated using initial calibration standard and sample responses for at least one sample).

f. UV traces from GPC cleanup (if performed).

g. If organochlorine pesticides are confirmed by GC/MS, the laboratory must submit copies of raw spectra and copies of background-subtracted mass spectra of target compounds that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. For multi-component pesticides confirmed by GC/MS, the laboratory will submit mass spectra of three major peaks of multi-component compounds from samples and standards.

3. Standards Data

a. Analytical Sequence Form -- in chronological order, by GC column, by instrument for all samples and quality control analyses.

b. Initial Calibration Data (Initial Calibration Summary Form [inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.], organochlorine pesticide standard chromatograms, and integration reports) -- for each initial calibration associated with SDG in chronological order, by GC column, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

c. Continuing Calibration Data (Continuing Calibration Summary Form [inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.], organochlorine pesticide standard chromatograms, and integration reports) -- for each continuing calibration associated with SDG in chronological order, by GC column, by instrument following the associated initial calibrations.

d. 4,4′-DDT and Endrin Breakdown Data (Percent Breakdown Summary Form, organochlorine pesticide chromatograms and integration reports) -- for each standard associated with SDG in chronological order by GC column, by instrument.

4. Raw QC Data

a. Blank Data -- in chronological order, by instrument:

i. Target Compound Results (modified CLP SOM01.2 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

b. Laboratory Control Sample Data:

i. Target Compound Results (modified CLP SOM01.2 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

c. Matrix Spike Data:

i. Target Compound Results (modified CLP SOM01.2 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

d. Matrix Spike Duplicate Data:

i. Target Compound Results (modified CLP SOM01.2 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

e. UV traces from GPC cleanup (if performed).

i. UV traces for the initial calibration standards and blanks. Compound names shall be written over the peaks or printed over the peaks, or retention times shall be written over the peaks, and a separate table listing compounds and retention times shall be provided.

ii. Chromatographs and data system reports for all standards used to quantify compounds in the GPC blanks.

iii. Chromatographs and data system reports for the GPC calibration check solution and all standards used to quantify compounds in the GPC calibration check solution.

f. Raw Florisil® data, arranged in chronological order.

i. Chromatographs and data system reports for the analysis of the Florisil® cartridge performance check.

ii. Chromatographs and data system reports for the standards used to quantify compounds in the Florisil® cartridge performance check analysis (*i.e.*, INDA, INDB, and the 2,4,5-trichlorophenol standards).

I) GC Polychlorinated Biphenyl (PCB) Data

1. QC Summary

a. Surrogate Percent Recovery Summary (modified CLP SOM01.2 SOW Form II ARO).

b. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Summary (modified CLP SOM01.2 SOW Form III ARO).

c. Duplicate Summary (modified CLP SOM01.2 SOW Form III ARO).

d. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) Summary (modified CLP SOM01.2 SOW Form III ARO).

e. Method Blank Summary (modified CLP SOM01.2 SOW Form IV ARO) -- arranged in chronological order by date of analysis of the blank, by instrument.

f. Internal Standard Summary (inclusive of sample/standard identification, area counts, recovery, control limits, etc.)

2. Sample Data

Sample data (including all field QC samples) shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for PCB samples. These sample packets should then be placed in increasing alphanumeric order by sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary (modified CLP SOM01.2 SOW Form I ARO).

b. Copies of PCB chromatograms.

c. Copies of PCB chromatograms from second GC column confirmation (if performed).

d. GC integration reports or data system printouts. The integration reports or data system printouts must include all peaks not just the peaks corresponding to the target analytes.

e. Exhibit work sheets (including example calibration showing how sample results are calculated using initial calibration and sample responses for at least one sample).

f. UV traces from GPC (if performed).

g. If PCBs are confirmed by GC/MS, then the laboratory must submit copies of raw spectra and background-subtracted mass spectra of target compounds that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. The laboratory will submit mass spectra of three major peaks of multi-component compounds from samples and standards for each PCB result confirmed by GC/MS.

3. Standards Data

a. Analytical Sequence Form -- in chronological order, by GC column, by instrument for all samples and quality control analyses.

b. Initial Calibration Data -- Initial Calibration Summary Form (inclusive of retention time windows, calibration factors or relative response factors, %RSDs, equations, correlation coefficients, etc.), PCB standard chromatograms, and integration reports for each initial calibration associated with SDG in chronological order, by GC column, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

c. Continuing Calibration Data -- Continuing Calibration Summary Form (inclusive of retention time windows, calibration factors or relative response factors, %Ds, etc.), PCB standard chromatograms, and integration reports for each continuing calibration associated with SDG in chronological order, by GC column, by instrument following the associated initial calibration.

4. Raw QC data

a. Blank Data -- in chronological order, by instrument:

i. Target Compound Results (modified CLP SOM01.2 SOW Form I ARO).

ii. PCB chromatograms and integration reports.

b. LCS/LCSD Data:

i. Target Compound Results (modified CLP SOM01.2 SOW Form I ARO).

ii. PCB chromatograms and integration reports.

c. Matrix Spike Data:

i. Target Compound Results (modified CLP SOM01.2 SOW Form I ARO).

ii. PCB chromatograms and integration reports.

d. Matrix Spike Duplicate Data:

i. Target Compound Results (modified CLP SOM01.2 SOW Form I ARO).

ii. PCB chromatograms and integration reports.

e. Duplicate Sample Data:

i. Target Compound Results (modified CLP SOM01.2 SOW Form I ARO).

ii. PCB chromatograms and integration reports.

f. UV traces from GPC cleanup (if performed).

i. UV traces for the initial calibration standards and blanks. Compound names shall be written or printed over the peaks, or retention times shall be written over the peaks, and a separate table listing compounds and retention times shall be provided.

ii. Chromatographs and data system reports for all standards used to quantify compounds in the GPC blanks.

iii. Chromatographs and data system reports for the GPC calibration check solution and all standards used to quantify compounds in the GPC calibration check solution (or used to assess the Aroclor pattern).

J) Inorganic Data for ICP or ICP/MS and/or mercury (CVAA or CVAFS)

1. Cover Page for the Inorganic Analyses Data Package.

2. Sample Results Summaries (modified CLP ILM05.3 SOW Form 1-INs) -- for all samples in the SDG, arranged in increasing alphanumeric order by sample identification.

3. Quality Control and Quarterly/Annual Verification of Instrument Parameters Summaries:

a. Initial Calibration Summary Form for multi-point calibrations, if performed (inclusive of calibration factors, %RSDs, equations, correlation coefficients, etc.)

b. Initial and Continuing Calibration Verification summaries (modified CLP ILM05.3 SOW Form 2A-INs).

c. Reporting Limit Standards summaries (if performed, modified CLP ILM05.3 SOW Form 2B-INs).

d. Blanks summaries (modified CLP ILM05.3 SOW Form 3-INs).

e. ICP or ICP/MS Interference Check Sample summaries (modified CLP ILM05.3 SOW Form 4A-INs or 4B-INs).

f. Matrix Spike/Matrix Spike Duplicate Sample Recovery summary (modified CLP ILM05.3 SOW Form 5A-IN).

g. Post-Digest Spike Sample Recovery forms (if performed, modified CLP ILM05.3 SOW Form 5B-IN).

h. Duplicates summary (modified CLP ILM05.3 SOW Form 6-IN).

i. Laboratory Control Sample summary (modified CLP ILM05.3 SOW Form 7-IN)

j. ICP or ICP/MS Serial Dilution summary (modified CLP ILM05.3 SOW Form 8-IN).

k. Method Detection Limits (MDL) and Reporting Limits (modified CLP ILM05.3 SOW Form 9-IN).

l. ICP Interelement Correction Factors (if performed, modified CLP ILM05.3 SOW Form 10A-IN and 10B-IN).

m. ICP or ICP/MS Linear Ranges (if performed, modified CLP ILM05.3 SOW Form 11-INs).

n. Preparation Logs (modified CLP ILM05.3 SOW Form 12-INs).

o. Analytical Run Logs (modified CLP ILM05.3 SOW Form 13-INs).

p. ICP/MS Tuning and Response Factor Criteria (CLP ILM05.3 SOW Form 14-IN).

q. ICP/MS Internal Standards Summary (CLP ILM05.3 SOW Form 15-IN).

4. Raw Data

For each reported value, the contracted laboratories will provide all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, and all sample analysis results. This statement

does not apply to the Quarterly/Annual Verifications Parameters submitted as part of each data package (Section A-9.1.1.G, items 3k-3m). Raw data must contain all instrument readouts used for the sample results. Each exposure or instrumental reading must be provided, including those readouts that may fall below the MDL. All instruments must provide a legible hard copy of the direct real-time instrument readout (*i.e.,* strip charts, printer tapes, etc.). A photocopy of the instrument's direct sequential readout must be included. A hard copy of the instrument's direct instrument readout for cyanide must be included if the instrumentation has the capability.

The order of raw data in the data package shall be ICP-AES, ICP/MS, and mercury.

K) General Chemistry/Conventionals Data

The general chemistry data will be arranged in the following order by individual parameter requested for the samples in the SDG.

1. Analytical Results Summaries -- for all samples in the SDG, arranged in increasing alphanumeric order by GE sample identification.

2. Quality Control Summaries

   a. Initial and Continuing Calibration Verification summaries.

   b. Blanks summaries.

c. Spike Sample/Spike Duplicate Recovery summary.

d. Duplicates summary.

e. Laboratory Control Sample summary.

f. Analytical Run Logs for instrumental analyses.

g. Preparation Logs.

3. Raw Data

For each reported value, the contracted laboratories will provide all raw data (instrument printouts or logbook pages) used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. Raw data must contain all instrument readouts/logbooks pages used for the sample results. Each exposure or instrumental reading must be provided, including those readouts/logbook pages that may fall below the quantitation limit. A photocopy of the instrument's direct sequential readout must be included if the instrumentation has the capability.

L) Preparation Logs

1. Volatile Preparation Logs.

2. Semivolatile/ Pesticide/ PCB Extraction Logs (by fraction).

2. Metals Digestion Logs.

3. General Chemistry Preparation Logs (by parameter).

A-9.2 Deliverables Reporting Requirements for GC/MS Volatile and Semivolatile Organic Analyses

The laboratory will be required to submit the following information as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (*i.e.*, recovery ranges, relative percent difference limits, *etc.*) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any recoveries that are outside of the acceptance criteria. The raw data associated with the samples, blanks, and standards must clearly identify the GE sample identifier, the laboratory sample number, the instrument, the laboratory file number for the analysis, and the peak areas/heights and retention times that correspond to the compounds of interest observed in all analyses reported. If the requirement of a summary form is not applicable to a particular sample, standard, or blank, the requirement should still appear on the form; however, no entry will be necessary on the form for that sample, standard, or blank.

A) 1. An analysis summary of the results for all target compounds for all sample analyses, matrix spike analyses, matrix spike duplicate analyses, laboratory control sample analyses, and method/storage blank analyses must be supplied. The summary must include an entry for each target compound, date(s) and time(s) of analysis, GE sample identification, laboratory sample number, date of sample collection, sample matrix, sample weight, sample

percent solids, heated or unheated purge, column type(s), column internal diameter, dilution factor, solid extract volume, solid aliquot volume, concentration units, and sample results. For semivolatile analyses, date of sample extraction, final extract volume, injection volume, and an indication of whether the GPC cleanup was performed (yes/no) is also required. If positive results below the lowest calibration standard are reported, they must be flagged as estimated ("J") on the analysis summary. "Not-detected" results will be represented by the GE required quantitation limit and a "U" flag. If a compound was detected in a sample as well as in the method blank associated with the sample, the result must be flagged with a "B" on the summary form. Additionally, if a dilution is performed on a sample because a target compound is above the calibration range, then the positive result for the particular compound should be flagged with a "D." If the compound is still above the calibration rage after a dilution is performed on the sample, then the compound should be flagged with an "E."

2. The raw data for the sample analyses, method blank analyses, and storage blank analyses by GC/MS methodologies will include the RICs, mass spectra for all target compounds identified, and quantitation reports for the target compounds and surrogates. The raw data for the matrix spike and matrix spike duplicate analyses will include the RIC and quantitation report for the target compounds. These are required only for Level B Deliverables.

B) A surrogate percent recoveries summary for all of the reported analyses (samples, blanks, *etc*.). The surrogate recovery forms should be segregated by method (*i.e*., high-level solid samples separate from low-level solid samples). The summary form

should also include the surrogate recovery limits and the laboratory should flag the compounds that do not meet the recovery limits, on the summary form.

C) A matrix spike/matrix spike duplicate concentration and percent recovery/relative percent difference summary for each matrix spike/matrix spike duplicate pair analyzed. The matrix spike/matrix spike duplicate summary form will indicate the GE identification of the unspiked sample, the MS/MSD sample, the spike concentrations, the matrix, and the concentrations of the compounds present in the unspiked sample and the MS/MSD sample. The summary form should also include the MS/MSD recovery criteria and RPD criterion. The laboratory should flag the compounds that do not meet the criteria. A similar form for the LCS must be included with the deliverables.

D) A method/storage blank summary form for each method/storage blank which identifies the samples associated with each method/storage blank. The date of analysis, time of analysis, file number, and matrix of the method/storage blank must also be reported on the summary form. Storage blanks are only required for volatiles analysis.

E) 1. A GC/MS tuning summary which summarizes the percent abundances for the mass ions of interest and the acceptance criteria for the mass ions. Additionally, the summary must include a list of the sample and QC sample analyses (sample names, file numbers, and dates and times of analysis) associated with the GC/MS tune. The summary should indicate the instrument identification, date and time of analysis, column type, diameter of the column, and the type of purge (heated or unheated for volatiles) used to analyze the samples.

2. The raw data for the GC/MS tuning summary, consisting of a summary of the mass ion abundances and a mass spectral representation of the tuning peak.

F) 1. For the internal standard calibration method, an initial calibration summary for each initial calibration performed, summarizing all of the relative response factors for each calibration standard, the average relative response factor, and the relative standard deviation among the relative response factors. If a calibration curve equation is utilized, the laboratory must summarize the curve equation and the coefficient of determination. Additionally, the summary should indicate the compounds that must meet a minimum relative response factor or a maximum relative standard deviation criterion and the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the file identifications of the analyses, the dates and times of calibration commencement and completion, column type, diameter of the column, and the type of purge (heated or unheated for volatiles) used to analyze the samples.

2. The raw data for the initial calibration, consisting of the reconstructed ion chromatogram and the raw quantitation report for each calibration standard. This is a requirement for the Level B Deliverables only.

G) 1. For the internal standard calibration method, a continuing calibration summary for each continuing calibration standard analyzed, summarizing the average relative response factors of the initial calibration associated with the continuing calibration standard, the relative response factors of the

continuing calibration standard, and the percent differences between the average relative response factors of the initial calibration and the relative response factors of the continuing calibration. If calibration curve equations are utilized the laboratory must summarize the true concentration, observed concentration, and the percent drift. Additionally, the summary must indicate the compounds that are subject to a minimum relative response factor criterion, the compounds that are subject to a maximum percent difference criterion, and the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the date of the initial calibration, the date and time of analysis, column type, diameter of the column, and the type of purge (heated or unheated for volatiles) used to analyze the samples.

2. The raw data for the continuing calibration, consisting of the reconstructed ion chromatogram and the raw quantitation report for each calibration standard. This is a requirement only for the Level B Deliverables.

H) An internal standard area counts summary, containing a summary of the area counts and retention times for the internal standards for a continuing calibration. The summary must indicate the acceptance windows for the internal standard retention times and area counts. This summary must supply a comparison of the continuing calibration internal standards to the mid-level initial calibration internal standards. Additionally, the summary must include a listing of the internal standard retention times and area counts for all of the samples, method blanks, matrix spikes, and matrix spike duplicates associated with the continuing calibration standard.

I) A copy of all of the extraction log information for semivolatiles is required. At a minimum, the extraction information must include the date the extraction was started, the date the extraction was completed, the initial sample weight or volume, final extraction volume, laboratory sample number, the amount and concentration of surrogate spike added, and the amount and concentration of matrix spike solution added. Additionally, the extraction log should indicate if a cleanup procedure was performed on the sample. If a medium-level extraction was performed for the volatiles analysis, all extraction logs for this analysis will be required. For volatile organics analyses that require weighing sample aliquots in the field, copies of the field measurement documentation will be included in this section.

A-9.3 Deliverables Reporting Requirements for Pesticide and PCB Analysis

The laboratory will be required to submit the following information as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (i.e., recovery ranges, relative percent difference limits, etc.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any recoveries that are outside of the acceptance criteria. The raw data associated with the samples, blanks, and standards must clearly identify the GE sample identification, the laboratory sample number, the instrument, the laboratory file number for the analysis, and the peak areas/heights and retention times that correspond to the compounds of interest observed in all analyses reported. If the requirement of a summary form is not applicable to a particular sample, standard or blank, the requirement should still appear on the form; however, no entry will be necessary on the form for that requirement.

A) 1. An analysis summary of the concentrations of all target compounds for all sample analyses, matrix spike analyses, matrix spike duplicate analyses, and

blank analyses. The blank analyses must consist of all of the extraction (method) blank analyses, injection blank analyses, and any blanks associated with cleanup procedures. The summary must include dates and times of analysis, GE sample identifications, laboratory sample numbers, dates of sample collection, date of sample receipt, dates of sample extraction, sample matrices, sample weights or volumes, sample percent solids, column types, column internal diameters, dilution factors, initial extract volumes/weights, final extract volumes, concentration units, the type of cleanup performed, and sample results. If positive results below the lowest calibration standard are reported, they must be flagged as estimated ("J") on the analysis summary. "Not-detected" results will be represented by the GE required quantitation limit and a "U" flag. If a compound was detected in a sample as well as in the method blank associated with the sample, the result must be flagged with a "B" on the summary form. Additionally, if a dilution is performed on a sample because a target compound is above the calibration range then the positive result for the particular compound should be flagged with a "D." If the compound is still above the calibration range after a dilution is performed on the sample, then the positive result for the compound should be flagged with an "E."

2. The raw data for the sample analyses, matrix spike analyses, matrix spike duplicate analyses, and blank analyses, consisting of the chromatograms indicating the surrogate peaks and target compound peaks and quantitation reports for the target compounds and surrogates. This is a requirement only for the Level B Deliverables.

B) A surrogate percent recovery summary for all of the reported analyses (samples, blanks, *etc*.). The surrogate recovery forms should be segregated by matrix and method. The summary form should also include the surrogate recovery limits and the laboratory should flag the compounds that do not meet the recovery limits on the summary form.

C) A matrix spike/matrix spike duplicate concentration and percent recovery/relative percent difference (RPD) summary for each matrix spike/matrix spike duplicate pair analyzed. The matrix spike/matrix spike duplicate summary form will indicate the GE identification of the unspiked sample, the MS/MSD sample, the spike concentrations, the matrix, and the concentrations of the compounds present in the unspiked sample and the MS/MSD sample. The summary form should also include the MS/MSD recovery criteria and RPD criterion. The laboratory should flag the compounds that do not meet the criteria. A similar form for each the LCS and LCSD should be included with the deliverables.

D) A duplicate sample relative percent difference summary for each laboratory duplicate analyzed. The duplicate summary form will indicate the GE identification of the parent sample, the duplicate sample, the matrix, and the concentrations of the compounds present in the parent sample and the duplicate sample. The summary form should also include the RPD criterion. The laboratory should flag the compounds that do not meet the criteria.

E) A method blank summary form for each method blank, identifying the samples associated with each method blank. The date, time, lab file number, and matrix of the method blank must also be reported on the summary form.

F) Initial Calibration Data: A summary of the initial calibration retention times, mean retention time, and a retention time window for all target compounds and surrogates must be provided for all initial calibrations. A second summary of the initial calibration standard calibration factors, average calibration factors, and relative standard deviations for all target compounds and surrogates must also be provided for all initial calibrations. If a calibration curve equations is utilized the laboratory must supply the curve equation and the correlation coefficient (or coefficient of determination). Both summaries should include the SDG number, instrument identification, GC column type and diameter, date(s) of analysis, the concentration level for each initial calibration standard (as a multiplication factor of the low calibration standard), and the acceptance limit for the relative standard deviation. Copies of the PCB standard chromatograms and integration reports associated with summaries should immediately follow the summary (only for the Level B Deliverables). Each initial calibration associated with the SDG must be presented in chronological order, by GC column and by instrument.

G) Continuing Calibration Data: A summary of the observed retention times, calculated compound concentrations, true concentrations, percent differences, and retention time window from the initial calibration (or from the daily retention time window update) must be provided for all continuing calibration standards. If calibration curve equations are utilized the laboratory must summarize the true concentration, observed concentration, and the percent drift. The summary should list the SDG number, GC column type and diameter, date and time of analysis, laboratory sample number, initial calibration dates, and acceptance limits. Copies of the PCB standard chromatograms and integration reports associated with summaries should immediately follow the summary (only for the Level B Deliverable). Each

continuing calibration associated with an SDG must be presented in chronological order, by GC column and by instrument.

H) A summary of the analytical sequence for each column and instrument used for the analysis of the project samples. The summary must contain the GC column number, the internal diameter of the column, initial calibration dates associated with the sequence, the instrument identification, the mean retention time(s) for the surrogate(s) utilized, a listing of the GE sample names, the laboratory sample numbers, dates and times of analysis, and the retention times for the surrogate(s). The summary should also indicate the retention time window for all surrogates used and any surrogate retention times that do not meet the acceptance criterion. The summary must contain all of the analyses for the samples, blanks, initial calibration standards, and continuing calibration standards associated with the sequence. All sequences will begin with an initial calibration and will terminate with a continuing calibration or breakdown check standard that meets all acceptance criteria.

I) When a GPC cleanup procedure is required for the samples, a summary for each check standard associated with the GPC calibration. The summary must contain the GPC column identification, the calibration date of the GPC column, the GC column(s) used for the analysis of the standard, the GC column internal diameter, the theoretical concentrations of the compounds in the GPC standard, the observed concentrations of the GPC standard, the percent recovery for each compound in the GPC standard, the GE sample identification, laboratory sample number, and the date(s) of analysis for all samples associated with the GPC standard. The limits for each compound in the GPC standard should be listed on the summary form. The laboratory should flag any compound if the percent recovery was not within the control limits.

J) For the internal standard quantitation technique, an internal standard area counts summary, containing a summary of the area counts and recoveries for all of the standards, samples, method blanks, LCS/LCSDs, PEs. duplicates, matrix spikes, and matrix spike duplicates. The summary must indicate the acceptance windows for the internal standard retention times and area counts.

K) Second column confirmation may be performed for PCB analyses when there is a positive result reported for a project sample. When the laboratory performs a dual column quantitative analysis for PCBs, a summary of the identified compounds and observed concentrations for the two columns utilized for sample analyses is required. The summary must contain the GE sample identification, the laboratory sample number, the dates and times of analysis, the instruments used for analysis, the GC columns, the GC column internal diameters, the retention time windows for each peak used to quantitate the compound, the observed retention time for each peak used to quantitate the compound, the calculated concentration for each peak used, the mean concentration for each column for each compound identified, and the percent difference between the mean concentrations calculated for each column.

If the percent difference between the results for the analyte from two GC columns is greater than 25% for the analysis, then the higher of the two values is reported and flagged with a "P." Finally, the "C" flag is used when the identification of a PCB result is confirmed by GC/MS.

A-9.4 Deliverables Reporting Requirements for Inorganic Analyses

The laboratory will be required to submit the following information as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (*i.e.,* recovery ranges, relative percent difference limits, *etc.*) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any quality control results that are outside the acceptance criteria. All instrument raw data printouts for the points discussed below must be provided in an orderly fashion. This applies to all required QA/QC measurements, and instrument standardization, as well as sample analysis results. Additionally, all associated extraction, digestion, and distillation logs must be supplied. The order of the raw data in the data package shouldl be ICP-AES, ICP/MS, and mercury.

A) 1. A sample reference list for all samples present in an SDG. This reference list must summarize and correlate the laboratory sample number, the GE designated sample identification, and any laboratory code (*i.e.*, truncation of GE designated sample number by the laboratory) for each sample in an SDG.

2. A Table of Contents listing page numbers associated with information such as:

a. Methodology Summary

b. Case Narrative

c. Sample Results

d. Quality Control Data

e. Verification of Instrument Parameters

f. Preparation and Analysis Logs

g. Raw Data, including but not limited to:

i. ICP-AES, ICP/MS, and Mercury Data

ii. Digestion Logs

iii. Confirmation Data

h. Chain-of-Custody Records

B) Analysis summaries of the concentrations of all target analytes for all sample analyses. The summary must include the GE designated sample number, the laboratory sample number, date of sample collection, date of sample receipt, sample matrix, sample percent solids, concentration units, sample results, data qualifier codes, analysis method codes, description of sample before and after analysis, and any comments relating to the sample.

C) Initial Calibration Data: A summary of the initial calibration he initial calibration standard response, equation, and correlation coefficient for all target analytes must be provided for all initial calibrations. Summaries should include the SDG number,

instrument identification, date(s) of analysis, the concentration level for each initial calibration standard, and the acceptance limit.

D) A summary of the initial and continuing calibration verifications for each calibration performed. This summary will include the concentrations observed as well as the true value of the analyte in the initial and continuing calibrations. A percent recovery will be summarized based on the observed and true values for each analyte.

E) A summary of the Reporting Limit (RL) standard analyses for all analyses. This summary will include the concentrations observed as well as the true value of the analyte in the RL standard. A percent recovery will be summarized based on the observed and true values for each analyte.

F) A summary of the initial and continuing laboratory blank analyses for each calibration performed. This summary will include the concentrations (positive or negative) observed of any analyte in the initial and continuing blank analyses at values greater than the MDL. The summary should also include the concentrations of any analyte observed in the laboratory preparation blank associated with each calibration sequence performed by the laboratory. For mercury analyses by EPA Method 1631E, the results of all bubbler, system, and reagent blanks should also be summarized (with mean and standard deviation, as applicable).

G) A summary of the ICP or ICP/MS interference check sample analysis for each analytical sequence performed. This form will summarize the true and found values (positive, negative, or zero) of all analytes present in Solutions A and AB of the ICP or ICP/MS interference check sample analysis. This form will also summarize the percent recoveries of the analytes/interferences present in the standards.

H) A summary of the pre-digestion matrix spike analysis. This form will summarize the percent recovery control limit for each analyte. Also, the sample result, the spike sample result, and the spike-added amount must be summarized on this form for all parameters analyzed. The laboratory-calculated percent recovery as well as the laboratory qualifier stating whether the calculated percent recovery was within control limits must also be summarized on this form.

I) A summary of the post-digestion matrix spike analysis. This form will require the same information described in item G.

J) A summary of the laboratory duplicate analysis. This form will summarize the percent differences observed between the sample and laboratory duplicate analyses. The appropriate control limits must be specified by the laboratory, and a summary of the sample and laboratory duplicate analyses must be provided. The percent solids for the sample and the duplicate sample should be included on the summary form.

K) A summary of the Laboratory Control Sample (LCS) analysis. This form will summarize the percent recovery, control limits, and true and found values for the solid sample analyses.

L) A summary of the ICP or ICP/MS Serial Dilution analyses performed by the laboratory. This summary will show the result of the initial sample analysis (in aqueous units, as observed from the raw data), the result of the five-fold serial dilution analysis, and the percent difference between the two analyses.

M) The summaries necessary for the verification of instrument parameters. These include an Method Detection Limit and Reporting Limit Summary, an ICP Interelement Correction Factor Summary (if performed) for each ICP and ICP/MS used for analysis, and an ICP or ICP/MS Linear Range Summary (if performed) for each ICP and ICP/MS used for analysis.

N) The analysis log summaries. These include a Sample Preparation log that provides the sample identification; the preparation date; the sample weight (in grams) used; and the digestion volume (in mL) used and an Analysis Run Log that provides the instrument identification, the sample identification, any dilution factors employed in the analysis, the date and time of analysis, the method of analysis, and the parameters analyzed.

A-9.5 Deliverables Reporting Requirements for General Chemistry/Conventionals Analysis

The laboratory will be required to submit the information detailed in Sections A-9.4 A) -D), A-9.4 F) and A-9.4 H), J), K), and N) as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (*i.e.*, recovery ranges, relative percent difference limits, *etc*.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any quality control results that are outside the acceptance criteria. All instrument raw data printouts for the points discussed in the above mentioned sections must be provided in an orderly fashion. This applies to all required QA/QC measurements, and instrument standardization, as well as sample analysis results. Additionally, a direct sequential readout must be included if the instrument has the capability.

APPENDIX P2-1
SAMPLE PREPARATION FOR VOLATILE ORGANICS BY SW-846 METHOD 5035A (S-LI-O-012-REV.00)

STANDARD OPERATING PROCEDURE

SAMPLE PREPARATION FOR VOLATILE ORGANICS

Reference Methods: Methods 5035A (5035A-L, 5035A-H)

Local SOP Number:	S-LI-O-012-rev.00
Effective Date:	Date of Final Signature

APPROVALS

[signature]	5/12/15
General Manager	Date
[signature]	5/9/15
Quality Manager	Date
[signature]	5/12/15
Department Manager	Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. IDENTIFICATION OF TEST METHOD

1.1. This Standard Operating Procedure (SOP) documents the procedures used by PASI – Long Island, NY for sample preparation of volatile organics. The laboratory utilizes this procedure described based on EPA Method 5035A in SW846.

1.2. In addition to the requirements of this SOP, the guidelines in the *Quality Assurance, Quality Control Manual* have to be observed.

2. SUMMARY OF METHOD

2.1. Low Level Procedure -5035A-L

2.1.1. For low concentrations, approximately 5.0g of sample is collected with a corer and transferred into a pre-weighed vial with stirring bar. The samples are frozen in the lab. Alternatively samples collected with the Encore sampler are transferred in the laboratory to a pre-weighed purge and trap vial, containing a stirring bar.

2.1.2. 1.0g to 10.0g of sample can be used as appropriate for the project.

2.1.3. After addition of water and spikes, the sample is heated to 40ºC and agitated with a stirring bar. The analytes are purged with a continuous flow of Helium into the headspace; the analytes are trapped, and consequently desorbed onto the analytical column.

2.2. Medium/high Level Procedure -5035A-H

2.2.1. For medium/ high level soils, samples are placed into vials containing methanol as preservative/ extraction solvent. 10.0mL of methanol is pipetted into each pre-weighed vial, before approximately 5.0g of sample is added from the corer.

2.2.2. As in the low method, the sample collected in Encore samplers may be transported to the laboratory, where they are transferred to methanol preserved vials.

2.2.3. 1000µL aliquots (undiluted or diluted) of the methanol solutions are spiked into 50.0mL of water and analyzed, by the same protocol as water, EPA Method 5030C.

3. SCOPE AND APPLICATION

3.1 This procedure follows the sampling and preparation procedures 5035A for both high and low level (5035A-H and 5035A-L).

3.2 The sampling procedure of this method may be used for sampling of samples for analysis by methods 8260C or 8015D.

3.3 The sampling procedure applies to sampling of solid samples. Limitations for the type of sample medium are set by the suitability of the sampling/coring device for the type of sample, which could consist of various types of soils.

3.4 The moisture content of the sample may also limit the usability of the coring device. The moisture content of the sample in calculated and applied to the results. Sample results are reported as dry weight unless otherwise stated. The method may also be used for sampling high oily samples, provided the oily

substance is soluble in a water miscible solvent. The solvent must not interfere with the analytical procedure.

3.5 To avoid the losses occurring in traditional bulk sampling in jars due to volatilization and biodegradation of analytes, samples are collected and analyzed headspace free.

3.6 The sampling procedure is applicable for volatile organics with boiling points below 200°C with low water solubility. Some modification of preservatives applies in 5035/A for analysis of water-soluble oxygenates that require heated purge and trap procedure. For a list of analytes, which can be analyzed by ambient or heated purge and trap procedure, refer to method 8260C.

3.7 For “closed-system procedure”, the soil samples are collected with a coring device and placed directly into the purge and trap vials (or methanol containing vials) and hermetically sealed. Alternatively, special coring devices are available (Encore samplers) that can be closed and thus used for transfer to the lab, where the soil is then extruded into the appropriate vials, purge and trap vials or vials with methanol for medium level analysis.

3.8 For Low Level Procedure, the entire sample is loaded on the purge and trap auto-sampler without opening, and the sampling vial is used as a sparge vessel.

3.8.1 This procedure is generally applicable to soils and other solid samples with VOC concentrations in the range of 2 to 200 µg/kg

3.9 For the medium/high level samples, the analytes are dissolved in methanol, which is added to the vials and the methanol solution in then analyzed.

3.9.1 The Medium/High Level Procedure is applicable to samples above 200µg/Kg or 1000µg/Kg, depending on what low level is calibrated in the determinative procedure

3.9.2 For very high samples, high dilutions may be prepared by adding only smaller aliquots of the methanol to the water.

4. APPLICABLE MATRICES

4.1. The method is intended for the analysis of compounds in all types of solid waste matrices, and soils.

5. LIMITS OF DETECTION AND QUANTITATION

5.1. For requirement and procedure refer to the determinative method.

6. INTERFERENCES

6.1. Interferences can be compounds in the sample “interfering” with the analysis or secondary contaminations from the instrument or introduced during sample storage or sample preparation.

6.2. If other analytes are interfering, (co eluting), identification of the targeted analytes is generally still possible, by comparison with the standard spectra.

6.3. Field blanks; trip blanks should be collected with each sampling batch to determine whether any contamination occurred during sampling or shipping.

6.4. An equipment rinsate blank from sampling device should be collected for each matrix.

6.5. To avoid secondary contaminations, the samples have to be stored away from standards in an area free of volatile contaminants which might permeate through the septa, namely Freons and Methylene chloride, which might be present in the lab atmosphere. Vials containing reagent water or solid matrix are stored together with the samples to serve as holding blanks.

6.6. Method blanks are required before samples are analyzed in each 12hr period to demonstrate that the instrument and reagents are free from interferences.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

7.1. **Table 7.1 – Sample Collection, Preservation, Storage, and Hold time**

	Option 1 (recommended)	Option 2 (EnCore or Core N One or CEL-5035)	Option 3 (If sample cannot be transported to the lab or frozen within 48hrs)
Preparation of Sampling Containers	2- pre-weighed 40mL vials containing a small magnetic stir bar (for low level analysis.) 2- additional vials with stir bars if client requires MS and MSD 1- pre-weighed 40mL vial for medium-high level analysis 2- 1oz jars for % solids and possible bulk/high level analysis. 1- disposable open barrel syringe with HDPE plunger, graduated ½mL to 5mL	3- EnCore (or equivalent) 5.0g samplers for low level analysis 2- additional Encore samplers are required if clients requires MS and MSD 2- 1oz jars for % solids and possible bulk/high level analysis.	3- pre-weighed Sodium Bisulfate preserved VOC vials with stir bars for low level analysis. 2-additional pre-weighed Sodium Bisulfate preserved VOC vials with stir bars if client requires MS and MSD 1- pre-weighed Methanol preserved VOC vial for high level analysis 2- 1oz jars for % solids and possible bulk/high level analysis.
Sampling Instructions	1. Push the syringe into the soil to approx. the 2.5mL mark. This will collect about 5.0g sample. 2. Immediately extrude the entire contents of the syringe into one of the 40mL pre-weighed vials and cap the vials. To seal properly there must be no particles between the cap liner and the vial rim. (Repeat process for additional vials) 3. Mark each sample container with your sample identification. Do not add any additional labels or tape to pre-weighted vials. 4. For the 1oz jars, pack these containers to full capacity with minimal headspace.	1. Uncap one of the EnCore samplers and push into the soil to fill its cup. This will collect about 5.0g of sample. Cap the sampler making sure that the soil particles are carefully cleared away that might cause a leak in the O-ring seal. 2. Repeat with additional EnCore samplers. 3. Mark each sample container with your sample identification. Do not add any additional labels or tape to pre-weighted vials. 4. For the 1oz jars, pack these containers to full capacity with minimal headspace.	1. Push the syringe into the soil to approx. the 2.5mL mark. This will collect about 5.0g sample. 2. Immediately extrude the entire contents of the syringe into one of the 40mL pre-weighed vials and cap the vials. To seal properly there must be no particles between the cap liner and the vial rim. (Repeat process for additional vials) 3. Mark each sample container with your sample identification. Do not add any additional labels or tape to pre-weighted vials. 4. For the 1oz jars, pack these containers to full capacity with minimal headspace.

Sample Receipt, Storage and Holding times	Follow receiving protocol for “routine” or CLP samples and Volatiles in Soil Method 5035A Sample Collection policy. Weigh and record the weight of each sample container to the nearest 0.1g. Samples must be received by the lab within 48hrs while stored at 4 ± 2°C. They will then be preserved by the laboratory either by freezing at < -7°C or with chemical preservatives before the 48hrs has expired. Samples are stored in a freezer located in an area free of volatile contaminants. To prevent cross contamination, samples suspected to contain high concentration levels are sealed in cans or bags containing carbon granules and are stored in a refrigerator designated for high concentration samples. The holding time for samples is 14 days from sample collection.	Follow receiving protocol for “routine” or CLP samples and Volatiles in Soil Method 5035A Sample Collection policy. Weigh and record the weight of each sample container to the nearest 0.1g. Samples must be received by the lab within 48hrs while stored at 4 ± 2°C. They will then be preserved by the laboratory either by freezing at < -7°C or with chemical preservatives before the 48hrs has expired. Samples are stored in a freezer located in an area free of volatile contaminants. To prevent cross contamination, samples suspected to contain high concentration levels are sealed in cans or bags containing carbon granules and are stored in a refrigerator designated for high concentration samples. The holding time for samples is 14 days from sample collection. There is no provision for freezing samplers stored in the original EnCore samplers in method 5035A.	Follow receiving protocol for “routine” or CLP samples and Volatiles in Soil Method 5035A Sample Collection policy. Weigh and record the weight of each sample container to the nearest 0.1g. Samples are stored at 4 ± 2°C Samples are stored an area free of volatile contaminants. To prevent cross contamination, samples suspected to contain high concentration levels are sealed in cans or bags containing carbon granules and are stored in a refrigerator designated for high concentration samples. The holding time for samples is 14 days from sample collection.

<u>NOTE</u>: If samples are not collected by 5035A collection techniques, a qualifier needs to be added to the LIMS. A note ***<u>MUST</u>*** be on the final report. *<u>Qualifier:</u>* Results may be biased low due to sample not being collected according to 5035A low level specifications.

<u>Note:</u> Sodium Bisulfate and Methanol preservation methods can cause problems. Sodium Bisulfate has been shown to cause analytical interferences, effervescence of samples, and damage to GCMS traps, destruction of target compounds and creation of acetone. Methanol has shipping restrictions due to the flammability and increased creation of hazardous waste. In addition, methanol and water losses from the vials are not unusual necessitating marking of vials or field weighing to account for such losses. For these reasons PASI-Long Island favors option 1 or option 2.

8. DEFINITIONS

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual.

9. EQUIPMENT AND SUPPLIES

9.1. **Table 9.1 – Instrumentation**

For instrumentation refer to the determinative method.

9.2. **Table 9.2 - Chromatography Supplies**

For Chromatography Supplies refer to the determinative method.

Table 9.3 – Glassware

Glassware	Description	Vendor/ Item # / Description
Glass Vials	40mL VOA vials unpreserved with Teflon-lined screw caps	C and G/ LV54-A000-A01A-P01 / Case of 80 *
Jars	1oz – Stand clear glass short wide mouth w/ PTFE lined caps 2oz- Stand clear glass short wide mouth w/ PTFE lined caps 4oz- Stand clear glass short wide mouth w/ PTFE lined caps 8oz- Stand clear glass short wide mouth w/ PTFE lined caps 16oz - Stand clear glass short wide mouth w/ PTFE lined caps	1oz – Sci/Spec / B70801 2oz- Sci/Spec / B70802 4oz- Sci/Spec / B70804 8oz- Sci/Spec / B70808 16oz- Sci/Spec / B70816*
Glass Pipets	9" glass pipets	Fisher/ 22-230490/ Case of 1000*
Volumetric Flasks	5mL, 50mL, 100mL	Class A*

*Or equivalent

9.3. **Table 9.4 - General Supplies**

Supply	Description	Vendor/ Item # / Description
Balance	Capable of weighting 100g ± 0.01g	Ohaus CS200*
Spatula	Narrow, stainless steel	
Ottowa Sand	Purified solid matrix	EMD/ SX0070-1*
Magnetic stir bars	Stirring bars to fit 40mL vials	
Gas tight syringes	10µL, 25µL, 50µL, 100µL, 250µL; 1mL, 2.5mL, 5mL; with Luerlok tip	Hamilton
Syringe valve	With male and female Luerlok connections	
Oven		

*Or equivalent

For any other supplies refer to the determinative method.

10. Reagents and Standards

10.1. **Table 10.1 – Reagents and Standards**

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Organic-free Water (OFW)	De-ionized water	Verify that background levels of volatile compounds are acceptable by analysis
Methanol	Purge and trap grade	Honeywell/ 232-1L*
Hydrochloric Acid	Solution of 1:1 of concentrated HCl and De-ionized water	
Sodium bisulfate	ACS reagent grade, granular	

* Or Equivalent

For other regents and standards refer to the determinative method.

11. Calibration and Standardization

For requirement and procedure refer to the determinative method.

12. Procedure

12.1. **Sample Preparation- Low Concentration Soil Samples**: 5035A-L

12.1.1. Weigh received vials to nearest 0.1 g, subtract tared weight, and record the net weight for the sample.

12.1.2. Obtain unpreserved vials from the Receiving Department for method blanks and lab fortified blanks. Add 5.0g of Ottawa sand to each of the vials.

12.1.3. Prepare vials for MS, MSD, and LFB by spiking 5.0g of the designated MS/MSD sample or Ottawa sand (for the LFB) with 5mL of the matrix spiking solution.

12.1.4. Check and refill reagent water dispenser of auto-sampler and reservoir for surrogate solution/ internal standard solution used for automatic spike addition.

12.1.5. After calibration for heated purge is performed according to analytical method, load Q. C. and sample vials on the auto-sampler and record sample information, and start purge and trap procedure for heated purge.

12.1.6. Separately determine percent moisture for each sample. This procedure may be performed in the inorganic department.

12.2. **Sample Preparation- Medium level soil samples:**

12.2.1. Transfer several mL of the methanol supernate in the sample vials into small storage vials. (A minimum of 4mL are needed for MS/MSD samples.) If the sample is not processed further, mark the level of the meniscus and store in the refrigerator at 4 ± 2°C.

12.2.2. In 50mL volumetric flasks, prepare water solutions with 1000 µL of the methanol extracts, or less, depending on prescreening results or prior knowledge. 5.0mL of this solution therefore contains 100µL of the methanol sample solution, which is equivalent to 1/100 of the 5.0g of sample extracted into 10mL of methanol. If the lowest calibration level is 50ng/5mL the reporting limit is equal to 1000µg/kg. For a low calibration of 25ng/5mL (5µg/L) the 5035A-H reporting limit is 500µg/kg.

12.2.3. The water solutions, containing the soil extract aliquots, are processed further as water samples as described in the determinative method. For the MS/MSD duplicates, prepare 100mL of water solution separately from the sample solution from the same methanol extract and spike with 5000ng (200µL of the matrix spiking solution). The resulting concentration corresponds to a soil concentration of 7500µg/kg for a 5.0g sample in 10mL methanol.

12.2.4. For the matrix spike blank, use 1000uL of methanol to spike 50mL of water.

12.2.5. Similarly, prepare 50mL of reagent water, containing 1000µL of methanol for lab-fortified blank solution and spike with 100µL of matrix spiking solution for a concentration corresponding to 7500 µg/kg for 5.0g in 10mL methanol.

12.3. **Sample Analysis**-

For requirement and procedure refer to the determinative method

13. Quality Control

For requirement and procedure refer to the determinative method

14. Data Analysis and Calculations

For requirement and procedure refer to the determinative method

15. Data assessment and Acceptance Criteria for Quality Control Measures

For requirement and procedure refer to the determinative method

16. Corrective Actions for Out-of-Control Data

For requirement and procedure refer to the determinative method

17. Contingencies for Handling Out-of-Control or Unacceptable Data

For requirement and procedure refer to the determinative method

18. Method Performance

For requirement and procedure refer to the determinative method

19. Instrument/Equipment Maintenance/Troubleshooting

For requirement and procedure refer to the determinative method

20. Safety

20.1. All in-house safety regulations have to be observed during sample preparation and analysis.

20.2. **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.

20.2.1. Read information and follow warnings listed on the labels of the containers of the chemicals.

20.2.2. In addition, the reference file of material safety data sheets (MSDS) should be consulted for properties of the chemicals used, to determine handling precautions.

20.3. Safety glasses, a lab coat, and disposable gloves must be worn when handling chemicals and samples.

20.4. Sample handling should be conducted in fume hoods.

20.5. The company wide Chemical Hygiene and Safety Manual contains additional information on safety.

20.6. **Equipment:** Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones. The purge and trap concentrator and autosampler use gas under pressure to purge samples and, in some cases, drive the robotic assemblies. These high pressures introduce the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

21. Waste Management

21.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste.

21.2. Refer to the lab's *Sample and Waste Management* SOP.

22. Pollution Prevention

For requirement and procedure refer to the determinative method

23. References

23.1. For 5035A: "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update July 2002

23.2. Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update Dec. 1996

23.3. National Environmental Laboratory Accreditation Conference (NELAC) Constitution, Bylaws and Standard, approved June 5, 2003, (EPA/600/R-04/003)

23.4. 40CFR Part 136 Appendix B

23.5. 2009 TNI Laboratory Accreditation Standards, EL-V1-2009.

24. Tables and Figures

For requirement and procedure refer to the determinative method

25. REVISIONS

Document Number	Reason for Change	Date
S-LI-O-012-rev.00	Transition to PACE format. Separated preparation method from determinative methods.	04/06/2015

APPENDIX P2-2
DETERMINATION OF VOLATILE ORGANICS BY GC/MS BY SW-846 METHOD 8260C
(S-LI-O-011-REV.00)

STANDARD OPERATING PROCEDURE

DETERMINATION OF VOLATILE ORGANICS BY GC/MS

Reference Methods: EPA SW-846 Methods 8260C

Local SOP Number:	S-LI-O-011-rev.00
Effective Date:	Date of Final Signature
Supersedes:	*8260C_r2*

APPROVALS

General Manager	Date 5/12/15
Quality Manager	Date 5/9/15
Department Manager	Date 5/12/15

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. IDENTIFICATION OF TEST METHOD

1.1. This Standard Operating Procedure (SOP) documents the procedures used by PASI – Long Island, NY 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 EPA Method 8260C in SW-846.

1.2. For work governed by the NYS DEC Analytical Service Protocol (ASP), the requirements for analysis and reporting of the DEC ASP have to be met.

1.2.1. For reporting of data packages with full documentation according to ASP requirements, all raw data have to be included, and summary tables of calibrations and Q. C. data have to be submitted on forms as specified in the DEC ASP.

1.3. In addition to the requirements of this SOP, the guidelines in the *Quality Assurance, Quality Control Manual* have to be observed.

2. SUMMARY OF METHOD

Volatile organic compounds are introduced into the gas chromatograph by a purge-and trap method. The analytes are purged from a sample aliquot with helium. The purged analytes are collected in a trap. At the completion of the purge time, the trap is rapidly heated and back flushed with helium 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 Table 11.1 represents a list of the targeted analytes (with the CAS registry) which have to be identified and quantified most frequently against calibration standards. Other volatile analytes may be included in the scan as required.

3.3 Due to their solubility in water oxygenated compounds like alcohols have poor purge efficiency. In order to include volatile oxygenated compounds, they may be purged at elevated temperature (80ºC). See Attachment VIII. A list of commonly analyzed fuel oxygenates is present in Attachment XI.

3.4 This method is applicable to most water and solid samples, regardless of moisture content. Common matrices are ground and surface water, wastewater, aqueous sludge, sediment, soils, and other solid samples. Procedures may need to be adapted to address limits in the method or equipment that might hinder or infere with sample analysis. All adaptations made to address matrix related modifications must be documented within the analytical data.

3.5 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.

3.6 This method cannot be substituted for other similar published methods where permit or regulatory compliance is required

4. APPLICABLE MATRICES

4.1. The method is intended for the analysis of volatile compounds in all types of solid waste matrices, soils and ground and surface water.

5. LIMITS OF DETECTION AND QUANTITATION

5.1. Reporting limits are presented in Table 11.1, “Practical Quantification Limits” (PQL).

5.2. Reporting limits must be determined at the start of a project and reporting conventions must be established with the client. Project specific requirements must be communicated to analysts prior to sample analysis.

5.3. MDL studies are performed annually by the analysis of seven low level standards at three to five times the expected MDL and calculated by the procedure defined in 40CFR Part 136 Appendix B.

5.3.1. The MDLs define the lowest levels, where positives will be found with 99 percent confidence with the particular analytical method in clean media.

5.3.2. Analyze the extracts on a calibrated instrument that meets all performance check criteria. Tabulate the results and statistically evaluate the standard deviations.

5.3.3. From the obtained standard deviation (S) calculate the MDL as follows:

Method Detection Limit (MDL)

$$MDL = t_{n-1} \times S$$

Where:

S = Standard deviation

t_{n-1} = Students t-Test value (for seven replicates $t_{n-1} = 3.14$)

5.4. Current Method Detection Limits (MDLs) are on file and available by request from the Quality Manager.

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 (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. All materials utilized during this analysis and the GC/MS system must be demonstrated to be free from contamination. Running frequent instrument blanks and method blanks along with using purge and trap grade solvents will assist with the monitoring of laboratory contaminants within the analytical system. When potential interfering peaks are noted in laboratory reagent blanks, the analyst must determine the source of contamination and correct the problem before analysis of samples may continue.

6.3. 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 two or more portions of organic free water between samples. Analyze one or more blanks to check for cross contamination prior to sample analysis.

6.4. Since methylene chloride and acetone are common laboratory solvents, special precautions must be taken. The volatiles analysis and sample storage area should 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, SHIPMENT AND STORAGE

7.1. **Table 7.1 – Sample Collection, Preservation, Storage, and Hold time**

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Two VOA vials	ALL DONE IN FIELD Acidified w/ 1:1 HCl (1-2 drops) to pH<2, no headspace AND Halogenated and aromatic compounds require preservation with thiosulfate, if samples are chlorinated. *NOTE:* 2-CEVE requires an unpreserved sample.	4 ± 2°C Must be free of organic solvent vapors and direct or intense light	pH Preserved: 14 days (ASP 2000 Protocol: 10 days from VTSR*) (ASP 2005 Protocol: 12 days from VTSR*)

Sample type	Collection per sample	Preservation	Storage	Hold time
Soils	Two pre-weighed 40mL VOA vials containing a small magnetic stir bar for low level analysis (Two additional vials are needed for MS/MSD) **ALSO** One pre-weighted 40mL VOA vial for medium-high level analysis. **AND** One ounce glass jar for % solids and possible bulk/high level analysis.	Refer to Method 5035A for soil preservation techniques *NOTE*: If samples are not collected by 5035A collection techniques, a qualifier needs to be added to the LIMS. A note ***MUST*** be on the final report. *Qualifier:* Results may be biased low due to sample not being collected according to 5035A low level specifications.	With sodium bisulfate or H2O: 4 ± 2°C Filled vials need to be frozen between< -7.0°C within 48 hours of sample collection.	Unpreserved or not stored frozen: 48 hours Preserved with sodium bisulfate or stored frozen: 14 days (ASP 2000 Protocol: 10 days from VTSR*) (ASP 2005 Protocol: 12 days from VTSR*)
TCLP Leachates	Two VOA vials	Filled and capped to eliminate any headspace	4 ± 2°C	14 days from end of leaching procedure

*VTSR = Verified Time of Sample Receipt

7.2. **Table 7.2 – Blank Requirements**

Trip Blank	Storage Blank	Field Blank	Method Blank
One 40mL VOA vial w/ reagent DI water Headspace free Preserved the same way as samples	One 40mL VOA vial w/ reagent DI water Headspace free Preserved the same way as samples	One 40mL VOA vial w/ reagent DI water Headspace free Preserved the same way as samples	One 40mL VOA vial w/ reagent DI water Headspace free Preserved the same way as samples

8. DEFINITIONS

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual.

8.1.1. **Toxicity Characteristic Leaching Procedure (TCLP) –** An extraction procedure used to determine if a sample is acceptable for upland disposal. The extraction procedure is meant to simulate the leaching of contaminants under the environmental conditions typically found in a landfill.

9. Equipment and Supplies

9.1. **Table 9.1 – Instrumentation**

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatograph	HP	5971/5972/5973	*
P&T Concentrator	HP/ Tekmar	LSC-2000 LSC-3000 LSC-3100	*
Chemstation	EnviroQuant Chemstation	G1701BA Version B.01.00	*
Autosampler	EST	Archon/ Centurion	*
Mass Spectral Library Program	NIST	Version 2.0	For the NIST/EPA/NIH Mass Spectral Library*

*Or equivalent

9.2. **Table 9.2 - Chromatography Supplies**

Item	Vendor	Model / ID	Catalog #	Description
Analytical Column	Restek	RTX-624	40924	20 meters, 0.18mmID, 1umdf*
Trap	Supelco	K trap	24920-U	Vocarb 3000*
Electron Multiplier	DeTech	2300	2300	*

*Or equivalent

9.3. **Table 9.3 – Glassware**

Glassware	Description	Vendor/ Item # / Description
Glass Vials	40mL VOA vials unpreserved with Teflon-lined screw caps	C and G/ LV54-A000-A01A-P01 / Case of 80 *
Glass Vials	40mL VOA vial Preserved 0.5mL trace grade HCl with Teflon-lined screw caps	Sci/Spec / 376840-.5THCL/Case of 72*
Jars	1oz – Stand clear glass short wide mouth w/ PTFE lined caps 2oz- Stand clear glass short wide mouth w/ PTFE lined caps 4oz- Stand clear glass short wide mouth w/ PTFE lined caps 8oz- Stand clear glass short wide mouth w/ PTFE lined caps 16oz - Stand clear glass short wide mouth w/ PTFE lined caps	1oz – Sci/Spec / B70801 2oz- Sci/Spec / B70802 4oz- Sci/Spec / B70804 8oz- Sci/Spec / B70808 16oz- Sci/Spec / B70816*
Glass Pipets	9" glass pipets	Fisher/ 22-230490/ Case of 1000*
Volumetric Flasks	5mL, 50mL, 100mL	Class A*

*Or equivalent

9.4. **Table 9.4 - General Supplies**

Supply	Description	Vendor/ Item # / Description
Gas tight syringes	10µL, 25µL, 50µL, 100µL, 250µL; 1mL, 2.5mL, 5mL; with Luerlok tip	Hamilton
Syringe valve	With male and female Luerlok connections	
pH paper	pH range 0-14	Fisher/ M1095350007/ 100 strips*
Balance	Capable of weighting 100g ± 0.01g	Ohaus CS200*
Spatula	Narrow, stainless steel	
Ottowa Sand	Purified solid matrix	EMD/ SX0070-1*
Magnetic stir bars	Stirring bars to fit 40mL vials	
Oven		
Helium		

*Or equivalent

10. Reagents and Standards

10.1. **Table 10.1 – Reagents and Stock Standards**

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Organic-free Water (OFW)	De-ionized water	Verify that background levels of volatile compounds are acceptable by analysis
Methanol	Purge and trap grade	Honeywell/ 232-1L
Hydrochloric Acid	Solution of 1:1 of concentrated HCl and De-ionized water	
Sodium bisulfate	ACS reagent grade, granular	
Stock Tuning standard	4-Bromofluorobenzene (2000µg/mL)	Ultra / STS-110N
Stock VOA calibration standard*	VOC Mix (200µg/mL)	Ultra/ DWM-580
Stock VOA calibration standard*	Custom Standard (2000 µg/mL)	Ultra/CUS-2244
Stock VOA calibration standard*	Custom Standard (5000µg/mL)	Ultra/CUS-6008
Stock VOA calibration standard*	Custom Standard (5000µg/mL)	Ultra/CUS-6009
Stock VOA calibration standard*	4-ethyltoluene (5000µg/mL)	Ultra/RAB-037
Stock VOA calibration standard*	1,2,4,5-tetramethylbenzene(5000µg/mL)	Ultra/RAB-010
Stock VOA calibration standard*	1,4-Diethylbenzene (5000µg/mL)	Sigma-Aldrich/E49806-55
Stock VOA Matrix Spiking Solution*	Volatiles Matrix Spiking Solution	Ultra/CLP-100N-1
Stock Internal mix*	2500µg/mL	Ultra/STM-272
Stock Internal mix*	2000µg/mL	Ultra/STM-341N
Stock Surrogate mix*	2500µg/mL	Ultra/STM-262

*Equivalent stock standards will be added or removed based on state and client analyte requirements.

10.2. **Table 10.2 - Standard Definitions**

Standard	Description	Comments
Tune Standard	4-Bromofluorobenzene (BFB) solution used to verify ion response ratios prior to analysis	Must be 50ng or less
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	Method requires a minimum of 5 levels
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify that the instrument response has not changed significantly since the initial calibration was performed.	
Second Source Verification Standard	A standard prepared from a source other than that used for the initial calibration. This mid-level standard verifies the accuracy of the calibration curve.	
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.	Pentafluorobenzene 1,4-Difluorobenzene Chlorobenzene-d5 1,4-Dichlorobenzene-d4 Bromochloromethane
Surrogate Standard	A solution added to all samples, spikes, control samples, and method blanks prior to analysis.	4-Bromofluorobenzene 1,2-Dichloroethane-d4 Toluene-d8
Spiking Standard	This solution contains all target analytes and is spiked one per batch	

10.3. **Table 10.3 - Standard Storage Conditions**

Standard Type	Description	Expiration	Storage
Stock Solutions	Concentrated reference solution purchased directly from approved vendor	§ Manufacturer's recommended expiration date for unopened ampulated standards. § All other stock standards must be replaced 6 months after ampule is opened or on expiration date, whichever is sooner.	§ Manufacturer's recommended storage conditions § Optionally the solutions may be kept in the freezer. § When standard is opened, record all information in the standard logbook.
Intermediate and Working Standard Solutions	Reference solutions prepared by dilutions of the stock solution	§ 2 to 6 months from preparation or the expiration date listed for the stock source. § Working solutions must be checked frequently and replaced if degradation or evaporation is suspected. Or if changes of concentrations are observed or suspected.	§ Store in amber vials with Teflon lined screw caps § Manufacturer's recommended storage conditions for stock source solution. § If stock source conditions conflict, store standard at coldest condition of any source.

10.4. **Standard Sources**: Standards are prepared from commercially available multi-compound stock solutions and neat materials by multiple dilutions. The sources of the stock solutions and neat materials, recipes for preparing dilutions and working standards are presented in Table 10.1 and 10.4. All intermediate standards are prepared using purge and trap grade methanol and stored in a freezer in glass vials with Teflon lined screw caps or as recommended by the standard manufacturer.

10.5. **Working Standard Preparation**: Working calibration standards are made into deionized water for the purpose of direct analysis by the analytical instrumentation. The standards must be made in a volumetric flask. Several alternatives exist but the method employed by PASI – Long Island, NY utilizes volumetric flask technique according to the following procedure. The individual standards can be made according to the details provided in Table 10.4.

10.6. **Volumetric flask preparation- Table 10.4 – Working Standard Dilutions and Concentrations**

Standard Concentration (BASED ON 100mL FINAL VOLUME)								
Stock Conc.	200µg/L	100µg/L	50µg/L	20µg/L	10µg/L	5.0µg/L	1.0µg/L	0.5µg/L
100µg/mL	200.0µL	100.0µL	50.0µL	20.0µL	10.0µL	5.0µL	*	**
200µg/mL	100.0µL	50.0µL	25.0µL	10.0µL	5.0µL	2.5µL	*	**
500µg/mL	40.0µL	20.0µL	10.0µL	4.0µL	2.0µL	1.0µL	*	**
1000µg/mL	20.0µL	10.0µL	5.0µL	2.0µL	1.0µL	0.5µL	*	**
2000µg/mL	10.0µL	5.0µL	2.5µL	1.0µL	0.5µL	0.25µL	*	**
2500µg/mL	8.0µL	4.0µL	2.0µL	0.8µL	0.4µL	0.2µL	*	**
5000µg/mL	4.0µL	2.0µL	1.0µL	0.4µL	0.2µL	0.1µL	*	**
10000µg/mL	2.0µL	1.0µL	0.5µL	0.2µL	0.1µL	NA 1:20	*	**
50000µg/mL	0.4µL	0.2µL	0.1µL	NA 1:5	NA 1:10	NA 1:20	*	**
*5mL of the 10µg/L standard up to 50mL **5mL of the 10µg/L standard up to 100mL NA standards use the appropriate dilution from the 100µg/L standard								

11. CALIBRATION AND STANDARDIZATION

11.1. **Tune Verification –** The mass spectrometer tune status must be verified prior to initial calibration and at the beginning of each analytical sequence. If the current tune status does not meet the ion ratio criteria in the method, follow the equipment manufacturers' instructions for re-tuning the mass spectrometer. The tune status must be verified after the tuning procedures. Refer to section 12.2 Procedures for details on the analysis and evaluation of this standard.

11.2. **Initial Calibration:**

11.2.1. **Analysis of Standards**: An initial calibration curve using a minimum of five points is analyzed prior to analyzing client samples. The lowest concentration must be at or below the equivalence of the standard reporting limit. The lowest calibration point reflects the practical quantitation limit for that compound, a level below which all reported results must be qualified as estimated values. Refer to table 11.1 for targeted analytes concentrations. For low level analysis include 0.4µg/L as required.

Table 11.1: Calibration standard compound concentrations*

Analyte	PQL water (µg/L)	PQL Soil (µg/L)	CAS No.	Std 1 µg/L	Std 1 µg/L	Std 2 µg/L	Std 2 µg/L	Std 3 µg/L	Std 3 µg/L	Std 4 µg/L	Std 4 µg/L	Std 5 µg/L	Std 5 µg/L	Std 6 µg/L	Std 6 µg/L	Std 7 µg/L	Std 8 µg/L
1,1,1,2-Tetrachloroethane	1.0	5.0	630-20-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,1,1-Trichloroethane	1.0	5.0	71-55-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,1,2,2-Tetrachloroethane	1.0	5.0	79-34-5	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,1,2,2-Tetrafluoro-1,2-dichloroethane (Freon-114)	1.0	5.0	76-14-2	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,1,2-Dibromo-1,2,2-trifluoroethane (Freon-113)	1.0	5.0	76-13-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,1,2-Trichloroethane	1.0	5.0	79-00-5	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,1-Dichloroethane	1.0	5.0	75-34-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,1-Dichloroethene	0.4	5.0	75-35-4	0.4	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,1-Dichloropropene	1.0	5.0	563-58-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,2,3-Trichlorobenzene	1.0	5.0	120-82-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,2,3-Trichloropropane	0.4	5.0	96-18-4	0.4	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,2,3-Trimethylbenzene	1.0	5.0	526-73-8	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,2,4,5-Tetramethylbenzene	1.0	5.0	95-93-2	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,2,4-Trichlorobenzene	1.0	5.0	95-63-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,2,4-Trimethylbenzene	1.0	5.0	95-63-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,2-Dibromo-3-chloropropane	1.0	5.0	96-12-8	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,2-Dibromoethane	0.4	5.0	106-93-4	0.4	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,2-Dichlorobenzene	1.0	5.0	95-50-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,2-Dichloroethane	0.4	5.0	107-06-2	0.4	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,2-Dichloropropane	1.0	5.0	78-87-5	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,3,5-Trichlorobenzene	1.0	5.0	108-70-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,3,5-Trimethylbenzene	1.0	5.0	108-67-8	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,3-Butadiene	1.0	5.0	106-99-0	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,3-Dichlorobenzene	1.0	5.0	541-73-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,3-Dichloropropane	1.0	5.0	142-28-9	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,4-Dichlorobenzene	1.0	5.0	106-46-7	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
1,4-Dioxane	125.0	125.0	123-91-1	NA	125.0	NA	250.0	125.0	500.0	250.0	1250.0	500.0	2500.0	1250.0	5000.0	2500.0	5000.0
2,2,4-Trimethylpentane	1.0	5.0	540-84-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
2,2-Dichloropropane	1.0	5.0	594-20-7	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
2-Butanone	1.0	5.0	78-93-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
2-Chloroethylvinyl ether	5.0	5.0	110-75-8	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
2-Chlorotoluene	1.0	5.0	95-49-8	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
2-Hexanone	1.0	5.0	591-78-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
2-Nitropropane	5.0	5.0	79-46-9	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
2-Propanol	125.0	125.0	67-63-0	NA	125.0	NA	250.0	125.0	500.0	250.0	1250.0	500.0	2500.0	1250.0	5000.0	2500.0	5000.0
4-Chlorotoluene	1.0	5.0	106-43-4	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
4-Isopropyltoluene	1.0	5.0	75-09-2	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
4-Methyl-2-pentanone	1.0	5.0	108-10-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Acetaldehyde	5.0	5.0	75-07-0	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Acetone	1.0	5.0	67-64-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Acetonitrile	1.0	25.0	75-05-8	NA	25.0	1.0	50.0	5.0	100.0	10.0	250.0	20.0	500.0	50.0	1000.0	100.0	200.0
Acrolein	1.0	25.0	107-02-8	NA	25.0	1.0	50.0	5.0	100.0	10.0	250.0	20.0	500.0	50.0	1000.0	100.0	200.0
Acrylonitrile	1.0	5.0	107-13-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Allyl Chloride	1.0	5.0	107-05-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Benzene	0.4	5.0	71-43-2	0.4	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Benzyl chloride	1.0	5.0	100-44-7	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0

Analyte	PQL water (µg/L)	PQL Soil (µg/L)	CAS No.	Std 1 µg/L	Std 1 µg/L	Std 2 µg/L	Std 2 µg/L	Std 3 µg/L	Std 3 µg/L	Std 4 µg/L	Std 4 µg/L	Std 5 µg/L	Std 5 µg/L	Std 6 µg/L	Std 6 µg/L	Std 7 µg/L	Std 8 µg/L
Bromobenzene	1.0	5.0	108-86-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Bromochloromethane	1.0	5.0	74-97-5	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Bromodichloromethane	1.0	5.0	75-27-4	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Bromoform	1.0	5.0	75-25-2	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Bromomethane	1.0	5.0	74-83-9	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Carbon disulfide	0.4	5.0	75-15-0	0.4	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Carbon Tetrachloride	1.0	5.0	56-23-5	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Chlorobenzene	1.0	5.0	108-90-7	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Chlorodifluoromethane	1.0	5.0	75-45-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Chloroethane	1.0	5.0	75-00-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Chloroform	1.0	5.0	67-66-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Chloromethane	1.0	5.0	74-87-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Chloroprene	1.0	5.0	126-99-8	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Cis-1,2-Dichloroethene	1.0	5.0	156-59-2	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
cis-1,3-Dichloropropane	1.0	5.0	10061-01-5	NA	5.0	1.0	10.0	0.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
cis-Decahydronaphthalene	1.0	5.0	493-01-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Cyclohexane	1.0	5.0	110-82-7	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Decane	5.0	5.0	124-18-5	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Dibromochloromethane	1.0	5.0	124-48-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Dibromomethane	1.0	5.0	74-95-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Dichlorodifluoromethane	1.0	5.0	75-71-8	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Diethyl ether	1.0	5.0	60-29-7	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Diisopropyl ether	1.0	5.0	108-20-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
D-Limonene	5.0	5.0	5989-27-5	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Dodecane	5.0	5.0	112-40-3	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Ethanol	125.0	125.0	64-17-5	NA	125.0	NA	250.0	125.0	500.0	250.0	1250.0	500.0	2500.0	1250.0	5000.0	2500.0	5000.0
Ethyl acetate	1.0	5.0	141-78-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Ethyl Methacrylate	1.0	5.0	97-63-2	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Ethyl tert-butyl ether	1.0	5.0	637-92-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Ethylbenzene	1.0	5.0	100-41-4	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Heptane	1.0	5.0	142-82-5	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Hexachlorobutadiene	1.0	5.0	87-68-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Hexane	1.0	5.0	110-54-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Iodomethane	1.0	5.0	74-88-4	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Isobutyl alcohol	25.0	25.0	78-83-1	NA	20.0	NA	50.0	25.0	100.0	50.0	250.0	100.0	500.0	250.0	1000.0	500.0	1000.0
Isopropylbenzene	1.0	5.0	99-87-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
m,p-xylene	2.0	10.0	108-38-3/ 106-42-3	NA	10.0	2.0	20.0	20.0	40.0	20.0	100.0	40.0	200.0	100.0	400.0	200.0	400.0
Methacrylonitrile	1.0	5.0	126-98-7	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Methyl Acetate	1.0	5.0	79-20-9	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Methyl isothiocyanate	1.0	5.0	556-61-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Methyl Methacrylate	1.0	5.0	80-62-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Methyl tert-butyl ether	1.0	5.0	1634-04-4	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Methylcyclohexane	1.0	5.0	108-87-2	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Methylene Chloride	1.0	5.0	75-09-2	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Naphthalene	1.0	5.0	91-20-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
n-butyl acetate	5.0	5.0	123-86-4	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
n-Butylbenzene	1.0	5.0	104-51-8	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Nonane	5.0	5.0	111-84-2	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
n-Propylbenzene	1.0	5.0	103-65-1	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0

Analyte	PQL water (µg/L)	PQL Soil (µg/L)	CAS No.	Std 1 µg/L	Std 1 µg/L	Std 2 µg/L	Std 2 µg/L	Std 3 µg/L	Std 3 µg/L	Std 4 µg/L	Std 4 µg/L	Std 5 µg/L	Std 5 µg/L	Std 6 µg/L	Std 6 µg/L	Std 7 µg/L	Std 8 µg/L
Octane	5.0	5.0	111-65-9	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
o-Xylene	1.0	5.0	95-42-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
p-Diethylbenzene	1.0	5.0	105-05-5	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Pentachloroethane	1.0	5.0	76-01-7	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
p-Ethyltoluene	1.0	5.0	622-96-8	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Propionitrile	5.0	5.0	111-84-2	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Sec-Butylbenzene	1.0	5.0	135-98-8	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Styrene	1.0	5.0	100-42-5	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
tert Amyl methyl ether	1.0	5.0	994-05-8	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
tert-amyl alcohol	5.0	5.0	75-85-4	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
tert-amyl ethyl ether	1.0	5.0	919-84-8	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
tert-Butyl Alcohol	25.0	25.0	75-65-0	NA	25.0	NA	50.0	25.0	100.0	50.0	250.0	100.0	500.0	250.0	1000.0	500.0	1000.0
Tert-Butylbenzene	1.0	5.0	98-06-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Tetrachloroethene	1.0	5.0	127-18-4	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Tetrahydrofuran	1.0	5.0	109-99-9	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Toluene	1.0	5.0	108-88-3	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Trans-1,2-Dichloroethene	1.0	5.0	156-60-5	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Trans-1,3-Dichloropropane	0.4	5.0	10061-02-6	0.4	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Trans-1,4-Dichloro-2-butene	1.0	5.0	110-57-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
trans-Decahydronaphthalene	1.0	5.0	493-02-7	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Trichloroethene	1.0	5.0	79-01-6	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Trichlorofluoromethane	1.0	5.0	75-69-4	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Undecane	5.0	5.0	1120-21-4	NA	5.0	NA	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Vinyl acetate	1.0	5.0	105-05-4	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Vinyl Chloride	0.4	5.0	75-01-4	0.4	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0
Xylene (total)	1.0	5.0	1330-20-7	NA	5.0	1.0	10.0	5.0	20.0	10.0	50.0	20.0	100.0	50.0	200.0	100.0	200.0

* Additional analytes can be added as per client and state requirements

11.2.2. An analyte must be present and calibration curve in control in order to be reported on the target analyte list. Analytes identified by mass spectral match but not present and in control in the calibration table may be reported as Tentatively Identified Compounds (TICs). Guidelines for identification are listed in Section 14.5. Results for these TICs should be reported only on a present/absent basis. However, quantitative results may be reported provided they are qualified as estimated values.

11.2.3. Calibration Response Factors: Response factors (RF) establish the relationship of the instruments response in comparison with the concentration of any given analyte. The RF includes the concentration and response of the internal standard as well. By relating the IS concentration and response in an inverse manner, the target analyte concentration is adjusted to account for drift in the instrument on a per injection basis. As instrument response increases as indicated by the response of the internal standard, the concentration of the target is mathematically decreased, and vice versa.

11.2.4. To calculate the RF for any given calibration standard (or calibration verification standard), tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured, see Attachment III and IV. Response factors are calculated using the following equation:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (µg/L).
C_x = Concentration of the compound being measured (µg/L).

11.2.5. **Calibration Curve Fit**: The calibration curve is a representation of the relationship of the instrument response and analyte concentration. The curve is used to quantitate the concentration of an unknown based on its response and this known relationship. The curve is produced in several ways depending on the nature of the "goodness of fit".

11.2.6. **Average Response Factor (ARF)**: The average response factor is determined by averaging the response factors calculated for each calibration level for each target analyte. The average RF can be used to calculate the concentration of target analytes in samples provided the criteria are met for consistency in the RFs for any given analyte. An average response factor is the default curve fitting option for calibrations. It is in the most basic sense, a linear regression that is forced through zero at the origin. Because of its simplicity and the interception of the y-axis at the origin, this is the preferred technique for curve fitting. A calculation of the percent relative standard deviation (%RSD) is used to determine the acceptability of the use of the ARF:

$$\%RSD = SD*100 / ARF$$

Where: SD = Standard deviation of the averaged RFs for a given compound

§ The RSD should be less than or equal to 20% for each target analyte. If this criterion is met then the average relative response factor may be used for quantitation.

§ If the RSD of any target analyte is greater than 20%, evaluate curve fit using additional calibration options as below:

11.2.7. **Linear Regression**: The 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+b where "a" is the slope of the line and "b" is the y intercept. In order to use this curve fit technique, a minimum of 5 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument is linear in nature but does not necessarily intercept the y-axis at the origin. However, because the 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. A calculation of the correlation coefficient "r" is used to determine the acceptability of a linear regressed curve. See Table 11.2 for acceptance criteria.

§ When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration.

11.2.8. **Non-linear Regression**: 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. This technique works well for calibrations where the response of the instrument gradually decreases with increasing

concentrations. Using this technique, an analyst may be able to generate calibration curves with correlation coefficients very close or equivalent to 1.000. However, 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. Likewise, high levels of contamination may not be able to be calculated due to regression equations with multiple intercepts of either axis on the calibration plot. See Table 11.2 for acceptance criteria.

11.2.9. Either the low or high calibration points may be dropped to meet linearity criteria provided the laboratory meets the minimum 5 calibration point requirements. Points within the center of the curve may not be dropped unless an obvious problem is discovered and documented. The point must be dropped in its entirety and reanalyzed. Re-analysis should be within the same 12-hour time window and must occur within 8 hours of the original analysis.

11.2.10. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet these criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes. The analyst should also strive to place more emphasis on meeting the calibration criteria for those compounds that are critical project compounds, rather than meeting the criteria for those less important compounds.

11.2.11. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too imprecise for analysis to begin and corrective action should be taken.

11.3. **Calibration Verification**:

11.3.1. **Second Source Verification**: In addition to meeting the linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. Accuracy is a function of both the “fit” of the curve to the points used and the accuracy of the standards used to generate the calibration points. By meeting the fit criteria, the accuracy relative to the goodness of fit is addressed. However, because all calibration points are from the same source, it is possible that the calibration points may meet linearity criteria but not be accurately made in terms of their true value.

11.3.2. Therefore, to assess the accuracy relative to the purity of the standards, a single standard from a secondary source must be analyzed and the results obtained must be assessed relative to the known true value. This step is referred to as ***Secondary Source Verification*** or, alternatively as ***Initial Calibration Verification***. This secondary source 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 difference from the true value according to the following equation:

$$\% \text{ Difference} = [\text{Result}_{SCV} - \text{TrueValue}_{SCV}] / \text{TrueValue}_{SCV} * 100$$

11.3.3. **Continuing Calibration Verification (CCV):** As part of the analytical process, the instrumentation must be checked periodically to determine if the response has changed significantly since the initial calibration was established. This verification process is known as ***Continuing Calibration Verification***. The validity of the initial calibration is checked at the beginning of every analytical sequence and every 12 hours thereafter for as long as the instrument is analyzing samples and is accomplished by analyzing a midpoint calibration standard (CCV).

11.3.4. The values obtained from the analysis of the CCV are compared to the true values and a percent change calculated. The percent change must meet the method specified criteria for the analysis to proceed for an additional 12 hours.

11.3.5. The actual determination of change in instrument response is based on the type of curve fit used for each analyte. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the CCV from the average RF established in the initial calibration. Calibration curves based on a linear or quadratic regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

$$\% \text{ Difference: } [RF_{CCV} - AvgRF_{CAL}] / AvgRF_{CALI} \times 100$$

$$\% \text{ Drift: } [Result_{CCV} - TrueValue_{CCV}] / TrueValue_{CCV} \times 100$$

11.2 Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter/Frequency	Criteria	Comments/ Corrective Actions
Initial Calibration (ICAL)	Average Response Factor Linear Regression Quadratic Regression	%RSD ≤ 20% $r^2 \geq 0.99$ $r^2 \geq 0.99$ Six point data curve, RSDs above 20%	If not met, try linear regression fit If not met, try Quadratic Regression (six point only) If not adjust moisture control parameters, replace analytical trap or column, replace moisture trap or adjust desorb time, and/or remake standards and recalibrate
	Minimum RFs must be obtained for certain analytes	See Attachment V for 8260C criteria See Attachment IX and X for ASP criteria	
	Relative retention time (RRT)	Within 0.06 for all calibration standards	
	Establish initially and as required. When continuing calibration check standards fail. When a change in procedure or instrument.		Evaluate the standards and equipment and reanalyze the initial calibration.
Initial Calibration Verification Standard (ICV) / Second Source Verification Standard	Immediately after the initial calibration. Prepared from a source different than the calibration standards.	70%-130% (Alternative acceptance limits may be appropriate based on the desired project-specific data quality objectives.)	If the requirements fail, reanalyze and recalibrate if necessary. Sample analysis cannot begin until a compliant Initial Calibration and ICV meet acceptance criteria. Samples analyzed after a non-compliant ICV must be reanalyzed.

Continuing Calibration Verification (CCV)	One per 20 samples and every 12 hours thereafter	80-120% %D cannot exceed 20% Acceptable criteria compounds: <20% for 80% of compounds	If the requirements for continuing calibration are not met, reanalyze. If a re-analysis of a CCV is still non-compliant, the standards preparation and equipment must be evaluated, prepare new standards if necessary and recalibrate. In cases where <20% of compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values.
		% drift ± 20%	Only for linear and non-linear calibration curves
	Internal Standard Response	50 – 200%	Use midpoint calibration standard as reference
	Internal Standard RT	RT ± 10 sec	Samples with non-compliant areas must be reanalyzed to demonstrate matrix interference
	Work required for ASP criteria	Quantitate using daily CCV (ASP 2000 only) See Attachment IX and X for ASP criteria	If the requirements for continuing calibration are not met, reanalyze. Note: Work requiring 8260C and ASP 2005 criteria quantitate using the initial calibration

12. PROCEDURE

12.1. **Operating Parameters:** Configure the GC/MS system to match the following operating parameters based on instrument configuration. The parameters themselves are saved as a method on the chromatography data system. By loading the last method used, the instrument will auto-configure to match the parameters from the last time the system was operated under that method. Verify that the settings in the software match the appropriate configuration. Operating conditions are variable to allow for optimum analytical results. Each instrument has specific conditions that give optimal results. The following are the typical ranges of analytical conditions:

12.2. **Table 12.1: Instruments and Operating Parameters**

Instrument IDs	Component	Settings and Consumables	
HP5971	Gas Chromatograph	**Waters**	**Soils**

<table>
<tr><th>Instrument IDs</th><th>Component</th><th colspan="6">Settings and Consumables</th></tr>
<tr><td></td><td></td><td colspan="3">Column: RTX-624
Carrier: Helium
Initial Temperature:45ºC
Initial Hold: 1 min
Ramp 1: 20ºC/min to 220ºC
Hold 1: 1.0 min
Injector Temp: 180ºC
Detector temp: 250ºC</td><td colspan="3">Column: RTX-624
Carrier: Helium
Initial Temperature:45ºC
Initial Hold: 1 min
Ramp 1: 20ºC/min to 220ºC
Hold 1: 1.0 min
Injector Temp: 180ºC
Detector temp: 250ºC</td></tr>
<tr><td></td><td>Mass Spectrometer</td><td colspan="3">Tune File: BFB.U
Separator Temp: 220ºC
Manifold Temp: 250ºC
Mass Range: 35 to 300
Scan Time: 0.38 sec
Number of scans: 3220
Threshold: 50
Minimum peak area: 500</td><td colspan="3">Tune File: BFB.U
Separator Temp: 220ºC
Manifold Temp: 250ºC
Mass Range: 35 to 300
Scan Time: 0.38 sec
Number of scans: 3220
Threshold: 50
Minimum peak area: 500</td></tr>
<tr><td>Notes: For Fuel Oxygenates it is recommended to purge at 80ºC.

*Recommended program; modify as needed to optimize</td><td>Purge & Trap Concentrator</td><td colspan="2">Sample: 5mL
Purge: 7 min
Purge Flow: 60mL/min
Purge Temp: Ambient
Dry Purge: 0.5 min
Desorb: 1.0 min
Desorb Temp: 250ºC
Bake: 6 mins</td><td colspan="2">Fuel Oxygenates
Sample: 5mL
Purge: 7 min
Purge Flow: 60mL/min
Purge Temp: 75ºC*
Dry Purge: 0.5 min
Desorb: 1.0 min
Desorb Temp: 250ºC
Bake: 6 mins</td><td colspan="2">Sample: 5mL
Purge: 7 min
Purge Flow: 60mL/min
Purge Temp: 40ºC
Dry Purge: 0.5 min
Desorb: 1.0 min
Desorb Temp: 250ºC
Bake: 6 mins</td></tr>
<tr><td></td><td>Autosampler</td><td colspan="2">Method: 1
Sample volume: 5mL
Rinse volume: 7mL
Rinses: 1
Standard bulb #: 1 or 2</td><td colspan="2">Method: 5
Sample volume: 5mL
Rinse volume: 7mL
Rinses: 1
Standard bulb #: 1 or 2</td><td colspan="2">Method: 2
Sample volume: 10mL
Rinse volume: 7mL
Rinses: 1
Standard bulb #: 1 or 2</td></tr>
</table>

12.2.1. **Tune Verification**: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer tune conditions must be verified. The tune is verified by analyzing a standard containing bromofluorobenzene (BFB). The tune verification standard can be combined with the CCV standard provided that the amount of BFB introduced into the system meets the method criteria.

12.2.2. The mass spectrum of BFB must be evaluated against the following criteria:

Mass (m/z)	Ion Abundance criteria
50	15.0-40.0% of m/z 95
75	30.0-60.0% of m/z 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of m/z 95
173	Less than 2.0% of m/z 174
174	>50% of m/z 95
175	5.0-9.0% of m/z 174
176	95.0-101.0% of m/z 174
177	5.0-9.0% of m/z 176

Note: All ion abundances must be normalized to m/z 95, the normal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

12.2.3. To evaluate the tune spectra, following the operating instructions for the chromatography data system to access the data file and obtain mass spectra for bromofluorobenzene. If the software has a program or macro for automatically selecting the spectra and evaluating the response ratios, use this option. Otherwise, the spectra must be obtained in one the following manners, in the listed order:

§ Using an average of three scans, centered on the apex of the peak; or,

§ Using an average of all scans across the width of the peak, taken at half height; or,

§ Using an average of all scans taken across the width of the peak from baseline to baseline.

12.2.4. Once obtained, evaluate the ion ratios against the criteria listed above. If the ratios meet the criteria, then analysis may proceed for 12 hours. The window for analysis is 12 hours from the injection date/time for the BFB tune verification. After that, the tune must be verified again to establish a new analytical window. The same ion abundance criteria used for the BFB tune coupled with the initial calibration must be used for all subsequent analyses associated with that initial calibration.

12.2.5. If the ratios do not meet the criteria, refer to the following corrective actions to address the problem:

§ Re-inject the BFB tune

§ If that fails again, adjust your mass spectrometers parameters.

12.3. **Calibration Verification**: After the instrument tune conditions are verified and the system meets tune criteria, the instrument must undergo calibration verification. If it has already been determined that the instrument needs to be recalibrated, follow the procedures listed in section 11.2.1 (Analysis of Standards). Otherwise, analyze a Continuing Calibration Verification Standard to determine the current calibration status.

12.4. If the CCV meets control criteria, the system is deemed to be in control and analysis of samples may commence. If the CCV does not meet control criteria, follow the corrective action in Table 11.2 (Calibration Acceptance and Verification Criteria.) If the tune verification has been combined with the CCV, the 12 hour analysis window begins from the analysis date / time of the CCV.

12.5. **Sample Preparation- Water Samples**: Water samples typically do not require any sample preparation unless they require a dilution to bring high level contaminants within calibration range or to minimize matrix interference. Refer to 5030C SOP.

12.5.1. Allow the samples to come to room temperature before analysis.

12.5.2. After analysis check the pH for each sample and standard analyzed. The pH should be <2; document results in the logbook. Record all failures in LIMS, and on checklist. Inspect the spent sample vials to ensure that each vial was sampled properly. If dilutions are required, pH preservation can be verified at the time the dilution is made using the sample remaining in the original sample container.

12.6. **Sample Preparation- Oxygenated analyte analysis:**

12.6.1. Oxygenated analytes like alcohols with high solubility have to be purged at an elevated temperature.

12.6.2. If these analytes are to be analyzed the pH should be adjusted ≥ 10 by addition of trisodium phosphate dodecahydrate (TSP).

12.7. **Sample Preparation- Low Concentration Soil Samples**: If sample is received in bulk proceed as follows, otherwise refer to 5035A SOP.

12.7.1. Weigh 5.0g of sample in a 40mL vial to the nearest 0.01g and record in the logbook.

12.7.2. Prepare method blank and LFB with 5.0g of Ottawa sand (or equivalent; record lot number in logbook). Add 5mL of the LFB solution, (prepared as water procedure in a 100mL volumetric flask) to additional portions of soil for MS and MSD.

12.7.3. Add 5mL of water and stirring bar to each sample and method blank. Add 5mL of LFB solution and stirring bar to each QC samples. Before purging the autosampler automatically is adding 10mL of reagent water and internal standard and surrogate spike as in Table 13.2.

12.8. **Sample Preparation- Medium level soil samples:** If sample is received in bulk proceed as follows, otherwise refer to 5035A SOP.

12.8.1. Weigh a 4.0g representative aliquot of sample to the nearest 0.01 g in a 40mL. Record the weight. (Percent moisture will be determined separately in the Inorganic department.) Immediately add 10mL methanol, cap, and shake for two minutes.

12.8.2. After the solids have settled, transfer an aliquot of the supernate between 100µL and 1000µL and add to 50mL reagent water in a volumetric flask. Pour solution into a 40mL vial with no headspace and load on autosampler.

12.9. Samples received in bulk jars must be qualified on the final report as biased low due to not being collected according to 5035A sampling procedures.

12.10. **Dilutions**

12.10.1. **Waters**- Dilutions on aqueous samples must be prepared in a volumetric fashion. Sample aliquots may be measured in either a volumetric pipette or syringe and brought to volume in a volumetric flask.

12.10.2. **Soils- a)** If the concentration of any target compound in any sample exceeds the initial calibration range, a smaller sample size must be analyzed. If the sample was collected in a jar, a smaller aliquot may be analyzed (2.5g, 1.0g, and 0.5g). In this case, follow the same guidelines as for the 5.0g analysis. If the sample was collected in a jar and a medium level soil analysis is required. Measure 4.0g into a 40mL vial, add 10mL of methanol and allow the analytes to extract for a minimum of 15 minutes. **b)** If the sample was collected by Method 5035A, follow the same guidelines as preparation of a medium level soil.

12.11. Batch Quality Control- Refer to Table 13.1 for details on Batch QC requirements.

12.11.1. Method Blank- Fill 40mL vial preserved with 1:1 HCl, headspace free, with DI water for the method blank, and cap with septum caps. See Table 7.2.

12.11.2. Lab Fortified Blank (LFB) – Prepare lab fortified blank (LFB) solutions in 100mL volumetric flasks. To achieve a concentration of 50 µg/L. Refer to Table 10.1 and Table 10.4 for proper stock standards and amounts to use. Prior to analysis preserve with 1:1 HCl.

12.11.3. MS/MSD Samples- For water samples, refer to Table 10.1 and Table 10.4 for proper stock standards and amounts to use (same as LFB). For soil samples use LFB solution. Refer to section 12.9.2 for proper preparation of the LFB.

12.12. Sample Analysis-

12.12.1. **Water**

§ Equilibrate samples at room temperature.

§ Load CCV, Method blank, LFB vials than 40mL sample vials onto the autosampler. Record the sequence in logbook. Record the container analyzed in the analytical run log. This information can be found on the sample label. The bottle analyzed must also be entered into the LIMS (using the drop down for Container ID).

§ Fill surrogate spiking solution and internal standard solution into the reservoir and start the auto-sequencing program of the instrument.

§ Set-up the sequence and instrument as described above (Table 12.1), and start the auto processing of the samples.

§ In this process the instrument sequentially draws the programmed amount of 5mL of each vial and spikes it with an approximate volume of 1µL of the surrogate standard solution. The 5mL aliquot is purged for the programmed time, and the compounds, released into the headspace, are transferred to the trap, where they are collected on the sorbent material.

§ After desorbing from the trap, the compounds are segregated on a fused silica capillary with a temperature program to optimize resolution. The compounds are eluted into the masspectrometer, where the molecules are broken into ions in the electron impact source.

§ The signals and spectra of the analyses are collected on the data system with the Enviroquant software and processed to LIMS software.

§ The pH of the samples is checked after the analysis is complete. Record pH in logbook.

12.12.2. **Soil**

§ Following the preparation of soils in section 12.7 and 12.8. Refer to section 12.11.1 for procedure.

13. QUALITY CONTROL

13.1. **Table 13.1 – Batch Quality Control Criteria**

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Initial Demonstration of Capability (IDC)	Method specified compounds: Full Target List compounds	Per analyst; Before any samples are analyzed Analyze four replicates of a LFB	70%-130% *NOTE*: Alternative acceptance limits may be appropriate	If requirements are not met evaluate standard preparation. Reanalyze and recalibrate if necessary.

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
MDL	Method specified compounds: Full Target List compounds	Annually Minimum 7 low level LFBs analyses over several days	40CFR Part 136 Appendix B	Evaluate, change spike amount, repeat study
Method Blank (MB)	Reagent water	One per batch Or one per 20 samples or 12 hour window (whichever is most frequent) Processed with and under the same conditions as samples and goes through all the steps of the analytical procedure.	Should not contain target analytes or interferences at concentrations that impact the analytical results for sample analyses. Target analytes should not be greater than PQL.	If the blank contains target analytes greater than the PQL, the blank needs to be re-analyzed. If the re-analysis passes criteria, then reanalyze the associated samples. If the blank still fails, the system needs to be evaluated for the source of contamination and affected samples re-analyzed. If re-analysis of samples is not possible, report data flagged to indicate method blank contamination.
Lab Fortified Blank (LFB)	Method specified compounds: Full Target List compounds _NOTE_: LFBs are processed in the same manner as the samples.	One per batch, Or 1 per 20 samples, whichever is more frequent. Prepared from the same source as the calibration standards. (different from the ICV)	In house limits (see LIMS for complete list) *Note: Acceptable limits 70%-130% if no in-house limits are created*	Reanalyze If a re-analysis of a LFB is still non-compliant, the standards preparation and equipment must be evaluated, prepare new Re-analyze any positive samples in the batch after re-calibration.
Matrix Spike (MS)	Method specified compounds: Full Target List compounds	One per 20 samples or daily, if fewer than 20 samples. _NOTE_: The MS sample (and MSD) has to be analyzed concurrently with the parent sample.	In-house limits	Check for errors in calculation and spike preparation. If the matrix spike still exceeds the limits, but the LFB has acceptable recovery, then the method is in control and sample matrix effects are likely the cause. The data should be qualified in the case narrative or using QC notes in the LIMS for non-package work.

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
MSD / Duplicate	MS Duplicate *OR (alternative)* Sample Duplicate	One per 20 samples or daily, if fewer than 20 samples.	± 30% RPD For result values less than five times the PQL, a control limit of ± the PQL will be used.	Check sample label, calculation, and dilution factors. If results are grossly different (i.e., very high result and non-detect) re-analyze to confirm. Report results with an appropriate footnote.
MSB	Method specified compounds	Only for ASP criteria work When client provides a MS and MSD	See Attachment VII	Reanalyze If a re-analysis of a MSB is still non-compliant, the standards preparation and equipment must be evaluated, prepare new

13.2. **Table 13.2 – Sample Quality Control Criteria**

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Internal Standard	Pentafluorobenzene 1,4-difluorobenzene chlorobenzene-d5 1,4-dichlorobenzene-d4 Bromochloromethane*	Added to all standards, samples, spikes, control samples, and method blanks prior to analysis	Areas must be between –50% and +100% and retention times within 0.5 minutes. Use midpoint calibration standard as reference. Internal standard working solution is at 200µg/mL	If the criteria are not meet, in any of the samples, re-analysis is required demonstrate matrix interference. If the criteria fails in any standard or method blank, than all the samples following the failed standard needs to be re-analyzed.
Surrogate Standards ** ***	1,2-Dichloroethane-d4 4-Bromofluorobenzene Toluene-d8	Added to all samples, spikes, control samples and method blanks prior to analysis	In-house limits Spiked at 50 µg/L. *See Attachment VI	If % recoveries are outside the range, re-analysis is required, unless no positives are found and surrogate recovery was high. If the sample re-analysis also fails the recovery criteria, report all data for the sample as "suspect".

* Bromochloromethane, 1,4-difluorobenzene and chlorobenzene-d5 (3 internals) are used together for ASP and DEC work. Pentafluorobenzene, 1,4-difluorobenzene, chlorobenzene-d5 and 1,4-dichlorobenzene-d4 (4 internals) are used for 8260C criteria work.

**For method 8260C, it is no longer necessary to include the surrogate compounds in the calibration solution to obtain multipoint calibrations. Instead, the surrogates are automatically spiked into the calibration analyses the same as the samples at 50µg/L. The response factors are averaged for a one-point calibration at medium level.

***However, for ASP requirements you must include the surrogate compounds in the calibration solution to obtain multipoint calibrations. See Attachment VI, VII, IX and X for criteria.

13.3. No re-injections are necessary for the sample used for MS spiking, if the surrogate recoveries of the MS also fail the requirements. Also, associated samples i.e. of the same matrix, that show the same recovery pattern, need not be repeated. Discuss the similar recoveries in the narrative.

13.4. According to NELAC, in order for the method blank not to be compliant, the concentration of the contaminant has to be > 1 times the PQL and **also** has to be larger than $1/10^{th}$ of the lowest sample. Therefore if the sample contains concentrations that are all at least 10 times higher than the blank, the data do not have to be reanalyzed or flagged with the 'B' qualifier.

14. Data Analysis and Calculations

14.1. Qualitative Analysis

14.1.1. **Retention Time Comparison:** The relative retention time (RRT) of the sample component must be within ±0.06 RRT units of the component in the calibration verification standard. Extracted Ion Current Plots (EICPs) may be used to provide a more reliable assignment of RT in the presence of coeluting components.

14.1.2. **Mass Spectrum Comparison:** All ions in the standard mass spectrum of a relative intensity greater than 10 % of the most abundant ion must be present, and the relative intensities of the ions must agree within ± 20 %. The ions greater than 10 % in the sample but not in the standard spectrum must be accounted for. Compounds are identified as present when the following criteria are met:

- § The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.
- § The relative intensities of the characteristic ions agree within 20% of the relative intensities of these ions in the reference spectrum.
- § Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times.

14.2. Quantitative Analysis- Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique.

14.2.1. **Raw Data Results:** The GC/MS data system will calculate the concentration of each analyte as µg/L (or ng/mL). For water samples, no further calculations are necessary unless a dilution of the sample has been performed. If the initial analysis of the sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be analyzed at a higher dilution. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

14.3. **Results Calculation- Aqueous Samples:**

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(I_s)(D)}{(A_{is})(RF)(V_o)}$$

Where:

A_x= Area of characteristic ion for compound being measured.
I_s = Amount of internal standard injected (ng).
A_{is} = Area of characteristic ion for the internal standard.
RF = Average Relative Response factor for compound being measured.

V_o = Volume of water purged (mL), taking into consideration any dilutions made
D= Dilution factor if the sample or extract was diluted prior to analysis. If no dilution was made, then D=1. The dilution factor is always dimensionless.

14.4. **Results Calculation- Soil/Solid Samples:**

$$\mathit{High}\,\text{Conc. (ug/kg)} = \frac{(A_x)(I_s)(V_t)(D)}{(A_{is})(RF)(V_i)(W_s)}$$

$$\mathit{Low}\,\text{Conc. (ug/kg)} = \frac{(A_x)(I_s)(D)}{(A_{is})(RF)(W_s)}$$

Where:

A_x, I_s, A_{is}, RF = Same as in water and water-miscible waste listed above.
V_t = Volume of total extract (mL). For purge-and-trap analysis where an aliquot of a solvent (methanol, water, etc.) extract is added to reagent water and purged, V_t is the total volume of the solvent extract. This also includes any contribution from water present in samples prior to solvent extraction - See 14.4.1.
V_i = Volume of extract added (mL) for purging.
W_s= Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data
D=Dilution Factor, if the sample or extract was diluted prior to analysis. If no dilution was made, then D=1. The dilution factor is always dimensionless.

14.4.1. Solid samples with a significant moisture content (>10) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture determination. This total volume is then expressed as V_t in the sample concentration calculations. Therefore, the calculated concentration needs to be corrected using the total solvent/water mixture volume represented as V_t. This total solvent/water volume is calculated as follows:

$$mL\ solvent / water V_t = \frac{mL\, of\ solvent + (\%\, moisture \times g\ of\ sample)}{100} \times 1000\, mL / mL$$

14.5. **Tentatively Identified Compounds (TICs)-** For some samples, identification may be desired for non-target compounds. Search up to 20 TICs. In the retention time window from 30 sec before the earliest targeted analyte and 3 min after the latest targeted analyte select the largest 20 peaks that are not identified as spiked compounds, targeted analytes. Total areas of the peaks have to be larger than 10% of the closest internal standards. A mass spectral library search may be conducted to attempt assignment of tentative identifications. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications.

14.5.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum;

14.5.2. The relative intensities of the major ions should agree within ± 20%;

14.5.3. Molecular ions present in the reference spectrum should be present in the sample spectrum;

14.5.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds;

14.5.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies

15. Data assessment and Acceptance Criteria for Quality Control Measures

15.1. The analyst is responsible for generating the data and also is the initial individual to review the data. The review must include at least the following procedures (where applicable):

15.1.1. Inspection of records in run log for completeness;

§ Standard and reagent lot numbers, support equipment, spike amounts, calculations, dilution factors, container/bottle used for analysis, reporting limits.

15.1.2. Determination of whether the results meet the laboratory-specific quality control criteria;

15.1.3. Checks to determine consistency with client/project-specific measurement quality objectives (MQOs) if such exists;

15.1.4. Checks to ensure that the appropriate sample preparatory and analytical SOPs and methods were followed, and that chain-of-custody and holding time requirements were met;

15.1.5. Checks to ensure that all calibration and quality control requirements were met;

15.1.6. Checks for complete and accurate explanations of anomalous results, corrective action, and the use of data qualifiers in the case narrative or LIMS QC notes.

15.1.7. Record of any non-standard condition of the test, test environment, sample or any deviation from standard operating procedure.

15.2. If analysis is deemed acceptable, data will be imported into the LIMS.

15.2.1. Another review is performed for correctness of results, including prep factors, dilution factors, spike amounts and recoveries, sample and QC references and appropriate qualifiers.

15.2.2. If additional information is to be communicated to the data user about a particular sample, a “QC Note” is entered by the analyst.

15.2.3. Once data has been reviewed in the LIMS, the analyst or supervisor will “QA” the sequence which indicates the data has been reviewed and is ready for reporting.

15.3. Refer to Tables 11.2, 13.1, and 13.2 for data assessment and acceptance criteria for quality control measures.

15.4. Once it has been established that the quality objectives are met, the finalized data are entered into the LIMS and may be reported.

16. Corrective Actions for Out-of-Control Data

16.1. Corrective action begins with the analysts who are responsible for knowing when the analytical process is out of control.

16.2. Corrective actions should be initiated for out of control events such as when QC checks exceed acceptance limits or exhibit trending, holding time failures, loss of sample, equipment malfunctions or evidence of sample contamination.

16.3. Refer to Tables 11.2, 13.1 and 13.2 for data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1. Refer to Tables 11.2, 13.1 and 13.2 for data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

17.2. All problems associated with the analysis of a sample group should be outlined in the case narrative of the data package or with the use of QC Notes in the LIMS, if required.

18. METHOD PERFORMANCE

18.1. The suitability of the method for the analytes tested was determined when the method was developed. During “method startup” in the laboratory, the capability of the analysts, suitability of equipment, materials and instrumentation was tested.

18.2. Internal method performance is established and monitored with use of the following (where applicable):

18.2.1. Method Detection Limit studies

18.2.2. Demonstration of Capability

§ Every analyst who performs this method must first document acceptable accuracy and precision by passing an initial demonstration of capability study (IDC). See Table 13.1

18.2.3. Precision and accuracy

18.2.4. Positive and negative controls

18.2.5. Measurement of sample matrix effects

18.2.6. Quality Control Samples (Proficiency Testing)

19. INSTRUMENT/EQUIPMENT MAINTENANCE/TROUBLESHOOTING

19.1. For information and guidance related to instrument/equipment maintenance, troubleshooting and computer hardware and software refer to the following documents:

Document # (latest revision)	Document Title
QAM	*Quality Assurance Quality Control Manual*
ADMIN002	*Computers and Programs*
MS001	*Tekmar ALS 2016/Als2032 User Manual Installation Guide*
MS002	*Archon Purge And Trap Autosampler System Operator's Manual*
MS008	*HP 5971A Mass Selective Detector Hardware Manual*
MS010B	*HP 5890 Series II Gas Chromatograph Operating Manual 05890-90260*
MS017	*HP Analytical CD-ROM MSD Productivity Chemstation Software*
MS022	*HP 6890 GC/ALS Service Information March 1996 G1530-80160*
MS023	*Tekmar - Includes Various Tabs On Different Analysis For Use With Concentrator Systems*
MS042	*Hp 5970b Mass Select Detector Hardware Manual*
MS044	*HP 6890 Series Gas Chromatograph*

MS046	*Tekmar LSC 2000 User Manual*
MS048	*Tekmar 3000 User Manual*
MS050	*5972a Mass Selective Detector Hardware Manual*
MS059	*5973n MSD Maintenance Collection Reva00.00 November 1999*

19.2. Troubleshooting procedures also come from experience and training. Problems that the analyst encounters may be elevated to the department supervisor. Other means of problem resolution may come from review of prior related corrective action reports, instrument maintenance records, internal and external audits and lab notes.

20. SAFETY

20.1. **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.

20.1.1. Read information and follow warnings listed on the labels of the containers of the chemicals.

20.1.2. In addition, the reference file of material safety data sheets (MSDS) should be consulted for properties of the chemicals used, to determine handling precautions.

20.2. Safety glasses, a lab coat, and disposable gloves must be worn when handling chemicals and samples.

20.3. Sample handling should be conducted in fume hoods.

20.4. The company wide Chemical Hygiene and Safety Manual contains additional information on safety.

20.5. **Equipment:** Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones. The purge and trap concentrator and autosampler use gas under pressure to purge samples and, in some cases, drive the robotic assemblies. These high pressures introduce the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

21. WASTE MANAGEMENT

21.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste.

21.2. Refer to the lab's *Sample and Waste Management* SOP.

22. POLLUTION PREVENTION

22.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Use pollution prevention techniques in laboratory operation whenever feasible.

22.2. Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

22.3. Control releases from hood and bench operations and comply with discharge permits and regulations, hazardous waste identification rules, and land disposal restrictions.

22.4. The generated waste has to be disposed in a manner not to cause pollution.

22.5. All wastes deemed toxic are collected in the laboratory and are disposed by professional companies licensed in waste disposal.

22.6. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

23. REFERENCES

23.1. "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update III, Dec. 1996.

23.2. "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update IV, February 2007

23.3. "New York State Department of Environmental Protection Analytical Services Protocol," June 2000

23.4. "New York State Department of Environmental Protection Analytical Services Protocol," April 2005

23.5. National Environmental Laboratory Accreditation Conference (NELAC) Constitution, Bylaws and Standard, approved June 5, 2003, (EPA/600/R-04/003)

23.6. 40CFR Part 136 Appendix B

23.7. 2009 TNI Laboratory Accreditation Standards, EL-V1-2009.

24. TABLES AND FIGURES

24.1. Attachment I: CHARACTERISTIC IONS FOR VOLATILE TARGETED COMPOUNDS
24.2. Attachment II:CHARACTERISTIC IONS FOR SURROGATE AND INTERNAL STANDARDS FOR VOLATILE ORGANIC COMPOUNDS
24.3. Attachment III: VOLATILE INTERNAL STANDARDS ASSIGNED FOR QUANTITATION OF TARGETED COMPOUNDS AND SYSTEM MONITORING COMPOUNDS (4INTERNALS)
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24.7. Attachment VII: QC LIMITS FOR MATRIX SPIKE/SPIKE DUPLICATE AND MATRIX SPIKE BLANK
24.8. Attachment VIII: INSTRUMENT OPERATING PARAMETERS FOR FUEL OXYGENATES
24.9. Attachment IX: RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF VOLATILE ORGANIC COMPOUNDS FOR ASP 2000 CRITERIA.
24.10. Attachment X: RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF VOLATILE ORGANIC COMPOUNDS FOR ASP 2005 CRITERIA.
24.11. Attachment XI: FUEL OXYGENATE COMPOUNDS PLUS AROMATICS

25. REVISIONS

Document Number	Reason for Change	Date
S-LI-O-011-rev.00	Transition to PACE format. Added ASP information, methanol volume correction for moisture.	05-9-2015

Attachment I: CHARACTERISTIC IONS FOR VOLATILE TARGETED COMPOUNDS

Compound	Primary Ion*	Secondary Ion	Tertiary Ion	Compound	Primary Ion*	Secondary Ion	Tertiary Ion
1,1,1,2-Tetrachloroethane	131	133	119	cis-Decahydronaphthalene	138	96	67
1,1,1-Trichloroethane	97	99	61	Cyclohexane	56	84	41
1,1,2,2-Tetrachloroethane	83	131	85	Decane	43	57	142
1,1,2,2-Tetrafluoro-1,2-dichloroethane (Freon-114)	135	85	-	Dibromochloromethane	129	127	-
1,1,2-Dibromo-1,2,2-trifluoroethane (Freon-113)	101	151	103	Dibromomethane	93	95	174
1,1,2-Trichloroethane	83	97	85	Dichlorodifluoromethane	85	87	-
1,1-Dichloroethane	63	65	83	Diethyl ether	59	75	45
1,1-Dichloroethene	96	61	63	Diisopropyl ether	45	43	87
1,1-Dichloropropene	75	110	77	D-Limonene	136	69	93
1,2,3-Trichlorobenzene	180	182		Dodecane	43	57	170
1,2,3-Trichloropropane	110	39	77	Ethanol	45	-	-
1,2,3-Trimethylbenzene	105	120	77	Ethyl acetate	43	61	70
1,2,4,5-Tetramethylbenzene	119	134	91	Ethyl Methacrylate	69	41	99
1,2,4-Trichlorobenzene	180	182	-	Ethyl tert-butyl ether	59	87	57
1,2,4-Trimethylbenzene	105	120	-	Ethylbenzene	106	91	-
1,2-Dibromo-3-chloropropane	75	155	157	Heptane	43	57	100
1,2-Dibromoethane	107	109	188	Hexachlorobutadiene	225	260	-
1,2-Dichlorobenzene	146	111	148	Hexane	57	43	86
1,2-Dichloroethane	62	98	-	Iodomethane	142	127	-
1,2-Dichloropropane	63	112	-	Isobutyl alcohol	43	42	-
1,3,5-Trichlorobenzene	180	182	145	Isopropylbenzene	105	120	-
1,3,5-Trimethylbenzene	105	120	77	m,p-xylene	106	91	-
1,3-Butadiene	54	53	39	Methacrylonitrile	67***	41	-
1,3-Dichlorobenzene	146	111	148	Methyl Acetate	43	74	-
1,3-Dichloropropane	76	78	-	Methyl isothiocyanate	73	72	45, 98
1,4-Dichlorobenzene	146	111	148	Methyl Methacrylate	69	41	100
1,4-Dioxane	88	58	43	Methyl tert-butyl ether	73	57	41
2,2,4-Trimethylpentane	57	41	99	Methylcyclohexane	83	55	98
2,2-Dichloropropane	77	97	-	Methylene Chloride	84	86	49
2-Butanone	43***	72	-	Naphthalene	128	-	-
2-Chloroethylvinyl ether	63	106	65	n-butyl acetate	43	56	73
2-Chlorotoluene	91	126	-	n-Butylbenzene	91	134	-
2-Hexanone	43	58	57	Nonane	43	57	128
2-Nitropropane	43	41	39	n-Propylbenzene	91	120	-
2-Propanol	45	59	-	Octane	43	57	114
4-Chlorotoluene	91	126	-	o-Xylene	106	91	-
4-Isopropyltoluene	119	134	91	p-Diethylbenzene	119	105	120
4-Methyl-2-pentanone	43	58	57	Pentachloroethane	167	117	119
Acetaldehyde	44	43	42	p-Ethyltoluene	105	120	-
Acetone	43	58	-	Propionitrile	54	55	-
Acetonitrile	41	39	-	Sec-Butylbenzene	105	134	-
Acrolein	56	55	-	Styrene	104	78	-
Acrylonitrile	53	52	-	tert Amyl methyl ether	73	43	87
Allyl Chloride	76	39	41	tert-amyl alcohol	59	73	55
Benzene	78	77	-	tert-amyl ethyl ether	59	87	73
Benzyl chloride	91	126	-	tert-Butyl Alcohol	59	57	41

Compound	Primary Ion*	Secondary Ion	Tertiary Ion	Compound	Primary Ion*	Secondary Ion	Tertiary Ion
Bromobenzene	156	77	158	Tert-Butylbenzene	119	91	-
Bromochloromethane	128	49	130	Tetrachloroethene	166	168	129
Bromodichloromethane	83	85	127	Tetrahydrofuran	42	71	72
Bromoform	173	175	-	Toluene	91	92***	-
Bromomethane	94	96	-	Trans-1,2-Dichloroethene	96	61	98
Carbon disulfide	76	78	-	Trans-1,3-Dichloropropane	75	110	-
Carbon Tetrachloride	117	119	-	Trans-1,4-Dichloro-2-butene	53	75	89
Chlorobenzene	112	77	114	trans-Decahydronaphthalene	138	96	67
Chlorodifluoromethane	51	67	69	Trichloroethene	95	130	132
Chloroethane	64	66	-	Trichlorofluoromethane	101	103	-
Chloroform	83	85	-	Undecane	43	57	156
Chloromethane	50	52	-	Vinyl acetate	43	86	-
Chloroprene	53	88	90	Vinyl Chloride	62	64	-
Cis-1,2-Dichloroethene	96	61	98	Xylene (total)	106	91	-
cis-1,3-Dichloropropane	75	110	-				

* The primary ion should be used unless interferences are present, in which case, a second ion may be used.
** m/z 43 is used for quantification of 2-Butanone, but m/z 72 must be present for positive identification.
*** Quantitation ion differs from primary ion.

Attachment II: CHARACTERISTIC IONS FOR SURROGATE AND INTERNAL STANDARDS FOR VOLATILE ORGANIC COMPOUNDS

Surrogate Standard Compounds

Compound	Primary Ion	Secondary Ion	Tertiary Ion	CAS No.
1,2-Dichloroethane-d4	65	120	-	17060-07-0
4-Bromofluorobenzene	95	174	176	460-00-4
Toluene-d8	98	70	100	2037-26-5

Internal Standards

Compound	Primary Ion	Secondary Ion	Tertiary Ion	CAS No.
1,4-dichlorobenzene-d4	152	150	115	03855-82-1
1,4-difluorobenzene	114	63	88	540-36-3
Bromochloromethane	49	128*	130	74-97-5
chlorobenzene-d5	117	82*	119	3114-55-4
Pentafluorobenzene	99	168*	-	363-72-4

*Quantitation ion differs from primary ion

Attachment III: VOLATILE INTERNAL STANDARDS ASSIGNED FOR QUANTITATION OF TARGETED COMPOUNDS AND SYSTEM MONITORING COMPOUNDS (4 INTERNALS)

Pentafluorobenzene (IS_1)	1,4-Difluorobenzene (IS_2)	Chlorobenzene-d5 (IS_3)	1,4-Dichlorobenzene-d4 (IS_4)
Chlorodifluoromethane (Freon 22)	1,1,1-Trichloroethane	Toluene-d8 (surrogate)	Trans-Decahydronaphthalene
1,1,2,2-Tetrafluoro-1,2-dichloroethane (Freon 114)	Carbon tetrachloride	4-Bromofluorobenzene (surrogate)	Isopropylbenzene
Dichlorodifluoromethane	2-Chloroethylvinyl ether	Tetrachloroethane	Cis-Decahydronaphthalene
Chloromethane	1,1-Dichloropropene	1,3-Dichloropropene	Bromobenzene
Vinyl chloride	Benzene	Dibromochloromethane	1,1,2,2- Tetrachloroethane
1,3-Butadiene	Methylcyclohexane	2-Hexanone	1,2,3-Trichloropropane
Acetaldehyde	Methyl methacrylate	Chlorobenzene	Trans-1,4-Dichloro-2-butene
Bromomethane	Trichloroethene	1,1,1,2-Tetrachloroethane	n-Propylbenzene
Ethanol	2-Nitropropane	Ethylbenzene	2-Chlorotoluene
Diethyl ether	1,2-Dichloropropane	m,p-Xylene	4-Chlorotoluene
Chloroethane	1,4-Dioxane	Nonane	p-Ethyltoluene
Acetone	Dibromomethane	D-Limonene	1,3,5-Trimethylbenzene
Iodomethane	Bromodichloromethane	o-Xylene	tert-Butylbenzene
Acetonitrile	Ethyl methacrylate	Xylene(total)	1,2,4-Trimethylbenzene
Acrolein	Methyl isothiocyanate	Styrene	sec-Butylbenzene
Acrylonitrile	Cis-1,3-Dichloropropene	Bromoform	1,3-Dichlorobenzene
Trichlorofluoromethane	4-Methyl-2-pentanone	Decane	4-Isopropyltoluene
1,1-Dichloroethene	n-Butyl acetate		1-4-Dichlorobenzene
2-Propanol	Toluene		1,2,3-Trimethylbenzene
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	Octane		Benzyl Chloride
Carbon disulfide	trans-1,3-Dichloropropene		1,2-Dichlorobenzene
Tert-butyl alcohol	1,1,2-Trichloroethane		p-Diethylbenzene
Allyl Chloride	1,2-Dibromoethane		n-Butylbenzene
Methyl acetate	1,2-Dichloroethane-d4 (surrogate)		Undecane
Methyl tert-butyl ether			1,2-Dibromo-3-Chloropropane
Methylene Chloride			1,2,4,5-Tetramethylbenzene
Hexane			Nitrobenzene
Trans-1,2-Dichloroethene			Dodecane
Vinyl acetate			1,2,4-Trichlorobenzene
1,1-Dichloroethane			Hexachlorobutadiene
Chloroprene			Naphthalene
Propionitrile			1,2,3-Trichlorobenzene
Ethyl tert-butyl ether			
2,2-Dichloropropane			
cis-1,2-Dichloroethene			
Tetrahydrofuran			
Bromochloromethane			
2,2,4-Trimethylpentane			
Chloroform			
2-Butanone			
Ethyl Acetate			
Cyclohexane			
tert-Amyl methyl ether			
Heptane			
1,2-Dichloroethane			

Attachment IV: VOLATILE INTERNAL STANDARDS ASSIGNED FOR QUANTITATION OF TARGETED COMPOUNDS AND SYSTEM MONITORING COMPOUNDS (3 INTERNALS)

Bromochloromethane (IS_1)	**1,4-Difluorobenzene (IS_2)**	**Chlorobenzene-d5 (IS_3)**
Chlorodifluoromethane (Freon 22)	1,1,1-Trichloroethane	4-Methyl-2-pentanone
Dichlorodifluoromethane	Cyclohexane	2-Hexanone
Chloromethane	Carbon tetrachloride	1,2-Dibromoethane
Bromomethane	2-Chloroethylvinyl ether	Tetrachloroethane
Vinyl chloride	Bromodichloromethane	1,1,2,2- Tetrachloroethane
Chloroethane	1,2-Dichloropropane	Toluene-d8 (surrogate)
Acrolein	Cis-1,3-Dichloropropene	Toluene
Acrylonitrile	Trichloroethene	Chlorobenzene
Methyl acetate	Methylcyclohexane	Ethylbenzene
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	Benzene	4-Bromofluorobenzene (surrogate)
Methylene Chloride	Dibromochloromethane	Styrene
Acetone	trans-1,3-Dichloropropene	m,p-Xylene
Carbon disulfide	1,1,2-Trichloroethane	o-Xylene
Tert-butyl alcohol	Bromoform	Xylene(total)
1,1-Dichloroethene		Isopropylbenzene
1,1-Dichloroethane		1,3-Dichlorobenzene
Trichlorofluoromethane		1-4-Dichlorobenzene
Vinyl acetate		1,2-Dichlorobenzene
Methyl tert-butyl ether		1,2-Dibromo-3-Chloropropane
Trans-1,2-Dichloroethene		1,2,4-Trichlorobenzene
cis-1,2-Dichloroethene		Naphthalene
1,2-Dichloroethene(total)		
2-Butanone		
Chloroform		
1,2-Dichloroethane-d4 (surrogate)		
1,2-Dichloroethane		

Attachment V: MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION FOR 8260C

Analyte	Minimum Response Factor	Typical Response Factor
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene Chloride	0.100	0.38
Trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-butyl ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382
Bromodichloromethane	0.200	0.424
Cis-1,3-Dichloropropene	0.200	0.537
Trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethane	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
m,p-Xylene	0.100	1.08
o-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2- Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332

Analyte	Minimum Response Factor	Typical Response Factor
1,2-Dibromo-3-Chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

*****The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.

Attachment VI: SYSTEM MONITORING COMPOUND RECOVERY LIMITS

(Typical limits. Updated limits can be found in the LIMS Data System)

	Routine Limits		ASP Limits	
Compound	**%Recovery Water**	**%Recovery Soil**	**%Recovery Water**	**%Recovery Soil**
1,2-Dichloroethane-d4	53-183	33-150	76-114	70-121
4-Bromofluorobenzene	63-140	34-145	86-115	59-113
Toluene-d8	60-135	43-157	88-110	84-138

Attachment VII: QC LIMITS FOR MATRIX SPIKE/SPIKE DUPLICATE AND MATRIX SPIKE BLANK

ASP Criteria

Compound	% Recovery Water	RPD Water	% Recovery Soil	RPD Soil
1,1-Dichloroethene	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

Attachment VIII: INSTRUMENT OPERATING PARAMETERS FOR FUEL OXYGENATES

(Recommended program; modify as needed to optimize)

Purge Process

Sample Size	5mL
Purge Time	9 min
Purge Flow	40mL/min
Purge Temp.	80ºC
Desorption Flow	8 to 30 mL/min
Desorption Time	1.4 ± 0.1min
Desorption Temp	180ºC

Attachment IX: RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF VOLATILE ORGANIC COMPOUNDS FOR ASP 2000 CRITERIA

Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
1,1,1-Trichloroethane	0.100	± 20.5	± 25.0
1,1,2,2-Tetrachloroethene	0.500	± 20.5	± 25.0
1,1,2-Trichloroethane	0.100	± 20.5	± 25.0
1,1-Dichloroethane	0.200	± 20.5	± 25.0
1,1-Dichloroethene	0.100	± 20.5	± 25.0
1,2-Dichloroethane	0.100	± 20.5	± 25.0
1,2-Dichloroethane-d4	0.010	± 100.0	± 100.0
1,2-Dichloroethene (Total)	0.010	± 100.0	± 100.0
1,2-Dichloropropane	0.010	± 100.0	± 100.0
2-Butanone	0.010	± 100.0	± 100.0
2-Hexanone	0.010	± 100.0	± 100.0
4-Methyl-2-pentanone	0.010	± 100.0	± 100.0
Acetone	0.010	± 100.0	± 100.0
Benzene	0.500	± 20.5	± 25.0
Bromodichloromethane	0.200	± 20.5	± 25.0
Bromofluorobenzene	0.200	± 20.5	± 25.0
Bromoform	0.100	± 20.5	± 25.0
Bromomethane	0.100	± 20.5	± 25.0
Carbon disulfide	0.010	± 100.0	± 100.0
Carbon Tetrachloride	0.100	± 20.5	± 25.0
Chlorobenzene	0.500	± 20.5	± 25.0
Chloroethane	0.010	± 100.0	± 100.0
Chloroform	0.200	± 20.5	± 25.0
Chloromethane	0.010	± 100.0	± 100.0
cis-1,3-Dichloropropene	0.200	± 20.5	± 25.0
Dibromochloromethane	0.100	± 20.5	± 25.0
Ethylbenzene	0.100	± 20.5	± 25.0
Methylene Chloride	0.010	± 100.0	± 100.0
Styrene	0.300	± 20.5	± 25.0
Tetrachloroethene	0.200	± 20.5	± 25.0
Toluene	0.400	± 20.5	± 25.0
Toluene-d8	0.010	± 100.0	± 100.0
Trans-1,3-Dichloropropene	0.100	± 20.5	± 25.0
Trichloroethene	0.300	± 20.5	± 25.0
Vinyl Chloride	0.100	± 20.5	± 25.0
Xylene(Total)	0.300	± 20.5	± 25.0

* Allowance is made for two compounds to exceed the %RSD or %D requirements, provided the minimum RRF is ≥0.010, and the %RSD or %D are ≤40% for initial and continuing calibrations, respectively.

Attachment X: RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF VOLATILE ORGANIC COMPOUNDS FOR ASP 2005 CRITERIA

Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
1,1,1-Trichloroethane	0.100	20.5	±25.0
1,1,2,2-Tetrachloroethane	0.300	20.5	±25.0
1,1,2-Trichloro-1,2,2-trifluoroethane	0.010	40.5	±40.0
1,1,2-Trichloroethane	0.100	20.5	±25.0
1,1-Dichloroethane	0.200	20.5	±25.0
1,1-Dichloroethene	0.100	20.5	±25.0
1,2,3-Trichlorobenzene	0.200	20.5	±25.0
1,2,4-Trichlorobenzene	0.200	20.5	±25.0
1,2-Dibromo-3-chloropropane	0.010	40.5	±40.0
1,2-Dibromoethane	0.010	40.5	±40.0
1,2-Dichlorobenzene	0.400	20.5	±25.0
1,2-Dichloroethane	0.100	20.5	±25.0
1,2-Dichloropropane	0.010	40.5	±40.0
1,3-Dichlorobenzene	0.600	20.5	±25.0
1,4-Dichlorobenzene	0.500	20.5	±25.0
1,4-Dioxane	0.010	40.5	±40.0
2-Butanone	0.010	40.5	±40.0
2-Hexanone	0.010	40.5	±40.0
4-Methyl-2-pentanone	0.010	40.5	±40.0
Acetone	0.010	40.5	±40.0
Benzene	0.400	20.5	±25.0
Bromochloromethane	0.050	20.5	±25.0
Bromodichloromethane	0.200	20.5	±25.0
Bromoform	0.050	20.5	±25.0
Bromomethane	0.100	20.5	±25.0
Carbon disulfide	0.010	40.5	±40.0
Carbon tetrachloride	0.100	20.5	±25.0
Chlorobenzene	0.500	20.5	±25.0
Chloroethane	0.010	40.5	±40.0
Chloroform	0.200	20.5	±25.0
Chloromethane	0.010	40.5	±40.0
cis-1,2-Dichloroethene	0.010	40.5	±40.0
cis-1,3-Dichloropropene	0.200	20.5	±25.0
Cyclohexane	0.010	40.5	±40.0
Dibromochloromethane	0.100	20.5	±25.0
Dichlorodifluoromethane	0.010	40.5	±40.0
Ethylbenzene	0.100	20.5	±25.0
Isopropylbenzene	0.010	40.5	±40.0

Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
m, p-Xylene	0.300	20.5	±25.0
Methyl acetate	0.010	40.5	±40.0
Methyl tert-butyl ether	0.010	40.5	±40.0
Methylcyclohexane	0.010	40.5	±40.0
Methylene chloride	0.010	40.5	±40.0
o-Xylene	0.300	20.5	±25.0
Styrene	0.300	20.5	±25.0
Tetrachloroethene	0.100	20.5	±25.0
Toluene	0.400	20.5	±25.0
trans-1,2-Dichloroethene	0.010	40.5	±40.0
trans-1,3-Dichloropropene	0.100	20.5	±25.0
Trichloroethene	0.300	20.5	±25.0
Trichlorofluoromethane	0.010	40.5	±40.0
Vinyl chloride	0.100	20.5	±25.0

Attachment XI: FUEL OXYGENATE COMPOUNDS PLUS AROMATICS

Compound
Benzene
Diisopropyl ether
Ethanol
Ethyl tert-butyl ether
Ethylbenzene
m,p-xylene
Methyl tert-butyl ether
o-Xylene
tert Amyl methyl ether
tert-amyl alcohol
tert-amyl ethyl ether
tert-Butyl Alcohol
Toluene

APPENDIX P2-3
SOP FOR THE PRESSURIZED FLUID EXTRACTION BY SW-846 METHOD 3545A
(S-LI-O-020-REV.00)

STANDARD OPERATING PROCEDURE

PRESSURIZED FLUID EXTRACTION
Reference Methods: EPA SW-846 METHOD 3545A

Local SOP Number:	S-LI-O-020-rev.00
Effective Date:	Date of Final Signature

APPROVALS

[signature]	5/22/15
General Manager	Date
[signature]	5/22/15
Quality Manager	Date
[signature]	5/22/15
Department Manager	Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Identification of Test Method

1.1. This Standard Operating Procedure (SOP) documents the procedures used by Pace Long Island for extracting non-volatile and semi-volatile organic compounds from soils, clays, sediments, sludges and waste solids using pressurized fluid extraction while meeting the requirements specified in EPA SW-846 Method 3545A.

1.2. For work governed by the NYS DEC Analytical Service Protocol (ASP), the requirements for analysis and reporting of the DEC ASP have to be met.

1.3. In addition to the requirements of this SOP, the guidelines in the *Quality Assurance, Quality Control Manual* have to be observed.

2. Summary of Method

2.1. Samples are prepared for extraction either by air drying the sample, or by mixing the sample with anhydrous sodium sulfate or pelletized diatomaceous earth.

2.2. The extraction cell containing the sample is heated to the extraction temperature, pressurized with the appropriate solvent system, and extracted for 5 minutes (or as recommended by the instrument manufacturer). The solvent system used for this procedure is Acetone/Dichloromethane (1:1 V/V).

2.3. The solvent is collected from the heated extraction vessel and allowed to cool.

2.4. The extract may be concentrated, if necessary, and, as needed, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3. Scope and Application

3.1. Method 3545A is a procedure for extracting water insoluble or slightly water soluble semivolatile organic compounds from soils, clays, sediments, sludges, and waste solids. The method uses elevated temperature (100°C) and pressure (1500-2000 PSI) to achieve analyte recoveries equivalent to those from Soxhlet extraction, using less solvent and taking significantly less time than the Soxhlet procedure.

3.2. This method is applicable to the extraction of semivolatile organic compounds, organophosphorus pesticides, organochlorine pesticides, chlorinated herbicides, and PCBs, which may then be analyzed by a variety of chromatographic procedures.

3.3. This method has been validated for solid matrices containing 250 to 12,500 μg/kg of semivolatile organic compounds, 250 to 2500 μg/kg of organophosphorus pesticides, 5 to 250 μg/kg of organochlorine pesticides, 50 to 5000 μg/kg of chlorinated herbicides and 1 to 1400 μg/kg of PCBs. The method may be applicable to samples containing these analytes at higher concentrations and may be employed after adequate performance has been demonstrated for the concentrations of interest (see Method 3500, Sec. 8.0).

3.4. This method is applicable to solid samples only, and is most effective on dry materials with small particle sizes. Therefore, waste samples must undergo phase separation and only the solid phase material is to be extracted by this procedure. If possible, soil/sediment samples may be air-dried and ground to a fine powder prior to extraction. Alternatively, if the loss of analytes during drying is a concern, soil/sediment samples may be mixed with pelletized diatomaceous earth. The total mass of material to be prepared is 15 g.

3.5. This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

4. Applicable Matrices

4.1. This Standard Operating Procedure (SOP) documents the procedures used by Pace Long Island for extracting non-volatile and semi-volatile organic compounds from soils, clays, sediments, sludges and waste solids using pressurized fluid extraction while meeting the requirements specified in EPA SW-846 Method 3545A.

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.

6.3. Extracts that exhibit interferences can be run through a cleanup procedure (see EPA method 3600C). Before using a cleanup method, the analyst should 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, Shipment and Storage

Table 7.1 – Sample Collection, Preservation, Storage, and Hold time

Sample Type	Collection per Sample	Preservation	Storage	Holding Time
Solid	8 Ounce glass jar Teflon-lined cap	None	4 ± 2°C	Extraction – 14 days from collection date. Analysis – 40 days from extraction 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. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies

Table 9.1 - Instrumentation

Equipment	Vendor	Model / Version
N-EVAP	Organomation	11155
Water Bath	Boekel/Grant	PB-2800
Accelerated Solvent Extractor*	Dionex	ASE350
Extraction Cell	Dionex	068103
Extraction Cell Filter	Dionex	56780
Analytical Balance	Ohaus	SP202

*See www.dionex.com/en-us/products/sample-preparation/ase/accessories/cells/lp-79266.html for replacement parts for extraction cells.

Table 9.2 - Glassware

Glassware	Vendor	Catalog #
Glass Funnels	Fisher	10-322F
250 mL Kuderna Danish Concentrators	Fisher	570035-0250
500 mL Kuderna Danish Concentrators	Fisher	570035-0500
250 mL Kuderna Danish Flasks	Fisher	570011-0250
500 mL Kuderna Danish Flasks	Fisher	570037-0500
Snyder Columns	Fisher	503000-0121
Concentrator Tubes	Fisher	570051-1025

Table 9.3 - General Supplies

Supply	Vendor	Catalog #
2 mL Amber Vials	Restek	24386
2 mL Clear Vials	Restek	24384
Crimp Caps	Restek	24370
Clips for KD flasks	Fisher	05-884D
Boiling chips	Fisher	09-191-20
5 ¾ inch Pasteur Pipets	Fisher	22-230-482
9 inch Pasteur Pipets	Fisher	22-230-490
pH Test Strips	Fisher	M1095350007
11 mL Clear Vials	Fisher	B69308
40 mL Amber Vials	SciSpec	B75741
40 mL Clear Vials	SciSpec	B75740
60 mL Clear Vials	SciSpec	176760
10 µL Syringe	Hamilton	80000
25 µL Syringe	Hamilton	80200
50 µL Syringe	Hamilton	80920/80900
100 µL Syringe	Hamilton	81020/81000
250 µL Syringe	Hamilton	81120
500 µL Syringe	Hamilton	81220
1000 µL Syringe	Hamilton	81320

10. Reagents and Standards

Table 10.1 – Reagents

Reagent	Vendor	Catalog #
Sand	Fisher	S253
Sodium Sulfate	Fisher	S-415-200LB
Dichloromethane	Fisher	D151-4
Acetone	Fisher	A949-4
Hexane	Fisher	H306-4
Hydromatrix	Fisher	50-010-1753

1:1 (v/v) Acetone/Dichloromethane – Combine 500 mL of Acetone and 500 mL of Dichloromethane in the 1L solvent bottle attached to the ASE.

Table 10.2 – 8270/SIM Stock Standards

Standard	Description	Concentration	Vendor	Catalog #
QC8270_1	1,3,5-Trinitrobenzene	1000 µg/mL	Restek	568614
QC8270_2	8270 Megamix	1000 µg/mL	Restek	31850
QC8270_3	Appendix IX Mix 2	1000 µg/mL	Restek	568635
QC8270_4	Decane	1000 µg/mL	SPEX	S-1112
QC8270_5	Dinoseb	1000 µg/mL	Restek	32251
QC8270_6	Octadecane	1000 µg/mL	SPEX	S-2850
QC8270_7	Appendix IX Mix 1	2000 µg/mL	Restek	32459
QC8270_8	Benzidine Mix	2000 µg/mL	Restek	31834
QC8270_9	Benzoic Acid	2000 µg/mL	Restek	31879
QC8270_10	Methapyrilene	2000 µg/mL	Restek	32460
QC8270_11	Organophosphorous Pesticides Mix	2000 µg/mL	Restek	32419
QC625_1	Acid Composite Mix	2000 µg/mL	Accustandard	CLP-HC-A-R
QC625_2	Base/Neutral Composite Mix	2000 µg/mL	Accustandard	CLP-HC-BN-R
QC625_3	Composite Mix 3	2000 µg/mL	Accustandard	Z-014F
QC625_4	Benzidines Mix	2000 µg/mL	Accustandard	Z-014E-R3
QC4.2_1	Acetophenone	5000 µg/mL	Restek	30621
QC4.2_2	Atrazine	1000 µg/mL	Restek	32208
QC4.2_3	Benzaldehyde	2000 µg/mL	Restek	33017
QC4.2_4	Biphenyl	2000 µg/mL	Supelco	4-8161
QC4.2_5	Caprolactam	2000 µg/mL	Restek	31833
SS952_1	BNA Surrogates, Acid/Base Indicator	100-150 µg/mL	Ultra	ISM-336XC-500
SS952SIM_1	SOM 1.0 SIM	2000 µg/mL	Restek	33913

Table 10.3 – DRO Stock Standards

Standard	Description	Concentration	Vendor	Catalog #
QCDRO_1	Diesel Fuel 2	50,000 µg/mL	Accustandard	DRO-AK-102-LCS-10X-R1
SSDRO_1	1,4-Dichlorobenzene-d4	2000 µg/mL	Accustandard	Z-014J-3-M-0.5X

Table 10.4 – 8081/8082 Stock Standards

Standard	Description	Concentration	Vendor	Catalog #
QC8081_1	alpha-Chlordane	1000 µg/mL	Accustandard	P-134S-A-10X
QC8081_2	gamma-Chlordane	1000 µg/mL	Accustandard	P-135S-A-10X
QC8081_3	Isodrin	100 µg/mL	Accustandard	P-471S
QC8081_4	Semi-Volatile Calibration Mix 6	2000 µg/mL	Restek	31012
QC8081_5	Chlordane	100 µg/mL	Accustandard	P-017S
QC8081_6	Toxaphene	1000 µg/mL	Accustandard	AS-E0111
QC8081_7	Mirex	100 µg/mL	Accustandard	P-066S
QC8082_1	Aroclor 1016	1000 µg/mL	Ultra	EPA-1282

QC8082_2	Aroclor 1260	1000 µg/mL	Ultra	EPA-1362
SS953_1	Pesticides Surrogate Mix	200 µg/mL	Supelco	CRM48460

Table 10.5 – 8141 Stock Standards

Standard	Description	Concentration	Vendor	Catalog #
QC8141_1	Organophosphorous Pesticide Mix	2000 µg/mL	Ultra	US-119
QC8141_2	Organophosphorous Pesticide Mix	200 µg/mL	Ultra	SPM-614
QC8141_3	Chloropyrifos	100 µg/mL	Ultra	PST-480M100A01
SS8141_1	Methidathion	100 µg/mL	Ultra	PST-1520A100A01

Table 10.7 - Standard Definitions

Standard	Description
Surrogate Standard	Surrogates are added to each sample and QC sample to monitor extraction efficiency.
QC Standard	This solution contains all targeted analytes and is added to the LFB and MS/MSD.

Table 10.8 - Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	§ Reference solution purchased directly from approved vendor	§ Unopened - Expiration date listed on standard ampule. § Opened - 6 months from date opened or listed expiration date (whichever is sooner)	§ Storage conditions listed on standard ampule.
Intermediate and Working Standard Solutions	§ Solutions prepared by diluting stock solution	§ 6 months from preparation or stock standard expiration date (whichever is sooner) § Replace solution sooner if degradation or evaporation is suspected.	§ Amber vials with Teflon lined screw caps § Method required storage conditions.

Table 10.9 – Working Level Standard Preparation

Solution Name	Concentration	Final Volume	Stocks	Solvent
QC8270	50 µg/mL	20 mL	1000 µL QC8270_1 1000 µL QC8270_2 1000 µL QC8270_3 1000 µL QC8270_4 1000 µL QC8270_5 1000 µL QC8270_6 500 µL QC8270_7 500 µL QC8270_8 500 µL QC8270_9 500 µL QC8270_10 500 µL QC8270_11	Methanol
QC625	100 µg/mL	20 mL	1000 µL QC625_1 1000 µL QC625_2 1000 µL QC625_3	Methanol
BENZIDINES	2000 µg/mL	1 mL	Undiluted QC625_4	Methanol

QC4.2	100 µg/mL	20 mL	400 µL QC4.2_1 2000 µL QC4.2_2 1000 µL QC4.2_3 1000 µL QC4.2_4 1000 µL QC4.2_5	Methanol
SS952	100 µg/mL 150 µg/mL	500 mL	Undiluted SS952_1	Methanol
QC625SIM	10 µg/mL	5 mL	500 µL QC625	Methanol
SS952SIM	10 µg/mL	20 mL	100 µL SS952SIM_1	Methanol
QCDRO	50,000 µg/mL	1 mL	Undiluted QCDRO_1	Acetone
SSDRO	2000 µg/mL	1 mL	Undiluted SSDRO_1	Methanol
QC8081	20 µg/mL	10 mL	QC8081_1 QC8081_2 QC8081_3 QC8081_4	Acetone
CHLORDANE	100 µg/mL	N/A	Undiluted QC8081_5	Methanol
TOXAPHENE	1000 µg/mL	N/A	Undiluted QC8081_6	Methanol
MIREX	0.4 µg/mL	25 mL	100 µL QC8081_7	Acetone
QC8082	10 µg/mL	100 mL	1000 µL QC8082_1 1000 µL QC8082_2	Acetone
SS953	0.2 µg/mL	1000 mL	1000 µL SS953_1	Acetone
QC8141	200 µg/mL	10 mL	1000 µL QC8141_1	Acetone
SPM614	20 µg/mL	20 mL	2000 µL QC8141_2	Acetone
CHLOROPYRIPHOS	100 µg/mL	1 mL	Undiluted QC8141_3	Acetone
SS8141	10 µg/mL	20 mL	2000 µl SS8141_1	Acetone

11. Calibration and Standardization

11.1. Not applicable to this SOP.

12. Procedure

12.1. Decant and discard any water layer, rocks, leaves, twigs, etc from the soil sample. Mix each sample well.

12.2. Weigh 15 g of sample on an aluminum weighing dish using an analytical balance. Record the mass to two decimal places for each sample.

12.3. Prepare two 15 g samples of hydromatrix to be used for Method Blank and Laboratory Fortified Blank.

12.4. Select one field sample from the batch and weigh two additional 15 g aliquots to be used for Matrix Spike and Matrix Spike Duplicate.

12.5. Spike the LFB, MS and MSD with QC solution according to table 12.3.

12.6. Spike each Field Sample and QC Sample with Surrogate solution according to table 12.2.

12.7. Combine the sample with 2 grams of hydromatrix and mix well.Add additional hydromatrix as needed to obtain a free flowing powder.

12.8. Transfer each sample to a clean extraction cell.

12.9. Tightly seal the top screw cap of each extraction cell and place cells on the Accelerated Solvent Extractor starting at position 1.

12.10. Place labeled 60 mL vials on the collection compartment of the ASE, making sure to use the same position number as the corresponding cell position number.

12.11. Prepare 1:1 (v/v) Acetone/Dichloromethane and connect solvent bottle to the adapter on the ASE.

12.12. Start the programmed extraction method (see table 12.4) and allow all samples to go through the extraction process.

12.13. When all samples have finished remove the 60 mL vials and pour each extract through a glass funnel containing Sodium Sulfate and into a 250 mL KD Evaporator or 200mL TurboVap tube.

12.14. Extract Concentration with KD Concentrators

12.14.1. Remove the drying funnel and discard the used sodium sulfate.

12.14.2. Add ~3 boiling chips, attach a Snyder Column to the top of the KD Flask.

12.14.3. Pre-wet the column by adding one mL MC to the top.

12.14.4. Place each sample in a hot water bath. The temperature of the water bath should be ~20°C above the boiling point of the solvent, however it must be adjusted as required to complete the concentration in 10-20 minutes.

12.14.5. Concentrate until the extract volume is ~3 mL. Do not allow the samples to go to dryness.

12.14.6. Remove the KD Concentrator from the water bath. Add a squirt of Methylene chloride into the Snyder column to rinse. Remove the clips and rinse the outside of the flask with Acetone to remove water. Allow the sample to sit for ~10 minutes.

12.14.7. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1 - 2 mL of methylene chloride. Detach the concentrator tube, place on the N-EVAP, as described below in Section 12.19. If solvent exchange is required, transfer the extract from the concentrator tube to a 11 mL vial, making sure to rinse the tube with Methylene chloride for a quantitative transfer, and then proceed to Section 12.19 for N-EVAP concentration.

12.15. Extract Concentration on TurboVap

12.15.1. The TurboVap provides automated solvent concentration with nitrogen and a sensor controlled endpoint.

12.15.2. Remove the drying funnel from the extracts collected in the TurboVap concentrator tubes and discard the used sodium sulfate.

12.15.3. Set temperature to 50°C and N2 pressure to 7 psi. Select end volume of 1 mL. Load tubes with sample extracts and close cover.

12.15.4. When extracts reach ~ 10 mL, it is necessary to rinse the glassware walls throughout the remainder of the concentration process. The number of rinse steps will be dependent on the sample matrix.

12.15.5. When “ready” light flashes, the end point is reached.

12.15.6. For Tests that need solvent exchange (8081, TCLP PEST, 8082, 8141) see Section 12.19.7.

12.15.7. For Tests that do not need solvent exchange (8270D, 8270D-SIM, 625, 8015D DRO, TCLP BNA) see Section 12.19.6

Note: Carefully rinse the walls and particularly the tip of the concentrator tubes for quantitative transfer of the samples. The rinsing process is very critical to obtain adequate recovery. Great care has to be taken to rinse the walls and especially the lower cone very thoroughly.

12.16. N-EVAP Concentration

12.16.1. Adjust the temperature of the N-EVAP to 30°C -35°C. Install fresh Pasteur pipets as nozzles for each new set of samples to avoid cross contamination.

12.16.2. Load tubes or vials with sample extracts on the manifold and lower nozzles until they almost touch the surface of the extracts.

12.16.3. Turn on the nitrogen supply at 10-20 psi and adjust the individual valves to evenly supply each position with a GENTLE stream of clean dry nitrogen (Observe distortion of solvent surface to gauge intensity.)

12.16.4. Submerge the tubes in the warm bath. This will prevent water condensation.

12.16.5. DO NOT allow sample to evaporate too low or lightweight compounds may be lost.

12.16.6. For Tests that do not need solvent exchange (8270D, 8270D-SIM, 625, 8015D DRO, TCLP BNA):

- When the volume reaches ~0.5 -1 mL, transfer to a 2 mL amber vial and adjust volume according to table 12.1 using final solvent rinse of concentrator tube.

12.16.7. For Tests that do need solvent exchange (8081, TCLP PEST, 8082, 8141):

- When the volume reaches ~0.5-1 mL, add ~3 mL of hexane and concentrate to slightly below 1 mL.
- Repeat hexane solvent exchange two additional times.
- Adjust volume according to table 12.1 using final solvent rinse of concentrator tube.
- Transfer a 1 mL aliquot to a 2 mL vial.

12.17. Additional cleanup procedures may be necessary:

12.17.1. For samples containing high concentrations of high molecular weight non-targeted analytes, Gel Permeation Chromatography by EPA 3640A may be performed.

12.17.2. For Pesticide and PCB samples Sulfur Cleanup by EPA 3660B is required.

For PCB samples Acid Cleanup by EPA 3665A is required

Table 12.1 – Method Initial Volume, pH, Final Volume and Final Solvent

Analytical Method	Initial Volume	Final Volume	Final Solvent
8270D	15 g	1.0 mL	Dichloromethane
8270DSIM	15 g	1.0 mL	Dichloromethane
8015D DRO	15 g	1.0 mL	Dichloromethane
8081B	15 g	5.0 mL	Hexane
8082A	15 g	5.0 mL	Hexane
8141B	15 g	5.0 mL	Hexane

Table 12.2 – Surrogate Spiking Chart

Analytical Method	Solution	Spike Volume
8270D	SS952	250 µL
8270DSIM	SS952SIM	100 µL
8015D DRO	SSDRO	75 µL

Analytical Method	Solution	Spike Volume
8081	SS953	1000 µL
8082	SS953	1000 µL
8141	SS8141	500 µL

Table 12.3 – QC Spiking Chart

Analytical Method	Solution	Spike Volume
8270	QC625	250 µL
8270	BENZIDINES	12.5 µL
8270	QC4.2	250 µL
8270	QC8270*	500 µL
8270SIM	QC625SIM	100 µL
8270DRO	QCDRO	50 µL
8081	QC8081	10 µL
8081	CHLORDANE**	20 µL
8081	TOXAPHENE**	10 µL
8081	MIREX	1000 µL
8082	QC8082	250 µL
8141	QC8141	25 µL
8141	SPM614**	250 µL
8141	CHLOROPYRIPHOS**	100µL

*QC8270 contains all currently certified analytes and may be used in place of QC625, BENZIDINES and QC4.2, which contain a smaller TCL list of compounds.

**Prepare an additional LFB for each of these standards.

Table 12.4 – Extarction Conditions for Semivolatiles, organophosphorus pesticides, organochlorine pesticides, herbicides, and PCBs

Oven Temp.	Pressure	Static Time	Flush Volume	Purge Time
100 °C	1500-2000 psi	5 min (after 5-min pre-heat equilibration) 1 cycle	60% of the cell volume	60 s

13. Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
MB	DI water	One per batch of up to 20 samples	All targeted analytes less than PQL	Re-extract batch if there are positives greater than PQL

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
LFB	All targeted analytes	One per batch of up to 20 samples	See Analytical SOPs	Re-extract batch if method specified LFB limits are not met.
MS/MSD	All targeted analytes	One set per batch of up to 20 samples	See Analytical SOPs	Add appropriate qualifiers for all MS/MSD compounds that fail QC requirements. No re-extract necessary.
Surrogates	Method specified Surrogate analytes	All samples and QC samples	See Analytical SOPs	Re-extract sample if method specified surrogate limits are not met.

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. The suitability of the method for the analytes tested was determined when the method was developed. During "method startup" in the laboratory, the capability of the analysts, suitability of equipment, materials and instrumentation was tested.

18.2. Internal method performance is established and monitored with use of the following (where applicable):

- 18.2.1. Method Detection Limit studies
- 18.2.2. Demonstration of Capability
- 18.2.3. Precision and accuracy
- 18.2.4. Positive and negative controls
- 18.2.5. Measurement of sample matrix effects
- 18.2.6. Quality Control Samples (Proficiency Testing)

19. Instrument/Equipment Maintenance/Troubleshooting

19.1. For information and guidance related to instrument/equipment maintenance, troubleshooting and computer hardware and software refer to the following documents:

Document # (latest revision)	Document Title
QAM	*Quality Assurance Quality Control Manual*
ADMIN002	*Computers and Programs*

19.2. Troubleshooting procedures also come from experience and training. Problems that the analyst encounters may be elevated to the department supervisor. Other means of problem resolution may come from review of prior related corrective action reports, instrument maintenance records, internal and external audits and lab notes.

20. Safety

20.1. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by personal protection and engineering measures. MSDS are available for all chemicals used in the lab and are available for review.

20.1.1. Read information and follow warnings listed on the labels of the containers of the chemicals.

20.1.2. In addition, the reference file of material safety data sheets (MSDS) should be consulted for properties of the chemicals used, to determine handling precautions.

20.2. Safety glasses, a lab coat, and disposable gloves must be worn when handling chemicals and samples.

20.3. Sample handling should be conducted in fume hoods.

20.4. The company wide Chemical Hygiene and Safety Manual contains additional information on safety.

21. Waste Management

21.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste.

21.2. Refer to the lab's *Sample and Waste Management* SOP.

22. Pollution Prevention

22.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Use pollution prevention techniques in laboratory operation whenever feasible.

22.2. Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

22.3. Control releases from hood and bench operations and comply with discharge permits and regulations, hazardous waste identification rules, and land disposal restrictions.

22.4. The generated waste has to be disposed in a manner not to cause pollution.

22.5. All wastes deemed toxic are collected in the laboratory and are disposed by professional companies licensed in waste disposal.

22.6. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

23. References

23.1. "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update III, Dec. 1996.

23.2. "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update IV, February 2007

23.3. "New York State Department of Environmental Protection Analytical Services Protocol," June 2000

23.4. "New York State Department of Environmental Protection Analytical Services Protocol," April 2005

23.5. National Environmental Laboratory Accreditation Conference (NELAC) Constitution, Bylaws and Standard, approved June 5, 2003, (EPA/600/R-04/003)

23.6. 40CFR Part 136 Appendix B

23.7. 2009 TNI Laboratory Accreditation Standards, EL-V1-2009.

24. Tables and Figures

24.1. Not applicable to this SOP.

25. Revisions

Document Number	Reason for Change	Date
S-LI-O-020-rev.00	Transition to PACE format.	5/21/15

APPENDIX P2-4
SOP FOR THE DETERMINATION OF SEMI-VOLATILE ORGANICS BY GC/MS BY SW-846 METHOD 8270D
(SOP S-LI-O-014-REV.00)

STANDARD OPERATING PROCEDURE

DETERMINATION OF SEMI-VOLATILE ORGANICS BY GC/MS

Reference Methods: EPA SW-846 Method 8270D

Local SOP Number:	S-LI-I-014-rev.00
Effective Date:	Date of Final Signature
Supersedes:	8270D_r3

APPROVALS

[signature]	5/12/15
General Manager	Date
[signature]	5/10/15
Quality Manager	Date
[signature]	5/12/15
Department Manager	Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Identification of Test Method

1.1. This Standard Operating Procedure (SOP) describes the procedure for the analysis of Semi-volatile Organic Compounds (SVOCs) in environmental samples by EPA SW-846 Method 8270D.

1.2. For work governed by the NYS DEC Analytical Service Protocol (ASP), the requirements for analysis and reporting of the DEC ASP have to be met.

1.2.1. For reporting of data packages with full documentation according to ASP requirements, all raw data have to be included, and summary tables of calibrations and Q. C. data have to be submitted on forms as specified in the DEC ASP.

1.3. In addition to the requirements of this SOP, the guidelines in the *Quality Assurance, Quality Control Manual* have to be observed.

2. Summary of Method

2.1 Sample extracts are prepared for analysis by an appropriate sample preparation method. The semivolatile organic compounds are introduced into the gas chromatograph (GC) by injecting an aliquot of the sample extract. The GC conditions are programmed to separate the analytes. The GC effluent is directly introduced to a mass spectrometer (MS) for both identification and quantification of analytes. Analytes are identified by comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected major (quantitation) ion relative to an internal standard using a multi-point calibration curve.

3. Scope and Application

3.1 This procedure may be used to determine concentrations of neutral, acidic, and basic semivolatile organic compounds in extracts prepared from many types of water samples, soil samples and wastes. Analytes must be soluble in dichloromethane and amenable to capillary gas chromatography. Specific compound classes include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols. A list of applicable compounds including Practical Quantitation Limits (PQL) for water and soil samples is shown in Table 1. PQLs are subject to change based on current analytical system performance and actual sample matrices.

3.2 This method is applicable to most water and solid samples, regardless of moisture content. Common matrices are ground and surface water, wastewater, aqueous sludge, sediment, soils, and other solid samples. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interfere with sample analysis. All adaptations made to address matrix related modifications must be documented within the analytical data.

3.3 This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of semi-volatile configured 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. The method is intended for the analysis of semivolatiles in water, soil/sediment, oil, sludge, and other solid waste materials.

5. Limits of Detection and Quantitation

5.1 Quantification limits are presented in **Table 1**, "Practical Quantitation Limits (PQL) and Limits of Quantitation" (LOQ).

5.2 Reporting limits must be determined at the start of a project and reporting conventions must be established with the client. Project specific requirements must be communicated to analysts prior to sample analysis.

5.3 MDL studies are performed annually by the analysis of seven low-level standards at or below the PQL and calculated by the procedure defined in 40CFR Part 136 Appendix B. Current Method Detection Limits (MDLs) are listed in the LIMS and are available by request from the Quality Manager.

5.4 Refer to the Quality Assurance Manual for definitions, procedures, and requirements for Limits of Detection and Quantitation including LOD, LOQ, and MDL.

6. Interferences

6.1 Interferences may be introduced into sample extracts by contaminants in solvents, reagents, glassware, and any other material that comes in contact with the sample or extract during extract preparation. These interferences must be closely monitored by analyzing Method Blanks and taking corrective action as required.

6.2 Matrix interferences may result from materials co-extracted from some samples.

6.3 Significant phthalate contamination may result at any time if consistent quality control is not practiced. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

7. Sample Collection, Preservation, Shipment and Storage

MATRIX	COLLECTION	PRESERVATION	STORAGE	HOLDING TIME
Aqueous	1 L amber glass with Teflon lined caps	None	4 ± 2°C Extracts should be stored in 1 mL glass autosampler vials, amber. 0-6°C	7 days from collection. (ASP Protocol: 5 days from VTSR*) Analysis within 40 days from extraction.
Solid	Wide mouth glass jars, 4 oz or larger.	None	4 ± 2°C Extracts should be stored in 1 mL glass autosampler vials, amber. 0-6°C	14 days from collection. (ASP Protocol: 12 days from VTSR*) Analysis within 40 days from extraction.

*VTSR = Verified Time of Sample Receipt

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual.

9. Equipment and Supplies

Note: Equivalent vendors/products of the specific items listed below may be used.

Table 9.1 - Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatograph	Agilent	6890N	Two GCs of this vendor/model in use
Mass Spectrometer	Agilent	5973	Two Mass Specs of this vendor/model in use
Data System	.Agilent	Environmental ChemStation	Two Data Systems of this vendor/model in use
Autosampler	Agilent	7683	Two Autosamplers of this vendor/model in use
Vacuum Pump (Rough)	Edwards	E2M2	NA
Vacuum Pump (Rough)	Edwards	E2M1.5	NA

Table 9.2 - Chromatography Supplies

Item	Vendor	Model / ID	Catalog #	Description
Analytical Column	Restek	Rxi-5SILMS with Integra-Guard	13623-124	30m, 0.25mmID, 0.25µm df with 5m guard column
Electron Multiplier	DeTech	2300	2300	NA
Splitless Inlet Liners	Restek	NA	20773	4mm/IP Deactivated inlet liners
O-rings	Restek	NA	20377	Fluorocarbon O-rings for inlet liners
Septa	Restek	NA	23307.5	Themolite injection port septa
Inlet seals	Restek	NA	22084	Gold plated/Dual vespel ring inlet seals
Ferrules	Restek	NA	20251	Graphite- for injection port
Ferrules	Restek	NA	20212	Vespel/Graphite- for detector nut

Table 9.3 - Glassware

Glassware	Description	Vendor / Item # / Description
Volumetric Flasks	5mL, 10mL	Fisher/Class A
Glass Vials	4mL, 12mL, amber with Teflon-lined screw caps	Fisher/ 03-339-23B
Autosampler Vials	1.5mL with Teflon-lined crimp caps	Fisher / 31811E-1232A

Table 9.4 - General Supplies

Supply	Description	Vendor/ Item #
Gas tight syringes	10 µl-1000 µl ; for standard prep	Fisher /Hamilton
Autosampler syringes	5 µl	Fisher /Hamilton 87994
Glass wool	For inlet liners	Fisher /OH Valley Specialty/ 22-289-336

10. Reagents and Standards

Note: Equivalent vendors/products of the specific items listed below may be used.

Table 10.1 - Standard Definitions

Standard	Description	Comments
Tune Standard	Decafluorotriphenylphosphine (DFTPP), 4,4'-DDT, pentachlorophenol, and benzidine solution in dichloromethane used to verify ion response ratios and system inertness prior to analysis	
Initial Calibration Standards	Standards containing all targeted compounds; prepared at varying levels to determine response and retention characteristics of instrument	Method requires a minimum of 5 levels
Continuing Calibration Verification Standard (CCV)	A calibration standard prepared at mid-level concentration for all targeted compounds. This standard is used to verify that the instrument response has not changed significantly since the initial calibration was performed.	This is the same 25 ng/µl standard as used in the initial calibration.
Second Source Verification Standard	A standard prepared from a source other than that used for the initial calibration. This mid-level standard contains all targeted compounds and verifies the accuracy of the calibration curve.	Also called Initial Calibration Verification (ICV)
Internal Standard	A solution added 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.	1,4 Dichlorobenzene-d4 Naphthalene-d_8 Acenaphthene-d_{10} Phenanthrene-d_{10} Chrysene-d_{12} Perylene-d_{12}
Surrogate Standard	A solution added to all samples, spikes, control samples, and method blanks prior to extraction to determine sample preparation efficiency and matrix effects.	Nitrobenzene-d_5 2-Fluorobiphenyl Terphenyl-d_{14} Phenol-d_5 2-Fluorophenol 2,4,6-Tribromophenol
Spiking Standard	This solution is spiked into the Laboratory Fortified Blank (LFB) and spiked samples (MS/MSD) to examine matrix effects.	Contains all targeted analytes and may be prepared from the same standards as the calibration standards. See prep SOPs for specific Spiking Standards.

Table 10.2 - Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	§ Concentrated reference solution purchased directly from approved vendor	§ Manufacturer's recommended expiration date.	§ Manufacturer's recommended storage conditions
Intermediate and Working Standard Solutions	§ Reference solutions prepared by dilutions of the stock solution	§ 1 year from preparation if the solution is prepared from a neat. 6 months if prepared from stock solution ampule. § Working solutions must be checked frequently and replaced if degradation or evaporation is suspected.	§ Store in amber vials with Teflon lined screw caps or crimp caps at ≤ 0°C.

Table 10.3 – Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Dichloromethane (Methylene Chloride)	NA	Fisher/D151-4
Methanol	NA	Fisher/A-454-4
Sylon CT	5% DMDCS in Toluene/For inlet liner conditioning	Supelco/33065-U

Table 10.4 Calibration Stock Standard Solutions

Standard	Concentration	Vendor/ Item #
8270 Megamix	1000 µg/ml	Restek/31850
Appendix IX Mix 2	1000 µg/ml	Restek/568591
Dinoseb	1000 µg/ml	Restek/32251
Decane	1000 µg/ml	Spex/S-1112
Octadecane	1000 µg/ml	Spex/S-2850
Appendix IX Mix 1	2000 µg/ml	Restek/32459
Methapyrilene	2000 µg/ml	Restek/32460
Benzidine	2000 µg/ml	Restek/31834
Organophosphorus Pesticides	2000 µg/ml	Restek/32419
Benzoic Acid	2000 µg/ml	Restek/31879
Alpha-terpineol	5000 µg/ml (prepared from neat)	Sigma Aldrich/432628
SVOASurrogates	2000 µg/ml	Accustandard/CLP-031-R2

Table 10.5 Second Source Stock Standard Solutions

Standard	Concentration	Vendor/ Item #
Benzidine/3,3-Dichlorobenzidine	2000 µg/ml	Accustandard/Z-014F
Appendix IX SVOA	2000 µg/ml	Accustandard/M-8270-08
B/N Composite	2000 µg/ml	Accustandard/CLP-HC-BN-R
Appendix IX SVOA	2000 µg/ml	Accustandard/M-8270-09
Acid Composite	2000 µg/ml	Accustandard/CLP-HC-A-R
Composite 3A	2000 µg/ml	Accustandard/ CLP-HC-X1-R1
Appendix IX SVOA	2000 µg/ml	Accustandard/M-8270-07

Standard	Concentration	Vendor/ Item #
Composite 3	2000 µg/ml	Accustandard/Z-014E-R3
Benzaldehyde	2000 µg/ml	Restek/33017
Caprolactam	2000 µg/ml	Restek/31833
Atrazine	1000 µg/ml	Restek/32208
Acetophenone	5000 µg/ml	Restek/30621
1,1-Biphenyl	2000 µg/ml	Supelco/4-8161
Custom Standard	2000 µg/ml	Ultra/CUS-14098
Acids Surrogate	2000 µg/ml	Ultra/ISM-295
B/N Surrogates	5000 µg/ml	Ultra/ISM-287

Table 10.6 Miscellaneous Stock Standard Solutions

Standard	Concentration	Vendor/ Item #
GC/MS Tuning	1000 µg/ml	Restek/31615
SVOA Internal Standard	4000 µg/ml	Ultra/US-108N

11. Calibration and Standardization

11.1 Standard Sources and Intermediate Standard Preparation: Standards are prepared from commercially available multi-compound stock solutions and neat materials by multiple dilutions. The sources of the stock solutions and neat materials are found in **Tables 10.4, 10.5, and 10.6**; recipes for preparing dilutions and working standards, and concentrations for all compounds are presented in **Table 11.2**. All intermediate standards are prepared using dichloromethane and stored according to guidelines listed in **Table 10.2**.

11.2 Working Standard and Calibration Standard Preparation: Working calibration standards made for direct analysis on the GC/MS are made in dichloromethane. Depending on the volume of each solution needed, the standards are brought to volume in volumetric flasks or prepared in smaller glass vials and brought to volume by additions of solvent with micro syringes. The calibration standards are made in a volumetric fashion. The individual standards can be prepared according to the details provided in **Table 11.2**. All preparations of Intermediate and Working Standards must be recorded in the Standard Prep Logbook or logged into the LIMS system.

Table 11.2 – Working Standard Dilutions and Concentrations

Standard	Standard Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Tune Intermediate	250 µl	Dichloromethane	4750 µl	5 ml	50 ng/µl
Internal Standard	2 ml	Dichloromethane	2 ml	4 ml	2000 ng/µl
Calibration Std Intermediate (Prepared from individual Stock Standards)	100/250/500 µl*	Dichloromethane	900 µl	5 ml	100 ng/µl
Working Calibration Standards (Prepared from Calibration Std Intermediate):					
Calibration Std 1	10 µl	Dichloromethane	990 µl	1 ml**	1 ng/µl
Calibration Std 2	50 µl	Dichloromethane	950 µl	1 ml**	5 ng/µl
Calibration Std 3	100 µl	Dichloromethane	900 µl	1 ml**	10 ng/µl
Calibration Std 4	250 µl	Dichloromethane	750 µl	1 ml**	25 ng/µl

Standard	Standard Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Calibration Std 5	400 µl	Dichloromethane	600 µl	1 ml**	40 ng/µl
Calibration Std 6	600 µl	Dichloromethane	400 µl	1 ml**	60 ng/µl
Calibration Std 7	800 µl	Dichloromethane	200 µl	1 ml**	80 ng/µl
Continuing CalibrationVerification	250 µl	Dichloromethane	750 µl	1 ml**	25 ng/µl
Second Source Verification Standard	5/12.5/25 µl*	Dichloromethane	807.5 µl	1 ml**	25 ng/µl

***100 µl of each Stock Standard at 5000 µg/ml; 250 µl of each Stock standard at 2000 µg/ml; 500 µl of each Stock standard at 1000 µg/ml. For Second Source Verification the amounts are 5 µl, 12.5 µl, and 25 µl.**

**** 10 µl of Internal Standard at 2000 ng/µl is added to each 1 ml Final Volume for a final concentration of 20 ng/µL of Internal Standard.**

11.3 Tune Verification – The mass spectrometer tune status must be verified prior to initial calibration and at the beginning of each analytical sequence. Refer to **Section 12.2** for details on the analysis and evaluation of this standard.

11.4 Initial Calibration

11.4.1 **Analysis of Standards**: An initial calibration curve must be analyzed using a minimum of five points. The lowest concentration must be at or below the equivalence of the standard reporting limit. The lowest calibration point reflects the practical quantitation limit for that compound, a level below which all reported results must be qualified as estimated values. Refer to **Table 11.2** for standard concentrations.

11.4.2 An analyte must be present and the calibration curve in control in order to be reported on the target analyte list. Analytes identified by mass spectral match but not present and in control in the calibration table may be reported as Tentatively Identified Compounds (TICs). Guidelines for identification are listed in **Section 14.1.3**. Quantitative results for TICs may be reported provided they are qualified as estimated values.

11.4.3 Calibration Response Factors: Response factors (RF) establish the relationship of the instrument response in comparison with the concentration of any given analyte. The RF includes the concentration and response of the internal standard as well. By relating the IS concentration and response in an inverse manner, the target analyte concentration is adjusted to account for drift in the instrument on a per injection basis. As instrument response increases as indicated by the response of the internal standard, the concentration of the target is mathematically decreased, and vice versa.

11.4.4 To calculate the RF for any given calibration standard (or calibration verification standard), tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standards selected for the calculation of the RF for targeted compounds are listed in **Table 4**. For any compound not listed in this table, the internal standard selected for the calculation should be the internal standard that has a retention time closest to the compound being measured. Response factors are calculated using the following equation:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (µg/L).
C_x = Concentration of the compound being measured (µg/L).

11.4.5 Minimum Response Factor: A minimum response for targeted analytes, as displayed in **Table 3**, should be achieved to demonstrate that these analytes are responding as expected. Meeting the minimum response criteria for the lowest calibration standard is also important in demonstrating desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet these criteria. The failing compounds may not be critical to the specific project and therefore may be used as qualified data or as estimated values.

11.4.6 Calibration Curve Fit: The calibration curve is a representation of the relationship of the instrument response and analyte concentration. The curve is used to quantitate the concentration of an unknown based on its response and this known relationship. The curve is produced in several ways depending on the nature of the "goodness of fit".

11.4.6.1 Average Response Factor: The average response factor is determined by averaging the response factors calculated for each calibration level for each target analyte. The average RF can be used to calculate the concentration of target analytes in samples provided the criteria are met for consistency in the RFs for any given analyte. Average response factor is the default curve fitting option for calibrations. It is in the most basic sense, a linear regression that is forced through zero at the origin. Because of its simplicity and the interception of the y axis at the origin, this is the preferred technique for curve fitting. The ChemStation software has an automatic function for the calculation of the percent relative standard deviation (%RSD) used to determine the acceptability of the use of the Average RF:

$$\%RSD = SD*100 / ARF$$

Where: SD = Standard deviation of the averaged RFs for a given compound

The average response factor is also used to diagnose the integrity of the chromatography system as it relates to calibration linearity. The compounds on the target analyte list must meet specific criteria for the calibration to be acceptable. The %RSD for each compound must be $\leq$ 20%. If the %RSD of any compound exceeds the criteria an alternative calibration curve must be evaluated for usability as follows:

11.4.6.2 Linear Regression: The 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+b where "a" is the slope of the line and "b" is the y intercept. In order to use this curve fit technique, a minimum of 5 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument is linear in nature but does not necessarily intercept the y axis at the origin. However, because the 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.

11.4.6.2.1 A calculation of the correlation coefficient "r" is used to determine the acceptability of a linear regressed curve. It should be noted that the ChemStation software calculates an r^2 term as a coefficient describing correlation. This statistic should not be confused with the correlation coefficient ("r"); they are not related.

11.4.6.3 Non-linear Regression: 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. This technique works well for

calibrations where the response of the instrument gradually decreases with increasing concentrations. Using this technique, an analyst may be able to generate calibration curves with correlation coefficients very close or equivalent to 1.000. However, 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. Likewise, high levels of contamination may not be able to be calculated due to regression equations with multiple intercepts of either axis on the calibration plot.

11.4.6.3.1 A calculation of the coefficient of determination (COD) is used to determine the acceptability of a non-linear regressed curve. Either the low or high calibration points may be dropped to meet linearity criteria provided the laboratory meets the minimum 6 calibration point requirements.

11.4.7 Corrective Action for Initial Calibration

Acceptance criteria for the Calibration Curve Fit types are listed in **Table 11.5**. The following steps should be taken if the calibration curve fails to meet the criteria.

- Examine the individual levels of the calibration curve to determine if one of the standards is an outlier, in which case that standard should be re-prepared and re-analyzed.

- If non-linearity is observed across a broad range of compounds or across all calibration levels, all calibration standards should be re-prepared and re-analyzed.

- If linearity cannot be achieved after re-analyzing the calibration standards, the ion source may need to be cleaned.

11.5 Calibration Verification:

11.5.1 **Second Source Verification**: In addition to meeting the linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. Because all calibration points are from the same source, it is possible that the calibration points may meet linearity criteria but not be accurately prepared in terms of their true value. Therefore, to assess the accuracy relative to the purity of the standards, a single standard from a secondary source must be analyzed and the results obtained must be assessed relative to the known true value. This step is referred to as **Second Source Verification** or alternatively as **Initial Calibration Verification (ICV)**. This secondary source 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 difference from the true value, calculated by the ChemStation software according to the following equation:

$$\% \text{ Difference} = [\text{Result}_{ICV} - \text{TrueValue}_{ICV}] / \text{TrueValue}_{ICV} * 100$$

See **Table 11.5** for ICV acceptance criteria.

11.5.2 **Continuing Calibration Verification (CCV)**: As part of the analytical process, the instrumentation must be checked periodically to determine if the response has changed significantly since the initial calibration was established. This verification process is known as **Continuing Calibration Verification**. The validity of the initial calibration is checked at the beginning of every analytical sequence and every 12 hours thereafter for as long as the instrument is analyzing samples. This is accomplished by analyzing a midpoint calibration standard at 25 ng/µl (CCV).

11.5.2.1. The values obtained from the analysis of the CCV are compared to the true values and a percent change calculated. The actual determination of change in instrument response is based on the type of curve fit used for each analyte. Calibration curves based on an average response factor

are assessed based on the percent difference of the RF calculated for the CCV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The ChemStation software has an automatic function for the calculation of these equations as follows:

$$\% \text{ Difference: } [RF_{CCV} - AvgRF_{CAL}] / AvgRF_{CALI} \times 100$$

$$\% \text{ Drift: } [Result_{CCV} - TrueValue_{CCV}] / TrueValue_{CCV} \times 100$$

The percent change must meet the method-specified criteria for the analysis to proceed for an additional 12 hours. See **Table 11.5** for the CCV acceptance criteria.

11.5.2.2 Internal Standard Retention Time—The retention times of the internal standards in the CCV must be evaluated against the mid-point (25 ng/µl) standard of the most recent initial calibration sequence.

Table 11.5: Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Average Response Factor Linear Regression Non-linear Regression	%RSD ≤ 20% r ≥ 0.995 COD ≥ 0.99	If not met, try linear regression fit Corresponds to ChemStation r^2 value of 0.990. If not met, try non-linear regression fit 6 Calibration levels required for non-linear regression. *Exceptions:*Compounds that do not meet %RSD requirements and for whom no acceptable functions can be found may be reported with a qualifier provided the non-linearity is specific to an individual compound and not the calibration curve in general. No more than 10% of compounds should fail %RSD/alternative curve fit options.
Second Source Verification Standard	Immediately after each initial calibration	% Diff ±30%	Acceptance criteria are ±30% for all analytes. Compounds with >30%Diff should be qualified.
Continuing Calibration Verification	Prior to the analysis of any samples and every 12 hours thereafter	% Diff ±20% Internal Standard	Allowance for up to 20% of compounds to have >20%Diff. Failed compounds that have low response must be qualified. Failed compounds that have high response **and are present in samples** must be qualified. If the requirements for continuing calibration are not met, the corrective actions listed in section 11.5.5 must be taken prior to reanalysis of standards. Only two injections of the same standard are permitted back to back. ± 30 seconds from RT in the mid-point calibration level of the most recent initial

Calibration Metric	Parameter / Frequency	Criteria	Comments
		Retention Time	calibration. Allowance for retention time shifts that occur due to analyst clipping of the analytical column for maintenance.
		Internal Standard Response	–50% to +100% of the area in the mid-point standard level of the most recent initial calibration.
Minimum Response Factor	For Initial Calibration and Continuing Calibrations	See Table 3	

11.5.3 Corrective Action for Continuing Calibration Verification

11.5.3.1 Failure of %Difference and %Drift

- Reanalyze the CCV standard to determine instrument consistency. Perform and document instrument maintenance such as clipping a portion of the analytical column, replacing the inlet liner and inlet seal, and baking the column. Reanalyze the CCV standard to determine if maintenance was effective in restoring performance. If maintenance did not improve the CCV performance, prepare and analyze a new CCV standard to determine preparation consistency/standard integrity. If the CCV still does not pass, recalibration of the instrument is necessary.

- If samples were analyzed in spite of verification failures, the results must be qualified on the sample report as follows:

  § Failed compounds that have high response and are **present in samples**

  § All failed compounds that have low response

11.5.3.2 Failure of Internal Standard Retention Time

- Reanalyze the CCV standard to determine instrument consistency. Check the chromatographic system (flow, pressure) for malfunctions and make corrections as necessary. If retention times are restored reanalyzed any samples associated with the failed CCV. If retention times cannot be restored, recalibrate the instrument and reanalyze any samples associated with the failed CCV.

  Exceptions: Retention times shifts over time due to clipping of the analytical column for routine maintenance are acceptable and therefore reanalysis of samples and/or recalibration is not necessary.

11.5.3.3 Failure of Internal Standard Response

- Reanalyze the CCV standard to determine instrument consistency. Check the mass spectrometer settings for errors or malfunctions. Adjustment to the electron multiplier voltage usually can restore internal standard responses. Reanalyze any samples associated with the failed CCV. If responses cannot be restored or consistency in responses cannot be achieved, additional maintenance may be required such as cleaning the ion source or replacing the electron multiplier.

12. Procedure

12.1 Operating Parameters: Configure the GC/MS system to match the following operating parameters based on instrument configuration. The parameters themselves are saved as a method on the chromatography data system.

Table 12.1: Instruments and Operating Parameters

Instrument IDs	Component	Settings and Consumables	
HP5973N	Gas Chromatograph	See Table 9.2 for GC supplies	Flow: 0.8 ml/min Initial Temperature: 40°C Initial Time: 2min Ramp 1: 45°C/min to 110°C Hold 1 min Ramp 2: 12°C/min to 200°C Hold 0 min Ramp 3: 35°C/min Final Temperature: 320°C Final Time: 6 min Injector Temperature: 250°C Detector Temperature: 280°C
	Mass Spectrometer	Tune File: dftpp.u	
	Autosampler		Injection volume: 1 µl
HP5973R	Gas Chromatograph	See Table 9.2 for GC supplies	Flow: 1.5 ml/min Initial Temperature: 40°C Initial Time: 2min Ramp 1: 45°C/min to 110°C Hold 1 min Ramp 2: 12°C/min to 200°C Hold 0 min Ramp 3: 35°C/min Final Temperature: 320°C Final Time: 6 min Injector Temperature: 250°C Detector Temperature: 280°C
	Mass Spectrometer	Tune File: dftpp.u	
	Autosampler		Injection volume: 1 µl

12.2Tune Verification: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer tune conditions must be verified by analyzing a tuning standard containing DFTPP. GC column performance and injection port inertness should also be verified and this is done simultaneously by the inclusion of DDT, benzidine and pentachlorophenol in the tuning standard. DDT is used to verify breakdown conditions; benzidine and pentachlorophenol are used to check for tailing due to system activity.

12.2.1 After the analysis of this standard, the mass spectrum of DFTPP must be evaluated against the following criteria:

Mass (m/z)	Ion Abundance criteria
51	30.0-60.0% of m/z 198
68	<2.0% of m/z 69
69	Present
70	<2.0% of m/z 69
127	40.0-60.0% of m/z 198
197	<1.0% of m/z 198
198	Base peak, 100% relative abundance
199	5.0-9.0% of m/z 198
275	10.0-30.0% of m/z 198
365	>1.0% of m/z 198
441	Present but less than m/z 443
442	40.0-110.0% of m/z 198
443	17.0-23.0% of m/z 442

The analytical software has a program for obtaining the DFTPP spectra and evaluating the response ratios.

12.2.1.1 Evaluate the ion ratios against the criteria listed above. If the ratios meet the criteria, analysis may proceed for 12 hours. The window for analysis is 12 hours from the injection date / time of the DFTPP tune verification. After 12 hours the tune must be verified again to

establish a new analytical window. The same Ion Abundance Criteria used for the DFTPP tune must be used for all subsequent analyses associated with that initial calibration.

Note: Because analysis using different analytical methods (8270D, 625, and ASP) commonly occurs within the same 12 hour sequence, the most stringent tune criteria of these methods is followed and is listed above in Table 12.2.1.

12.2.1.2 If the ratios do not meet the criteria, refer to the following corrective actions to address the problem. Any changes made to the system must be followed with the reanalysis of a tune verification standard. Any maintenance performed on the physical mass spectrometer components requires recalibration. “Autotunes” and adjustments to the mass spec parameters may be performed without recalibration provided the subsequent CCV meets all criteria for response, retention time and sensitivity.

- Reinject the tuning standard to determine instrument consistency.
- Adjustment to the repeller often helps to optimize response for mass 51 and mass 442. Other adjustments to the mass spectrometer components may be done based on analyst experience with each individual instrument and how the mass spectrometer responds to such adjustments.
- If adjustment to the mass spectrometer components fails to produce a passing tune, cleaning of the ion source may be required.

12.2.2 Tailing Factor Verification-Benzidine and pentachlorophenol should be present at their normal responses and should not exceed a tailing factor of 2 given by the equation displayed in **Figure 1**. The ChemStation software has an automatic function to calculate the tailing factor using this equation.

- Excessive tailing may be improved by baking the column or by replacing the inlet liner and inlet seal.

12.2.3 Breakdown Verification- The GC/MS system must be sufficiently inert such that DDT will not breakdown excessively while in the injection port. The inertness is assessed by calculating the percent breakdown of DDT into the products DDD and DDE. The calculation is performed as follows:

$$\%\ \text{Breakdown} = \frac{\text{(Responses of DDD + DDE)}}{\text{(Responses for DDT+ DDD+DDE)}} \times 100$$

The ChemStation software has an automatic function to calculate % Breakdown using this equation.

12.2.3.1 The % breakdown should not exceed 20%. If the breakdown of DDT exceeds this amount, corrective action should be taken prior to analysis of samples. Replacing the inlet liner and the inlet seal should improve breakdown.

12.3 Calibration Verification: After the instrument tune conditions are verified and the system meets tune criteria, the instrument must undergo calibration verification. Analyze a Continuing Calibration Verification (CCV) standard following the procedure and criteria listed in **Section 11.5.2** and **Table 11.5** to assess the Continuing Calibration Verification and determine the current calibration status.

12.3.1 If the CCV meets control criteria, the system is deemed to be in control and analysis of samples may commence. If the CCV does not meet control criteria, follow the corrective action procedures listed **Section 11.5.3**.

12.4 Sample Preparation- Water Samples: Aqueous samples are prepared according to EPA 3510C or 3520C. These procedures are contained in a separate standard operating procedure. Refer to these separate SOPs for details on the preparation of aqueous samples.

12.4.1 Spike the 1 ml sample extracts with 10 µl of internal standard for a final concentration of 20 µg/ml. NOTE: Although most extracts for aqueous samples have a final volume of 1 ml which requires 10 µl of internal standard, due to limited volume some samples may have a final volume 0.5 ml and therefore should be spiked with 5 µl of internal standard.

12.5 Sample Preparation- Soil Samples: Soil samples are prepared according to EPA 3545A. This procedure is contained in a separate standard operating procedure. Refer to this SOP for details on the preparation of soil or solid samples.

12.5.1 Spike the 1 ml sample extracts with 10 µl of internal standard for a final concentration of 20 µg/ml. NOTE: If the soil extracts undergo GPC cleanup they will have a final volume of 0.5 ml and therefore should be spiked with 5 µl of internal standard.

12.6 Dilutions Dilutions must be made on sample extracts that have compounds that exceed the calibration range. The dilutions are prepared in a volumetric fashion. Sample aliquots should be taken in volumetric syringes and brought to volume by the addition of dichloromethane via an appropriate syringe. In the event a dilution is made to bring target analytes into calibration range, the analyst should prepare the dilution such the response of the major constituents fall in the upper half of the linear range of the curve.

- If dilutions are made on extracts that already contain internal standards, a proportional aliquot of internal standard solution must be added to the diluted extract based on the volume of diluent used so that the concentration of internal standard is 20 ng/µl.

12.7 Sample Analysis.

Extracts for quality control samples (method blanks and spiked blanks) and samples are loaded onto the autosampler and their corresponding IDs are entered into the analytical software sequence. 1 µl of each extract is sequentially injected and data from the injections are collected on the data system with Chemstation software and processed with Enviroquant software into a data file. Injections may continue within a 12-hour sequence beginning with the first injection of the Tune Verification.

Each data file is subjected to qualitative and quantitative analysis by the analytical software as well as by the analyst as described in Section 14. Each data file for quality control samples and samples is reviewed by the analyst for Quality Control requirements as described in Section 13.

13. Quality Control

Table 13.1 – Batch Quality Control Criteria

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples	Target analytes must be less than reporting limit.	Re-analyze blank to confirm failure. Qualify results and/or re-extract associated samples. The source of the contamination should be investigated and appropriate measures taken to eliminate the problem. ***Exceptions:*** If sample ND, report sample without qualification. If sample cannot be re-extracted: Samples may be reported if the concentration of positives in the samples is not critical to the usability of the data. As a guideline, sample concentrations at or above 20X the blank level can be regarded as genuine. Any positive results reported under these conditions must be qualified.
Laboratory Fortified Blank(LFB)	Full Target List compounds	One per batch of up to 20 samples	Laboratory derived limits. (Listed in the LIMS) Marginal exceedances allowed.	Re-analyze the LFB to verify failure; If LFB passes, review samples for potential injection problems; If problem persists, check spike solution. Re-extract samples where possible if multiple compounds are below limits or if recoveries are <10%. If re-extraction is not possible, affected data must be qualified. Treatment of associated samples affected by recoveries in an LFB is subject to judgement and the effect that poor recoveries may have on sample results must be considered. ***Exceptions:*** If LFB recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike/MSD (MS/MSD)	Full Target List compounds	One per batch of up to 20 samples	Laboratory derived limits RPD limit 30% Exceedances allowed due to matrix effects.	If LFB is acceptable, the MS/MSD and associated samples may be reported with appropriate qualifiers indicating matrix interferences. No corrective action or qualification required for RPD failures.
Duplicate	Sample Dup	One per batch of 20 samples where there are positivie hits in the samples.	30% RPD Exceedances allowed due to matrix effects.	Matrix Spike Duplicate(MSD) can be used as a substitute for the Duplicate if no positive hits are found in samples or if sample history is unknown.

Table 13.2 – Sample Quality Control Criteria

Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Internal Standard	1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Added to all standards, samples, spikes, control samples, and method blanks prior to analysis	**Retention Time:** RT should be ± 30 seconds from most recent CCV in all samples. **Response: -**50%-+100% of the response in the most recent CCV.	**Retention Time Failure:** If matrix interference is NOT probable, the analytical system must be checked for source of retention time shifting; **Response Failure**: Affected samples should be reanalyzed to confirm matrix effects. (Data may be qualified without reanalysis in the case of obvious matrix interference.)
Surrogate Standards	Nitrobenzene-d5 2-Fluorobiphenyl Terphenyl-d14 Phenol-d5 2-Fluorophenol 2,4,6-Tribromophenol	Added to all samples, spikes, control samples and method blanks prior to analysis	Acceptance limits and guidelines allowing for 1 B/N surrogate and 1 Acid surrogate outside limits adapted from NYSDEC ASP. Limits are listed in the LIMS.	Re-analyze extract to confirm failure. Re-extract samples that have 2 or more B/N surrogates or 2 or more acid surrogates outside limits, or any surrogate that has <10% Recovery. Surrogate recoveries are reported to client; additional information regarding confirmation by re-extraction, explanation of matrix interference, or inability to re-extract due to limited sample volume may be conveyed to client using LIMS QC Notes.

14. Data Analysis and Calculations

14.1 Qualitative Analysis Qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity if fewer than three such ions occur in the reference spectrum.

14.1.1 Compounds are identified when the following criteria are met:

Retention Time Comparison:

- The relative retention time (RRT) of the sample component is within ±0.06 RRT units of the component in the calibration verification standard.

Mass Spectrum Comparison:

- The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

14.1.2 The following should be considered when evaluating mass spectrum comparison:

- Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times.

- Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene).

- Examination of extracted ion current profiles can aid in the qualitative identification of compounds when analytes coelute.

14.1.3 Tentatively Identified Compounds (TICs) – Identification of non-targeted compounds may be requested for some samples. A mass spectral library search is conducted to attempt assignment of tentative identifications. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

- The relative intensities of the major ions should agree within ± 30%.

- Molecular ions present in the reference spectrum should be present in the sample spectrum.

- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

14.2 Quantitative Analysis- Once a targeted analyte has been identified, the concentration of the analyte is computed by the software program with the response factors of the calibration. Quantitation is based on the integrated abundance of the target analyte's primary ion using the internal standard technique.

14.2.1 It is highly recommended to use the integration produced by the software to ensure consistent integrations. However, manual integrations may be necessary when the software does not produce proper integrations because the baseline selection is improper; the correct peak is missed; a coelution is integrated; the peak is partially integrated; etc. Manual integrations should not be substituted for proper instrument maintenance or setup of the method. The analyst is responsible for ensuring that integration is correct whether performed by the software or done manually. The analyst must refer to SOP S-LI-Q-003 (current revision) for detailed instructions on manual integrations.

14.2.2 The GC/MS data system will calculate the concentration of each analyte as µg/mL. If the initial analysis of the sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be analyzed at a higher dilution. Details on dilution procedures can be found in **Section 12.6.**

14.2.3 Results Calculation- Aqueous Samples:

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(IS)(V_t)(DF)}{(Ais)(RRF)(Vo)(Vi)}$$

14.2.4 Results Calculation- Soil/Solid Samples:

$$\text{Concentration } (\mu g/kg) = \frac{(A_x)(IS)(V_t)(DF)(2^*)}{(Ais)(RRF)(Ws)(Vi)(D)}$$

Where:

A_x = Area of character ion of analyte
A_{is} = Area of character ion of internal standard
IS = Amount of internal standard injected (ng)
RRF = Relative response factor of analyte
V_t = Final volume of extract (µL)
Vo = Initial volume of sample (mL)
V_i = Volume of extract injected (µl)
DF = Dilution factor.
W_s = Weight of soil sample extracted (g)
D = Factor for solids content

$$= \frac{100\% - \%\text{moisture}}{100\%}$$

- 2 = GPC factor (if applicable). This factor is used to account for the fact that a 5 mL aliquot (from 10 mL final volume) is subjected to GPC cleanup. This means that the final volume of extract that is analyzed and entered into the equation is representative of half the total sample.

14.2.5 Quantification of Tentatively Identified Compound(TICs)

- The calculation for concentration is performed with the same formula as above, however, since no calibration factors are available a response factor of **1** is assumed.
- Unlike in the case of targeted analytes, the total area for all ions is determined and likewise the total area for the associated internal standard is used in the computation. The nearest internal standard that is free from interferences should be used.
- The resulting concentration represents an estimated value and therefore must be qualified on the data report.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. The analyst is responsible for generating the data and also is the initial individual to review the data. The review must include at least the following procedures (where applicable):

15.1.1. Inspection of records in run log for completeness.

15.1.2. Checks to ensure that all tuning, calibration and quality control requirements were met as listed in Section 11, Section 12 and Section 13.

15.1.3. Checks to determine consistency with client/project-specific requirements if such exists.

15.1.4. Checks to ensure that the appropriate sample preparatory and analytical SOPs and methods were followed, and that chain-of-custody and holding time requirements were met.

15.1.5. Checks for complete and accurate explanations of anomalous results, corrective action, and the use of data qualifiers in the case narrative or LIMS QC Notes.

15.1.6. Record of any non-standard condition of the test, test environment, sample or any deviation from standard operating procedure.

15.2. If analysis is deemed acceptable the data is imported into the LIMS.

15.2.1. If additional information is to be communicated to the data user about a particular sample, a "QC Note" is entered by the analyst.

15.2.2. Another review is performed for correctness of results, including prep factors, dilution factors, spike amounts and recoveries, sample and QC references and appropriate qualifiers.

15.2.3. Once data has been reviewed in the LIMS, the analyst, a second analyst, or supervisor will "QA" the sequence which indicates the data has been reviewed and is ready for reporting.

16. Corrective Actions for Out-of-Control Data

16.1. Corrective action begins with the analysts who are responsible for knowing when the analytical process is out of control.

16.2. Corrective actions should be initiated for out of control events such as when QC checks exceed acceptance limits or exhibit trending, holding time failures, loss of sample, equipment malfunctions or evidence of sample contamination.

16.3. Refer to Tables 11.5, 13.1, and 13.2 for data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Tables 11.5, 13.1, and 13.2 for data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

17.2. All problems associated with the analysis of a sample group should be outlined in the case narrative of the data package or with the use of QC Notes in the LIMS, if required.

18. Method Performance

18.1. The suitability of the method for the analytes tested was determined when the method was developed. During "method startup" in the laboratory, the capability of the analysts, suitability of equipment, materials and instrumentation was tested.

18.2. Internal method performance is established and monitored with use of the following (where applicable):

- Method Detection Limit studies
- Demonstration of Capability
- Precision and accuracy
- Measurement of sample matrix effects
- Quality Control Samples (Proficiency Testing)

19. Instrument/Equipment Maintenance/Troubleshooting

19.1 For information and guidance related to instrument/equipment maintenance, troubleshooting and computer hardware and software refer to the following documents:

Document # (latest revision)	Document Title
QAM	*Quality Assurance Quality Control Manual*
MS017	*HP Analytical CD-ROM MSD Productivity Chemstation Software*
MS044	*HP 6890 Series Gas Chromatograph Operating Manual*
MS040	*Agilent Technologies 5973 Mass Selective Detector Hardware Manual*
MS052	*HP 7673 Automatic Sampler Operating and Installation Manual*

19.2 Troubleshooting procedures also come from experience and training. Problems that the analyst encounters may be elevated to the department supervisor or other experienced analysts. Other means of problem resolution may come from review of prior related corrective action reports, instrument maintenance records, internal and external audits and lab notes.

20. Safety

20.1 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 (MSDS) are on file in the laboratory and available to all personnel. Standard solutions msut be prepared in a fume hood.

20.2 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 fume hood.

20.3 Equipment: Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones. The GC pneumatic system uses gas under high pressure. This high pressure introduces the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

20.4 The company wide Chemical Hygiene and Safety Manual contains additional information on safety.

21 Waste Management

21.1 All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste including solvents, standards, and sample extracts.

21.2 Refer to the lab's *Sample and Waste Management* SOP.

22 Pollution Prevention

22.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Use pollution prevention techniques in laboratory operation whenever feasible.

22.2 Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

22.3 Control releases from hood and bench operations and comply with discharge permits and regulations, hazardous waste identification rules, and land disposal restrictions.

22.4 The generated waste has to be disposed in a manner not to cause pollution.

22.5 All wastes deemed toxic are collected in the laboratory and are disposed by professional companies licensed in waste disposal.

22.6 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention and must be followed.

23 References

23.1 "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update III, Dec. 1996.

23.2 "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update IV, February 2007

23.3 "New York State Department of Environmental Protection Analytical Services Protocol," June 2000

23.4 "New York State Department of Environmental Protection Analytical Services Protocol," April 2005

23.5 National Environmental Laboratory Accreditation Conference (NELAC) Constitution, Bylaws and Standard, approved June 5, 2003, (EPA/600/R-04/003)

23.6 40CFR Part 136 Appendix B

23.7 2009 TNI Laboratory Accreditation Standards, EL-V1-2009.

24 Tables and Figures

Table 1

Practical Quantitation Limits(PQL) and Limits of Quantitation(LOQ)

Analyte	PQL H2O µg/L	LOQ H2O SEPF µg/L	LOQ H2O CONT µg/L	PQL Soil µg/kg	LOQ Soil µg/kg
Benzidine	5	5	n/a	330	660
3,3'-Dichlorobenzidine	5	1	10	330	330
3,3'-Dimethylbenzidine	5	5	10	330	660
1-Chloronaphthalene	5	1	5	330	130
2-Chloronaphthalene	5	1	5	330	130
Hexachlorobenzene	5	1	5	330	130
Hexachlorobutadiene	5	5	5	330	130
Hexachloroethane	5	1	5	330	130
Hexachlorocyclopentadiene	5	5	5	330	330
Hexachloropropene	5	5	5	330	130
Pentachlorobenzene	5	5	5	330	130
1,2,4-Trichlorobenzene	5	5	5	330	130
1,2,4,5-Tetrachlorobenzene	5	5	5	330	130
Bis(2-chloroethyl)ether	5	1	5	330	130
Bis (2-chloroisopropyl) ether	5	1	5	330	130
Bis(2-chloroethoxy)methane	5	1	5	330	130
4-Chlorophenylphenyl ether	5	1	5	330	130
4-Bromophenylphenyl ether	5	1	5	330	130
1,3-Dinitrobenzene	5	1	5	330	n/a
1,3,5-Trinitrobenzene	5	1	5	330	130
1,4-Naphthoquinone	5	1	5	330	330
2,4-Dinitrotoluene	5	1	5	330	130
2,6-Dinitrotoluene	5	1	5	330	130
Isophorone	5	1	5	330	130
Nitrobenzene	5	1	5	330	130
N-Nitrosodiethylamine	5	1	5	330	130
N-Nitrosodimethylamine	5	2	5	330	130
N-Nitrosodiphenylamine	5	1	5	330	130
N-Nitrosodi-n-butylamine	5	1	5	330	130
N-nitrosomethylethylamine	5	1	5	330	660
N-nitrosomorpholine	5	1	5	330	130
N-Nitrosodi-n-propylamine	5	1	5	330	130
N-nitrosopiperidine	5	1	5	330	130
N-Nitrosopyrrolidine	5	1	5	330	130
Benzyl butyl phthalate	5	1	5	330	130
Bis(2-ethylhexyl) phthalate	5	1	5	330	130
Diethyl phthalate	5	1	2	330	130
Dimethyl phthalate	5	1	5	330	130
Di-n-butyl phthalate	5	1	2	330	130
Di-n-octyl phthalate	5	1	2	330	130
2-Acetylaminofluorene	5	1	5	330	130
Acenaphthene	5	1	5	330	67
Anthracene	5	1	5	330	67
Acenaphthylene	5	1	5	330	67

Analyte	PQL H2O µg/L	LOQ H2O SEPF µg/L	LOQ H2O CONT µg/L	PQL Soil µg/kg	LOQ Soil µg/kg
Benzo(a)anthracene	5	1	2	330	67
Benzo(a)pyrene	5	1	2	330	67
Benzo(b)fluoranthene	5	1	2	330	67
Benzo(ghi)perylene	5	1	2	330	67
Benzo(k)fluoranthene	5	1	5	330	67
Chrysene	5	1	2	330	67
Dibenzo(a,h)anthracene	5	1	2	330	67
7,12-Dimethylbenzyl (a) anthracene	5	1	5	330	67
Fluoranthene	5	1	2	330	67
Fluorene	5	1	5	330	67
Indeno(1,2,3-cd)pyrene	5	1	2	330	67
Naphthalene	5	1	5	330	67
3-Methylcholanthrene	5	1	5	330	330
Phenanthrene	5	1	2	330	67
Pyrene	5	1	2	330	67
4-Chloro-3-methylphenol	5	1	5	330	130
2-Chlorophenol	5	1	5	330	130
2,4-Dichlorophenol	5	1	5	330	130
2,6-Dichlorophenol	5	1	5	330	130
2,4-Dimethylphenol	5	2	5	330	330
2,4-Dinitrophenol	10	5	5	670	670
2-Methyl-4,6-dinitrophenol	10	5	5	670	670
2-Nitrophenol	5	2	5	330	330
4-Nitrophenol	10	5	5	670	670
2-Methylphenol	5	1	5	330	130
3-Methylphenol	5	1	5	330	130
4-Methylphenol	5	1	5	330	130
Cresols, Total	5	n/a	n/a	330	n/a
Pentachlorophenol	10	5	5	670	660
Phenol	5	5	5	330	130
2,3,4,6 Tetrachlorophenol	5	1	5	330	330
2,4,5-Trichlorophenol	5	1	5	330	130
2,4,6-Trichlorophenol	5	1	5	330	130
Chlorobenzilate	5	1	2	330	130
Diallate	5	5	5	330	130
Pentachloronitrobenzene	5	1	5	330	130
Dinoseb	5	5	5	330	n/a
Atrazine	5	1	5	330	130
Disulfoton	5	1	10	330	330
Dimethoate	5	1	5	330	130
Parathion ethyl	5	1	5	330	130
Phorate	5	1	5	330	130
Thionazin	5	1	5	330	130
Aniline	5	1	10	330	130
4-Chloroaniline	5	1	10	330	130
1,2-Diphenylhydrazine	5	1	5	330	130
2-Nitroaniline	5	1	5	330	130
3-Nitroaniline	5	1	5	330	130
4-Nitroaniline	5	1	5	330	130

Analyte	PQL H2O µg/L	LOQ H2O SEPF µg/L	LOQ H2O CONT µg/L	PQL Soil µg/kg	LOQ Soil µg/kg
5-Nitro-o-toluidine	5	1	5	330	130
Carbazole	5	1	2	330	130
Diphenylamine	5	1	5	330	130
Pronamide	5	1	2	330	130
Pyridine	5	5	n/a	330	330
o-Toluidine	5	1	10	330	330
Acetophenone	5	1	5	330	130
4-Amino biphenyl	5	1	10	330	330
Aramite	5	1	2	330	130
Benzoic Acid	5	n/a	10	330	n/a
Benzyl alcohol	5	2	5	330	660
Benzaldehyde	5	1	10	330	130
1,1'-Biphenyl	5	1	5	330	130
Caprolactam	5	5	10	330	130
1,2-Dichlorobenzene, Semi-volatile	5	1	5	330	130
1,3-Dichlorobenzene, Semi-volatile	5	1	5	330	130
1,4-Dichlorobenzene, Semi-volatile	5	1	5	330	130
Dibenzofuran	5	1	5	330	130
p-Dimethylaminoazobenzene	5	1	5	330	n/a
Ethyl methanesulfonate	5	1	5	330	130
Isosafrole	5	1	5	330	130
Methyl methanesulfonate	5	1	5	330	130
2-Methylnaphthalene	5	1	5	330	130
2-Picoline	5	5	10	330	130
Phenacetin	5	1	2	330	130
Safrole	5	1	5	330	130
O,O,O-Triethyl phosphorothioate	5	1	5	330	130

TABLE 2
Characteristic Ions for Semivolatile Compounds

Analyte	Primary Ion	Secondary Ion	Tertiary Ion
N-Nitrosodimethylamine	74	42	44
Pyridine	79	52	51
2-Aminopyridine	94	67	41
2-Picoline	93	66	92
Aniline	93	66	65
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (IS)	152	115	
1,4-Dichlorobenzene	146	148	113
Benzyl alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
N-Nitrosomethylethylamine	42	88	43
Bis(2-chloroisopropyl)ether	45	77	79
Thiophenol	66	109	84
Methyl methanesulfonate	80	79	65
N-Nitrosodi-n-propylamine	70	42	130
Hexachloroethane	117	201	199
Nitrobenzene	77	123	65
Isophorone	82	138	
N-Nitrosodiethylamine	102	42	57
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Bis (2-chloroethoxy)methane	93	95	123
Benzoic acid	105	77	122
2,4-Dichlorophenol	162	164	98
Ethyl methanesulfonate	79	109	97
1,2,4-Trichlorobenzene	180	182	145
Napthalene-d8 (IS)	136	68	
Napthalene	128	129	127
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	
2-Methylphenol	108	107	79
Hexachloropropene	213	211	215
Hexachlorocyclopentadiene	237	235	272
N-Nitrosopyrrolidine	100	41	42
Acetophenone	105	77	120
3-4-Methylphenol	108	107	77
2,4,6-Trichlorophenol	196	198	200
o-Toluidine	106	107	77
1,1-Biphenyl	154	153	76
2-Chloronaphthalene	162	164	127
N-Nitrosopiperidine	42	114	55
1,4-Phenylenediamine	108	80	53

TABLE 2(continued)

Characteristic Ions for Semivolatile Compounds

Analyte	Primary Ion	Secondary Ion	Tertiary Ion
1-Chloronaphthalene	162	127	164
2-Nitroaniline	65	92	138
Dimethyl phthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	89	121
3-Nitroaniline	138	108	92
Acenaphthene-d10 (IS)	164	162	160
Acenapthene	153	152	154
2,4-Dinitrophenol	184	63	154
2,6-Dichlrophenol	162	164	198
4-Chloroaniline	127	129	
Isosafrole	162	131	104
Dibenzofuran	168	139	
2,4-Dinitrotoluene	165	63	182
4-Nitrophenol	109	139	65
2-Naphthylamine	143	115	116
1,4-Naphthoquinone	158	102	76
Diethyl phthalate	149	177	150
Fluorene	166	165	167
N-Nitroso-di-n-butylamine	84	57	41
4-Chlorophenyl phenyl ether	204	206	141
4,6-Dinitro-2-methylphenol	198	121	105
N-nitrosodiphenylamine/Diphenylamine	169	168	77
Safrole	162	104	131
1,2,4,5-Tetrachlorobenzene	216	214	218
1-Naphthylamine	143	115	116
1,2-Diphenylhydrazine	77	105	182
4-Bromophenyl phenyl ether	248	250	141
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	97
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
5-Nitro-o-toluidine	152	77	106
Thionazine	97	96	107
4-Nitroaniline	138	92	108
Phenanthrene-d10 (IS)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
1,3-Dinitrobenzene	168	75	92
Sulfotepp	97	322	202
Diallate	86	43	234
Pentachlorobenzene	250	252	248
Pentachloronitrobenzene	237	142	295

TABLE 2(continued)

Characteristic Ions for Semivolatile Compounds

Analyte	Primary Ion	Secondary Ion	Tertiary Ion
4-Nitroquinoline-1-oxide	174	101	128
Di-n-butyl phthalate	149	150	104
2,3,4,6-Tetrachlorophenol	232	230	131
Isodrin	193	195	263
Fluoranthene	202	101	100
1,3,5-Trinitrobenzene	75	74	213
Benzidine	184	92	185
Trifluralin	306	43	264
Pyrene	202	101	100
Phorate	75	121	97
Phenacetin	108	109	179
Dimethoate	87	93	125
4-Aminobiphenyl	169	168	170
a.a-Dimethylphenethylamine	58	91	65
Pronamide	173	175	145
Dinoseb	211	163	147
Disulfoton	88	97	186
Butyl benzyl phthalate	149	91	206
Methyl parathion	109	125	263
Benzo(a)anthracene	228	229	226
Chrysene-d12 (IS)	240	120	236
3,3-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Kepone	272	274	237
Parathion	109	97	291
Bis (2-ethylhexyl) phthalate	149	167	279
3,3-Dimethylbenzidine	212	106	196
Methapyrilene	58	97	191
Di-n-octyl phthalate	149	150	
Aramite	185	191	319
p-Dimethylaminoazobenzene	120	77	225
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Chlorobenzilate	139	251	111
Famphur	218	125	93
Benzo(a)pyrene	252	253	125
Perylene-d12 (IS)	264	260	265
7,12-Dimethylbenz(a)anthracene	256	241	239

TABLE 2 (continued)

Characteristic Ions for Semivolatile Compounds

Analyte	Primary Ion	Secondary Ion	Tertiary Ion
2-Acetylaminofluorene	181	180	223
3-Methylcholanthrene	268	252	253
Indeno(1,2,3-cd)pyrene	276	138	
Dibenzo(a,h)anthracene	278	139	279
Benzo(g,h,I)perylene	276	138	
2-Fluorobiphenyl (surr.)	172	171	
2-Fluorophenol (surr.)	112	64	
Nitrobenzene-d5 (surr.)	82	128	54
Phenol-d5 (surr.)	99	42	71
Terphenyl-d14 (surr.)	244	122	212
2,4,6-Tribromophenol (surr.)	330	332	141
2-Chlorophenol-d4 (surr.)	132	68	134

Table 3

Minimum Response Factors(RF)

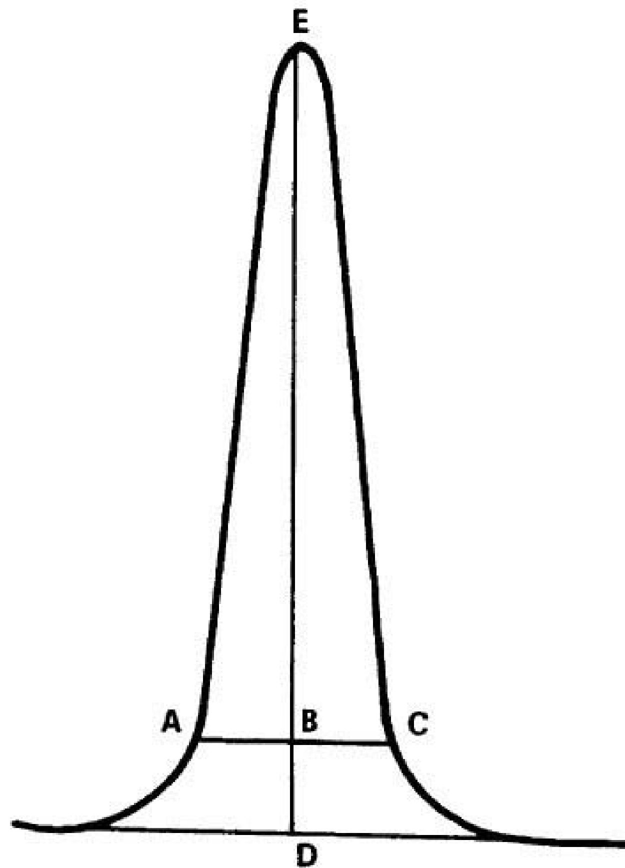
Semivolatile Compound	Minimum Response Factor (RF)	Semivolatile Compound	Minimum Response Factor (RF)
Benzaldehyde	0.01	Diethylphthalate	0.01
Phenol	0.8	1,2,4,5-Tetrachlorobenzene	0.01
Bis(2-chloroethyl)ether	0.7	4-Chlorophenyl-phenyl ether	0.4
2-Chlorophenol	0.8	Fluorene	0.9
2-Methylphenol	0.7	4-Nitroaniline	0.01
2,2-Oxybis-(1-chloropropane)	0.01	4,6-Dinitro-2-methylphenol	0.01
Acetophenone	0.01	4-Bromophenyl-phenyl ether	0.1
4-Methylphenol	0.6	N-Nitrosodiphenylamine	0.01
N-Nitroso-di-n-propylamine	0.5	Hexachlorobenzene	0.1
Hexachloroethane	0.3	Atrazine	0.01
Nitrobenzene	0.2	Pentachlorophenol	0.05
Isophorone	0.4	Phenanthrene	0.7
2-Nitrophenol	0.1	Anthracene	0.7
2,4-Dimethylphenol	0.2	Carbazole	0.01
Bis(2-chloroethoxy)methane	0.3	Di-n-butyl phthalate	0.01
2,4-Dichlorophenol	0.2	Fluoranthene	0.6
Napthalene	0.7	Pyrene	0.6
4-Chloroaniline	0.01	Butyl benzyl phthalate	0.01
Hexachlorobutadiene	0.01	3,3'-Dichlorobenzidine	0.01
Caprolactam	0.01	Benzo(a)anthracene	0.8
4-Chloro-3-methylphenol	0.2	Chrysene	0.7
2-Methylnaphthalene	0.4	Bis(2-ethylhexyl)phthalate	0.01
Hexachlorocyclopentadiene	0.05	Di-n-octyl phthalate	0.01
2,4,6-Trichlorophenol	0.2	Benzo(b)fluoranthene	0.7
2,4,5-Trichlorophenol	0.2	Benzo(k)fluoranthene	0.7
1,1-Biphenyl	0.01	Benzo(a)pyrene	0.7
2-Chloronaphthalene	0.8	Indeno(1,2,3-cd)pyrene	0.5
2-Nitoaniline	0.01	Dibenzo(a,h)anthracene	0.4
Dimethylphalate	0.01	Benzo(g,h,I)perylene	0.5
2,6-Dinitrotoluene	0.2	2,3,4,6-Tetrachlorophenol	0.01
Acenaphthylene	0.9		
3-Nitroaniline	0.01		
Acenaphthene	0.9		
2,4-Dinitrophenol	0.01		
4-Nitrophenol	0.01		
Dibenzofuran	0.8		
2,4-Dinitrotoluene	0.2		

Table 4

INTERNAL STANDARDS ASSIGNED FOR QUANTITATION OF SEMIVOLATILE ANALYTES		
1,4-Dichlorobenzene-d4	**Naphthalene-d8**	**Acenapthene-d10**
Pyridine	Nitrobenzene	1,2,4,5-Tetrachlorobenzene
N-Nitrosodimethylamine	Nitrobenzene-d5 (surr.)	Hexachlorocyclopentadiene
2-Picoline	N-Nitrosopiperidine	2,4,6-Trichlorophenol
N-Nitrosomethylethylamine	Isophorone	2,4,5-Trichlorophenol
Methyl methanesulfonate	2-Nitrophenol	2-Fluorobiphenyl (surr.)
2-Fluorophenol (surr.)	2,4-Dimethylphenol	Isosafrole
N-Nitrosodiethylamine	Benzoic acid	1,1-Biphenyl
Ethyl methanesulfonate	Bis (2-chloroethoxy)methane	2-Chloronaphthalene
Thiophenol	O,O,O-Triethylphosphorothioate	1-Chloronaphthalene
Aniline	2,4-Dichlorophenol	2-Nitroaniline
Phenol-d5 (surr.)	1,2,4-Trichlorobenzene	1,4-Naphthoquinone
Benzaldehyde	Alpha Terpineol	1,3-Dinitrobenzene
Pentachloroethane	Napthalene	Dimethyl phthalate
Phenol	4-Chloroaniline	Acenaphthylene
Bis(2-chloroethyl)ether	2,6-Dichlorophenol	2,6-Dinitrotoluene
Decane	Hexachloropropene	3-Nitroaniline
2-Chlorophenol-d4(surr.)	a,a-Dimethylphenethylamine	Acenapthene
2-Chlorophenol	Hexachlorobutadiene	2,4-Dinitrophenol
2-Aminopyridine	N-Nitroso-di-n-butylamine	4-Nitrophenol
1,3-Dichlorobenzene	Caprolactam	Dibenzofuran
Benzyl alcohol	4-Chloro-3-methylphenol	Pentachlorobenzene
1,4-Dichlorobenzene	Safrole	2,4-Dinitrotoluene
1,2-Dichlorobenzened4(surr.)	2-Methylnaphthalene	1-Naphthylamine
1,2-Dichlorobenzene	1-Methylnaphthalene	2-Naphthylamine
2-Methylphenol	p-Phenylenediamine	2,3,5,6-Tetrachlorophenol
Bis(2-chloroisopropyl)ether		2,3,4,6-Tetrachlorophenol
Acetophenone		Diethyl phthalate
N-Nitrosopyrrolidine		Fluorene
N-Nitrosomorpholine		4-Chlorophenyl phenyl ether
o-Toluidine		Thionazine
3/4-Methylphenol		5-Nitro-o-toluidine
N-Nitrosodi-n-propylamine		4-Nitroaniline
Hexachloroethane		2,4,6-Tribromophenol (surr.)
Phenanthrene-d10	**Chrysene-d12**	**Perylene-d12**
1,2-Diphenylhydrazine	Benzidine	Di-n-octyl phthalate
4,6-Dinitro-2-methylphenol	Pyrene	Benzo(b)fluoranthene
N-nitrosodiphenylamine/Diphenylamine	Terphenyl-d14 (surr.)	Benzo(k)fluoranthene
Sulfotepp	Aramite	7,12-Dimethylbenz(a)anthracene
Diallate	p-Dimethylaminoazobenzene	Benzo(a)pyrene
Phorate	Chlorobenzilate	3-Methylcholanthrene
4-Bromophenyl phenyl ether	3,3-Dimethylbenzidine	Indeno(1,2,3-cd)pyrene
1,3,5-Trinitrobenzene	Butyl benzyl phthalate	Dibenzo(a,h)anthracene
Phenacetin	2-Acetylaminofluorene	Benzo(g,h,I)perylene
Hexachlorobenzene	Benzo(a)anthracene	
Dimethoate	3,3-Dichlorobenzidine	
Atrazine	Chrysene	
4-Aminobiphenyl	Bis (2-ethylhexyl) phthalate	
Pentachlorophenol	Famphur	
Pentachloronitrobenzene	Kepone	
Phenanthrene		
Anthracene		
Octadecane		
Disulfoton		
Dinoseb		
Methyl parathion		
Carbazole		
Di-n-butyl phthalate		
Pronamide		
4-Nitroquinoline-1-oxide		
Parathion		
Methapyrilene		
Isodrin		
Fluoranthene		

Figure 1

Tailing Factor Calculation



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

25 Revisions

Document Number	Reason for Change	Date
S-LI-O-014-rev.00	Transition to PACE format.	5/10/15

APPENDIX P2-5
SOP FOR THE DETERMINATION OF GASOLINE RANGE AND DIESEL RANGE ORGANICS BY GC/FID BY SW-846 METHOD 8015D
(S-LI-O-007-REV.01)

STANDARD OPERATING PROCEDURE

DETERMINATION OF GASOLINE RANGE AND DIESEL RANGE ORGANICS BY GC/FID

Reference Methods: EPA METHOD 8015D

Local SOP Number:	S-LI-I-007-rev.01
Effective Date:	Date of Final Signature
Supersedes:	S-LI-I-007-rev.00

APPROVALS

	7/23/15
General Manager	Date
	7/23/15
Quality Manager	Date
	7/23/15
Department Manager	Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Identification of Test Method

This Standard Operating Procedure (SOP) documents the procedures used by PASI – Long Island to determine the concentration of Gasoline Range Organics (GRO) and Diesel Range Organics (DRO) in environmental samples. The laboratory utilizes GC/FID and bases these documented procedures on those listed in EPA SW-846 Method 8015D**.**

1.1. For work governed by the NYS DEC Analytical Service Protocol (ASP), the requirements for analysis and reporting of the DEC ASP have to be met.

1.1.1. For reporting of data packages with full documentation according to ASP requirements, all raw data have to be included, and summary tables of calibrations and Q. C. data have to be submitted on forms as specified in the DEC ASP.

1.2. In addition to the requirements of this SOP, the guidelines in the *Quality Assurance, Quality Control Manual* have to be observed.

2. Summary of Method

2.1. This method provides gas chromatographic conditions for the detection of DRO and GRO compounds.

2.2. Depending on the analytes of interest, samples may be introduced into the GC by a variety of techniques, including:

2.2.1. Purge-and-trap (Method 5030C, 5035A)

2.2.2. Direct injection following solvent extraction (Methods 3510C, 3545A. Other appropriate techniques may be employed with state approval).

2.3. An appropriate column and temperature program are used in the gas chromatograph to separate the organic compounds. Detection is achieved by a flame ionization detector (FID).

3. . Scope and Application

3.1. This method is used to determine concentrations of GRO and DRO from many types of water samples, soil samples, and wastes.

3.2. Method 8015D defines GRO as corresponding to the range of alkanes from C6 to C10 and covering a boiling point range of approximately 60°C - 170°C. DRO corresponds to the range of alkanes from C10 to C28 and covering a boiling point range of approximately 170°C - 430 °C.

3.2.1. The ranges listed in TNI’s Field of Proficiency Testing for Solid and Chemical Materials for GRO are C5 – C10.

3.2.2. The Lab is to report the carbon range that is appropriate for the project’s data quality objectives.

3.2.3. All results must clearly indicate the range and identify the type of reference standard used for quantification.

3.3. Practical quantitation limits (PQL) are provided in Table 1.

3.4. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC/FID systems and interpretation of data and complex chromatograms. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.5. This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

4. Applicable Matrices

4.1. The method is intended for the analysis of GRO and DRO compounds in water, soil/sediment, oil, sludge, and solid waste materials.

5. Limits of Detection and Quantitation

5.1. Reporting limits are presented in **Table 1**, "Practical Quantification Limits" (PQL).

5.2. Reporting limits must be determined at the start of a project and reporting conventions must be established with the client. Project specific requirements must be communicated to analysts prior to sample analysis.

5.3. MDL studies are performed annually by the analysis of seven low level standards at three to five times the expected MDL and calculated by the procedure defined in 40CFR Part 136 Appendix B.

5.4. Current Method Detection Limits (MDLs) are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary.

6.2 When analyzing for volatile organics, samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and subsequent storage and handling will serve as a check on such contamination.

6.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample syringe or purging device must be rinsed out between samples with an appropriate solvent. Whenever an unusually concentrated sample is encountered, it should be followed by injection of a solvent blank to check for cross contamination.

6.3.1 Clean purging vessels with a detergent solution, rinse with distilled water, and then dry in a 105°C oven between analyses. Clean syringes or autosamplers by flushing all surfaces that contact samples using appropriate solvents.

6.3.2 All glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water.Drain the glassware and dry it in an oven at 130°C for several hours or rinse it with methanol and drain. Store dry glassware in a clean environment.

6.4 The flame ionization detector (FID) is a non-selective detector. There is a potential for many non-target compounds present in samples to interfere with this analysis. There is also the potential for analytes to be resolved poorly, especially in samples that contain many analytes. The data user should consider this and may wish to alter the target analyte list accordingly.

7. Sample Collection, Preservation, Shipment and Storage

MATRIX	COLLECTION	PRESERVATION	STORAGE	HOLDING TIME
Aqueous (DRO)	One 1L amber glass	10% Sodium Thiosulfate if chlorine is present	≤6°C	7 days
Soil/Solid (non-aqueous) (DRO)	One 4oz wide glass jar	None	≤6°C	14 days
Extracts (DRO)	2mL autosampler vial	None	≤ 4°C	40 days
Aqueous(GRO)	2-40mL vials w/Teflon septa caps and no headspace	1:1 HCl to pH<2	≤6°C	14 days
Soil/Solid (non-aqueous) (GRO)	Sample is extruded into 3-40mL empty sealed vials 2 w/stir bars and 1 bulk 2 oz jar cooled to 4°C ± 2° C for no more than 48 hours then frozen to < -7°C upon receipt at laboratory.	None	frozen to < -7°C	14 days

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary.

9. Equipment and Supplies

9.1. **Table 9.1- Instrumentation**

Equipment	Vendor	Model / Version	Description / Comments
Gas chromatograph	Perkin Elmer	AutoSystem	FID
Data System	Perkin Elmer Total Chrome	V6.3.1	
Autosampler	Varian	Archon	
Purge and Trap	Tekmar	3000	

9.2. **Table 9.2 - Chromatography Supplies**

Item	Vendor	Model / ID	Catalog #	Description
Rtx-35 (DRO)	Restek	Rtx-35	10439	30 m x 0.32 mm ID x 0.5µm df
Rtx-502.2 (GRO)	Restek	Rtx-502.2	10908	30m x 0.53 mm ID x 3 µm df
Inlet Liner	Perkin Elmer	Uniliner	20838	Open Top Uniliner w/wool
Analytical Trap	Supelco	Purge Trap K Vocarb 3000	21066-U	Purge Trap
FID Jet	Perkin Elmer	Jet		

9.3. **Table 9.3 - Equipment and Supplies for GRO Preparation**

Supply	Description	Vendor/ Item # / Description
Vials - 40 mL with Teflon lined septum caps	Preserved with 1:1 HCl	
Jar - 2 oz. wide mouth, with Teflon lined cap		
Micro-syringes		
Balance-capable of weighing 100 g ± 0.1 g		
Volumetric flasks - 50 mL, 100 mL		
Spatula	narrow, stainless steel	
Stir Bar	Teflon	

9.4. **Table 9.4 - Equipment and Supplies for Sample Preparation for DRO**

Supply	Description	Vendor/ Item # / Description
Separatory funnel-2000 mL Teflon with Teflon stopcock		
Weighing dishes - aluminum		
Micro-syringes		
Balance-capable of weighing 100 g ± 0.01 g	Top loading	
Kuderna-Danish apparatus, comprising:		
Receiving vial - 10 mL		
Evaporative flask - 500 mL		
Snyder column - three ball, macro		

Supply	Description	Vendor/ Item # / Description
Snyder column - three ball micro (optional)		
Clips - for 19/22 and 24/40 joints		
Spatula	Wood, disposable	
Glass funnels		
Boiling chips - Teflon		
Water bath		
Nitrogen evaporator - 12 position N-EVAP		Organomation or equivalent
Vials	calibrated for 10 mL	
Glass wool		
Auto sampler vials	2 mL amber or clear with Teflon	
Pasteur pipets	disposable, 5.5 inch	
pH paper	wide range	
Nitrogen	instrument grade, with dual stage	
Automated Nitrogen evaporator	Turbo Vap II from	Biotage
TurboVap Tubes	200 mL for Turbo Vap II	
ASE 350		
Microwave Extractor		

10. Reagents and Standards

10.1. **Table 10.1 – Reagents and Stock Standards**

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
De-ionized (DI) Water	ASTM Type I	N/A
Sodium sulfate	granular anhydrous reagent grade, heated in kiln for at least four hours at 400°C	
Methylene chloride	pesticide grade	
Acetone	pesticide grade	
Methanol	pesticide grade	
Sodium hydroxide solution	10N, dissolve 40 g of reagent grade NaOH in 100 mL reagent water	
Surrogate solution for GRO (GC/FID)	Chlorofluorobenzene at 2000 ng/uL in methanol	
Surrogate solution for	1,4-Dichlorobenzene at 2000 µg/mL in methanol	Accustandard Cat#Z-014J-3-M-0.5X

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
extractable pet. hyd. (DRO) by GC/FID		
Matrix/ Q. C. spiking solutions for GRO	Gasoline of 5000 µg/mL in methanol	UltraScientific Cat#RGO-601
Calibration Standard for GRO	Normal Alkane Standard (GRO Defining Mix)	Accustandard Cat#GRO-AK-101-NAS
Matrix/ Q. C. spiking solutions for DRO extractions	Diesel Fuel #2 at 50 mg/mL in acetone	AccuStandard Cat# DRO-AK-102-LCS
Calibration Standard for DRO	Diesel Fuel #2 at 20 mg/mL in Methylene Chloride	AccuStandard Cat# FU-009-D-40X-PA
Surrogate Standard for Calibration	1,4-Dichlorobenzene at 2000 µg/mL in methylene chloride	UltraScientific Cat#ATS-130
Pelletized diatomaceous earth	Hydromatrix	
Acetone/methylene chloride	1:1v/v-	
Initial calibration verification standard	Diesel Fuel #2 at 50000 µg/mL in methylene chloride	Restek Cat#31258

10.2. **Table 10.2-** GRO standard preparation

Stock Standard	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
CalGRO_Lot#	25µL	H_2O	99.9mL	100mL	2500µg/L
QCGRO_Lot#	25µL	H_2O	49.9mL	50mL	2500µg/L

10.3. **Table 10.3-**GRO spiking preperation

Matrix	Stock Standard	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Aqueous	QCGRO_Lot #	23µL	Aqueous Sample	43mL	43mL	2500µg/L
Soil	Take 5mL of QCGRO solution in a 43mL VOA vial and add 5.0g of soil					

11. Calibration and Standardization

11.1. **Table 11 Calibration and Standardization**

Calibration Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	• Establish an initial 5 point calibration • When continuing calibration check standards fail. • When there is a change in procedure or instrument.	• Linear regression Correlation Coefficient ≥ 0.995. • Quadratic Correlation Factor ≥0.99 (6 points required) • Average Calibration factor <20%	• Evaluate the standards and equipment and reanalyze the initial calibration.
Initial Calibration Verification Standard (ICV)	• Immediately after the initial calibration. • Prepared from a source different than the calibration standards.	• 80-120% recovery	• Reanalyze and recalibrate if necessary. • Sample analysis cannot begin until a compliant Initial Calibration and ICV meet acceptance criteria. • Samples analyzed after a non-compliant ICV must be reanalyzed.
Continuing Calibration Verification Standard (CCV)	• Every 12 hours minimum every 20 samples • The analytical sequence must begin and end with a compliant CCV.	• 80-120% recovery	• Samples that are not bracketed with compliant CCVs are to be reanalyzed. • If a re-analysis of a CCV is still non-compliant, the standards and equipment must be evaluated, prepare new standards if necessary and recalibrate.

12. PROCEDURE

12.1. Sample Preparation

12.1.1. Preparation of Low Level/Medium Level Soil Samples and Aqueous samples for GRO - See EPA Method 5030C/5035A SOP.

12.1.2. Preparation of Aqueous samples for DRO – See EPA Method 3510C SOP for Separatory Funnel Extraction.

12.1.3. Preparation of Soil samples for DRO - See EPA Method 3545A SOP for Low Level Extraction of Soil/Sediment Samples with Accelerated Fluid Extractor (Pressurized Fluid Extraction.

12.2. Instrument Analysis Procedure

12.2.1. Setup and Parameters

- Typical operating parameters for the analytical system are presented in Tables 2A, and 2B. Optimize the GC operating conditions to achieve best resolution for all peaks .
- Make sure that conditions are not changed once calibration is performed. E.g., maintain detector makeup gas flow since it is critical for the response.
- According to the manuals, set up the auto-sampler program and data acquisition.

12.2.2. Sample Analysis

- For DRO samples, the method blank, Q. C. extracts and samples are loaded on the auto-sampler and 1 µL of each extract are sequentially injected with the same instrument parameters as the calibration standard(s). If solvent evaporation can be observed, adjust all sample and Q. C. extracts to one mL.
- For GRO samples the 40 mL vials of the method blank, Q. C. samples and client samples are loaded on the auto-sampler of the purge and trap instrument. The vials are then analyzed according to the conditions in Table 2A.
- Data of all injections are collected on the data system with the TotalChrome Software and reported in Omega.
- Instrument Blank
  - Check whether any peaks larger than the practical quantification limit are found within the retention time windows of the targeted analytes.
  - If positives are found, examine the origin of the interferences by checking a fresh blank solution, before servicing the instrument.
  - If the interference for the analytes stems from the instrument, remedial action must be taken before samples can be injected. Any samples showing positives that were injected after the last compliant instrument blank, have to be re-analyzed.

12.2.3. Retention Time Window (RTW) for GC Analysis

- Variances for the windows are established for the system, and the daily windows computed using the standard retention times as window midpoints.
- Statistically determine the expected typical window variances from at least three standard injections over a 72 hour period. (If the variance is calculated for a particular analytical sequence, include standard retention times for beginning, middle and end of sequence).
- Calculate the retention time window variances as three times the standard deviation.

Retention Time Window Variance (RTWV)

$$RTWV = 3S$$

$$S = \sqrt{\sum (RT_i - RT_{avg})^2 / (n-1)}$$

Where:
RTWV = Retention Time Window Variance
S = Standard deviation
Rti = Retention time of the ith standard analysis
Rtavg = Average retention time of the analyte
n = Number of runs

- If a window variance is less than 0.03 minutes, replace it by the default value of 0.03.
- Examine the values and modify them based on experience, if necessary. ("The experience of the analyst should weigh heavily in the interpretation of chromatograms.")
- Establish new window variances with three standard injections, if major modifications are made to the instrument, e.g. a new column is used or operating parameters are changed.

13. Quality Control

13.1. **Table 13 Quality Control Acceptance Criteria and Corrective Action Plan**

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Limit of Quantification Verification Standard (LOQ)/Reporting Limit Verification (RLV)	• Analyzed annually • Prepared at the same concentration as the lowest calibration standard to verify accuracy at the reporting level. • Processed under the same conditions as samples and goes through all the steps of the analytical procedure. • If the reporting level is higher than the lowest calibration standard, the LOQ solution may be prepared at the reporting level.	• 50-150% recovery • Or client data quality objectives, if such exist.	• Evaluate standard preparation. • Reanalyze and recalibrate if necessary.
Method Blank (MB)	• One per batch, • Or 1 per 20 samples, whichever is more frequent. • Processed with and under the same conditions as samples and goes through all the steps of the analytical procedure.	• Should not contain target analytes or interferences at concentrations that impact the analytical results for sample analyses. • Target analytes should not be greater than ½ of RL and >1/10th of the lowest sample or regulatory limit, which ever is lower.	• If the blank contains target analytes greater than the PQL, the blank needs to be re-analyzed. If the re-analysis passes criteria, then reanalyze the associated samples. • If the blank still fails, the system needs to be evaluated for the source of contamination and affected samples reanalyzed/re-extracted. • If reanalysis of samples is not possible, report data flagged to indicate method blank contamination.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Laboratory Fortified Blank (LFB)	• One per batch, • Or 1 per 20 samples, whichever is more frequent.	• In house QC Limits	• Reanalyze, if re-analysis still fails all associated samples need to be re-extracted (DRO) or reanalyzed (GRO) with a compliant LFB. • If reanalysis of the LFB is not possible, report data flagged to indicate the LFB failed recovery.
Matrix Spike/Matrix Spike Duplicate Sample (MS/MSD)	• One per 20 samples or one per batch whichever is more frequent	• In house limits	• If the recovery is outside the limits check for errors in calculation and spike preparation. • If the matrix spike/matrix spike duplicate exceeds the limits, but the LFB has acceptable recovery, then the method is in control and sample matrix effects are likely the cause. The data should be qualified in the case narrative or using QC notes in the LIMS for non-package work.

14. Data Analysis and Calculations

14.1 Identification of Analytes

14.1.1 Check that the surrogate compounds elute in the RTW for all samples and blank injections.

16.1.1 Determine the GRO and DRO range in the samples based on the retention times of the alkanes in the standard analysis.

16.1.2 Sum the areas of the peaks in the targeted ranges omitting the areas of the surrogate standards.

16.1 Quantification and Reporting of Results for GC Analysis

16.2.1 Quantify positives with the average response factors according to the equation below, using the sum of the peaks in the targeted range for the area.

Analyte Concentration (Conc)

$$\text{Conc.} = \frac{A}{RF_{med}} \times \frac{1}{V_i} \times \frac{V}{W} \times \text{—} \quad DF$$

Where:
Conc. = Concentration in sample as ug/L or ug/kg
A = Peak area
RF_{med} = Calibration factor for midpoint external standard as area per ng
Vi = Volume injected in uL
V = Extract volume (after GPC) in uL
W = Volume of sample in mL or dry weight in g
DF = Dilution factor

16.2.2 Alternatively, utilize the software for computation with a function for linear or quadratic fit.

16.2.3 If positives above the calibration range are encountered, dilute the sample to bring the concentration within the upper half of the calibration range, and analyze the diluted sample. For GRO soil samples that are above the calibration range analyze by the medium level procedure.

16.2.4 Report the undiluted and diluted analyses and submit both sets of data in the data package. For "routine reporting" without a package, report the results of the undiluted run for low analytes that did not require dilution.

16.3.1 Due to interference level from non-petroleum hydrocarbons, quantities below the quantification limit (PQL) are not reported. **Levels found in the method blank are not subtracted.**

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. The analyst is responsible for generating the data and also is the initial individual to review the data. The review must include at least the following procedures (where applicable):

15.1.1. Inspection of records in run log for completeness;

- Standard and reagent lot numbers, support equipment, spike amounts, calculations, dilution factors, container/bottle used for analysis, reporting limits.

15.1.2. Determination of whether the results meet the laboratory-specific quality control criteria;

15.1.3. Checks to determine consistency with client/project-specific measurement quality objectives (MQOs) if such exists;

15.1.4. Checks to ensure that the appropriate sample preparatory and analytical SOPs and methods were followed, and that chain-of-custody and holding time requirements were met;

15.1.5. Checks to ensure that all calibration and quality control requirements were met;

15.1.6. Checks for complete and accurate explanations of anomalous results, corrective action, and the use of data qualifiers in the case narrative or LIMS QC notes.

15.1.7. Record of any non-standard condition of the test, test environment, sample or any deviation from standard operating procedure.

15.2. If analysis is deemed acceptable, data will be imported into the LIMS.

15.2.1. Another review is performed for correctness of results, including prep factors, dilution factors, spike amounts and recoveries, sample and QC references and appropriate qualifiers.

15.2.2. If additional information is to be communicated to the data user about a particular sample, a “QC Note” is entered by the analyst.

15.2.3. Once data has been reviewed in the LIMS, the analyst or supervisor will “QA” the sequence which indicates the data has been reviewed and is ready for reporting.

15.3. Refer to **the TABLE 13** for data assessment and acceptance criteria for quality control measures.

15.4. Once it has been established that the quality objectives are met, the finalized data are entered into the LIMS and may be reported.

15.4.1. For all DRO and GRO samples it must be noted on the report or in the data package as to what was used to quantify the samples (ie. Diesel Fuel #2, Alkane Mix C6-C10).

16. Corrective Actions for Out-of-Control Data

16.1. Corrective action begins with the analysts who are responsible for knowing when the analytical process is out of control.

16.2. Corrective actions should be initiated for out of control events such as when QC checks exceed acceptance limits or exhibit trending, holding time failures, loss of sample, equipment malfunctions or evidence of sample contamination.

16.3. Refer to the QC and calibration **TABLE 11 and 13** for data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to the QC and calibration **TABLEs 11 and 13** for data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

17.2. All problems associated with the analysis of a sample group should be outlined in the case narrative of the data package or with the use of QC Notes in the LIMS, if required.

18. Method Performance

18.1. The suitability of the method for the analytes tested was determined when the method was developed. During “method startup” in the laboratory, the capability of the analysts, suitability of equipment, materials and instrumentation was tested.

18.2. Internal method performance is established and monitored with use of the following (where applicable):

18.2.1. Method Detection Limit studies

18.2.2. Demonstration of Capability

18.2.3. Precision and accuracy

18.2.4. Positive and negative controls

18.2.5. Measurement of sample matrix effects

18.2.6. Quality Control Samples (Proficiency Testing)

19. Instrument/Equipment Maintenance/Troubleshooting

19.1. For information and guidance related to instrument/equipment maintenance, troubleshooting and computer hardware and software refer to the following documents:

Document # (latest revision)	Document Title
QAM	*Quality Assurance Quality Control Manual*
ADMIN002	*Computers and Programs*
GC001	*HP 6890 Series Gas Chromatograph Operating Manual Volume 2.Inlets*
GC002	*HP 6890 Series Gas Chromatograph Operating Manual Volume 3. Detectors*
GC003	*HP 6890 Series Gas Chromatograph Operating Nanual Volume 1. General Information*
GC021A	*Perkin Elmer Instruments Total Chrom Workstation Chromatography Software Application Manager's Guide (Book 1of 5)*
GC021B	*Perkin Elmer Pe Nelson Division Turbochrom Workstation Application Manager's Guide (Book 2 Of 5)*
GC021C	*Perkin Elmer Instruments Total Chrom Chromatography Software Tutorial (Book 3 Of 5)*
GC021D	*Perkin Elmer Instruments Total Chrom Chromatography Software Users's Guide Volume Ii (Book 4 Of 5)*
GC021E	*Perkin Elmer Instruments Total Chrom Chromatography Software Users's Guide Volume I (Book 5 Of 5)*

19.2. Troubleshooting procedures also come from experience and training. Problems that the analyst encounters may be elevated to the department supervisor. Other means of problem resolution may come from review of prior related corrective action reports, instrument maintenance records, internal and external audits and lab notes.

20. Safety

20.1. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by personal protection and engineering measures. MSDS are available for all chemicals used in the lab and are available for review.

20.1.1. Read information and follow warnings listed on the labels of the containers of the chemicals.

20.1.2. In addition, the reference file of material safety data sheets (MSDS) should be consulted for properties of the chemicals used, to determine handling precautions.

20.2. Safety glasses, a lab coat, and disposable gloves must be worn when handling chemicals and samples.

20.3. Sample handling should be conducted in fume hoods.

20.4. The company wide Chemical Hygiene and Safety Manual contains additional information on safety.

21. Waste Management

21.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste.

21.2. Refer to the lab's *Sample and Waste Management* SOP.

22. Pollution Prevention

22.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Use pollution prevention techniques in laboratory operation whenever feasible.

22.2. Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

22.3. Control releases from hood and bench operations and comply with discharge permits and regulations, hazardous waste identification rules, and land disposal restrictions.

22.4. The generated waste has to be disposed in a manner not to cause pollution.

22.5. All wastes deemed toxic are collected in the laboratory and are disposed by professional companies licensed in waste disposal.

22.6. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

23. References

23.1. “Test Methods for Evaluating Solid Waste,” EPA/SW-846, USEPA, Water Characterization Branch, Update III, Dec. 1996.

23.2. “Test Methods for Evaluating Solid Waste,” EPA/SW-846, USEPA, Water Characterization Branch, Update IV, February 2007

23.3. “New York State Department of Environmental Protection Analytical Services Protocol,” Update IVa, January 1998

23.4. “New York State Department of Environmental Protection Analytical Services Protocol,” April 2005

23.5. National Environmental Laboratory Accreditation Conference (NELAC) Constitution, Bylaws and Standard, approved June 5, 2003, (EPA/600/R-04/003)

23.6. 40CFR Part 136 Appendix B

23.7. 2009 TNI Laboratory Accreditation Standards, EL-V1-2009.

24. Tables and Figures

TABLE 1
Practical Quantitation Limits (PQL)

Analyte	PQL (µg/L)	PQL (mg/Kg)
Gasoline Range Organics C6-C10	100	0.1
Diesel Range Organics C10-C28	100	6.7
Petroleum Hydrocarbons C10-C45 (approx.)	100	6.7

TABLE 2A

INSTRUMENT OPERATING PARAMETERS FOR GRO BY GC/FID

PURGE PROCESS

Sample Size	5 mL
Purge Time	11 min
Purge Flow	40 mL/min
Purge Temp.	Ambient for water and medium level soil, 40°C for low level soil
Desorption Flow	15 mL/min
Desorption Time	4.0 min
Desorption Temp.	180°C

GC CONDITIONS

Column	30 m long x 0.53 mm ID, 3.0 um film thickness, fused silica wide bore capillary column, RTX-502.2 Restek or equivalent
Carrier	Helium
Flow Rate	15 mL/min
Temperature Program:	Initial temp: 35°C Initial hold: 2 min Ramp 1: 9°C/min to 220°C Hold 1: 2.0 min
Detector	FID
Hydrogen flow	45 mL/min
Air flow	450 mL/min

TABLE 2B

INSTRUMENT OPERATING PARAMETERS FOR DRO BY GC/FID

Column	30 m long x 0.32 mm ID, 0.5 um film thickness, fused silica capillary column, RTX-35 Restek or equivalent
Carrier	Helium
Flow Rate	15 mL/min
Temperature Program:	Initial temp: 60°C Initial hold: 1 min Ramp 1: 12°C/min to 280°C Hold 1: 15 min
FID	300 C
Hydrogen flow	45 mL/min
Air flow	450 mL/min
Injector	220 C

25. Revisions

Document Number	Reason for Change	Date
S-LI-O-009-rev.00	Transition to PACE format. Sec.14.1 changed reporting units from ug/L to mg/L. Spike MS before preservation. Table 1 – added determinative method PQLs. Removed DoD references.	5/21/14
S-LI-O-009-rev.01	Added Table 10.2-GRO standard preparation and Table 10.3 GRO spiking preparation. Added frequency to MS /MSD in Table 13.1. Took out all definitions and added "8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary."	7/21/15

APPENDIX P3-1
SOP FOR THE SAMPLE PREPARATION FOR VOLATILE ORGANICS BY SW-846 METHOD 5030C
(S-LI-O-013-REV.00)

STANDARD OPERATING PROCEDURE

SAMPLE PREPARATION FOR VOLATILE ORGANICS

Reference Methods: Methods 5030C from SW-846

Local SOP Number:	*S-LI-O-013-rev.00*
Effective Date:	Date of Final Signature

APPROVALS

[signature]	5/12/15
General Manager	Date
[signature]	5/9/15
Quality Manager	Date
[signature]	5/12/15
Department Manager	Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. IDENTIFICATION OF TEST METHOD

1.1. This Standard Operating Procedure (SOP) documents the procedures used by PASI – Long Island, NY for the sample preparation of volatile organic compounds (VOCs) in aqueous samples and water miscible liquid samples based on EPA Method 5030C from SW-846.

1.2. In addition to the requirements of this SOP, the guidelines in the *Quality Assurance, Quality Control Manual* have to be observed.

2. SUMMARY OF METHOD

2.1. Aqueous Samples: An inert gas is bubbled through a portion of the aqueous sample at ambient temperature or an elevated temperature depending on the desired target analytes, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and back flushed with inert gas to desorb the components onto a gas chromatographic column.

2.2. High Concentration Extracts from Method 5035A: An aliquot of the methanol extract prepared in Method 5035A is combined with organic free water in the purging chamber. It is then analyzed by purge-and-trap GC or GC/MS following the normal aqueous method.

3. SCOPE AND APPLICATION

3.1 This method describes a purge-and-trap procedure for the analysis of volatile organic compounds (VOCs) in aqueous samples and water miscible liquid samples. It also describes the analysis of high concentrated soil and waste samples extracts prepared in Method 5035A. The determinative steps are found in GC Method 8015Dand GC/MS Method 8260C.

3.2 Method 5030C can be used for most volatile organic compounds that have boiling points below 200ºC and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, quantitation limits (by GC or GC/MS) in some cases are approximately ten times higher with erratic precision because of poor purging efficiency. The method is also limited to compounds that elute as sharp peaks from a coated capillary column. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. The purging efficiency can be improved for water soluble analytes, e.g. ketones and alcohols, when purging at an elevated temperature of 80ºC as compared to 20º or 40ºC.

3.3 Method 5030C, in conjunction with Method 8015D (GC/FID), may be used for the analysis of aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons, e.g. gasoline.

3.4 Water samples can be analyzed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water can be determined by direct injection of the sample into the chromatographic system or by dilution of the sample prior to the purge-and-trap process.

As with any preparative method for volatiles, samples should be screened if possible to avoid contamination of the purge-and-trap system by samples that contain very high concentrations of purgeable material above the calibration range of the low concentration method. In addition, because the sealed sample container cannot be opened to remove a sample aliquot without compromising the integrity of the sample, multiple sample aliquots should be collected to allow for screening and reanalysis.

3.5 Use of this method is restricted to use by, or under supervision of, appropriately experienced and trained laboratory analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

4. APPLICABLE MATRICES

4.1. The method is intended for the analysis of compounds in aqueous samples and water miscible liquid samples.

5. LIMITS OF DETECTION AND QUANTITATION

5.1. For requirement and procedure refer to the determinative method.

6. INTERFERENCES

6.1. Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials may out-gas organic compounds which will be concentrated in the trap during the purge operation. The compounds will result in interferences or false positives in the determinative step.

6.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipments and storage. A trip blank prepared from an appropriate organic-free matrix and sample container, and carried through sampling and handling protocols, serves as a check on such contamination.

6.3. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free reagent water is not necessary. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the entire system is required.

6.4. The laboratory where volatiles analysis is performed should be completely free of solvents. Special precautions must be taken when analyzing for methylene chloride. The analytical and sample storage areas should be isolated from all atmospheric sources of methylene chloride. Otherwise random background levels can result. Since methylene chloride can permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed can also lead to random background levels and the same precautions must be taken.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

7.1. **Table 7.1 – Sample Collection, Preservation, Storage, and Hold time**

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Two VOA vials	ALL DONE IN FIELD Acidified w/ 1:1 HCl (1-2 drops) to pH<2, no headspace AND Halogenated and aromatic compounds require preservation with thiosulfate, if samples are chlorinated. *Note:* Depending on the desired class of target analytes sample preservation may or may not be necessary; see determinative method	4 ± 2°C Must be free of organic solvent vapors and direct or intense light	pH Preserved: 14 days
Soils	Refer to Method 5035A for soil collection techniques	Refer to Method 5035A for soil preservation techniques	Refer to Method 5035A for soil storage techniques	Refer to Method 5035A for soil holding time techniques

8. DEFINITIONS

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual.

9. EQUIPMENT AND SUPPLIES

9.1. **Table 9.1 – Instrumentation**

For instrumentation refer to the determinative method.

9.2. **Table 9.2 - Chromatography Supplies**

For Chromatography Supplies refer to the determinative method.

Table 9.3 – Glassware

Glassware	Description	Vendor/ Item # / Description
Glass Vials	40mL VOA vials unpreserved with	C and G/ LV54-A000-A01A-P01

	Teflon-lined screw caps	/ Case of 80 *
Glass Vials	40mL VOA vial Preserved 0.5mL trace grade HCl with Teflon-lined screw caps	Sci/Spec / 376840-.5THCL/Case of 72*
Jars	1oz – Stand clear glass short wide mouth w/ PTFE lined caps 2oz- Stand clear glass short wide mouth w/ PTFE lined caps 4oz- Stand clear glass short wide mouth w/ PTFE lined caps 8oz- Stand clear glass short wide mouth w/ PTFE lined caps 16oz - Stand clear glass short wide mouth w/ PTFE lined caps	1oz – Sci/Spec / B70801 2oz- Sci/Spec / B70802 4oz- Sci/Spec / B70804 8oz- Sci/Spec / B70808 16oz- Sci/Spec / B70816*
Glass Pipets	9” glass pipets	Fisher/ 22-230490/ Case of 1000*
Volumetric Flasks	5mL, 50mL, 100mL	Class A*

*Or equivalent

9.3. **Table 9.4 - General Supplies**

Supply	Description	Vendor/ Item # / Description
Gas tight syringes	10µL, 25µL, 50µL, 100µL, 250µL; 1mL, 2.5mL, 5mL; with Luerlok tip	Hamilton
Syringe valve	With male and female Luerlok connections	
pH paper	pH range 0-14	Fisher/ M1095350007/ 100 strips*
Balance	Capable of weighting 100g ± 0.01g	Ohaus CS200*
Spatula	Narrow, stainless steel	
Ottowa Sand	Purified solid matrix	EMD/ SX0070-1*
Magnetic stir bars	Stirring bars to fit 40mL vials	
Oven		
Helium		

*Or equivalent

For any other supplies refer to the determinative method.

10. REAGENTS AND STANDARDS

10.1. **Table 10.1 – Reagents and Standards**

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Organic-free Water (OFW)	De-ionized water	Verify that background levels of volatile compounds are acceptable by analysis
Methanol	Purge and trap grade	Honeywell/ 232-1L*
Hydrochloric Acid	Solution of 1:1 of concentrated HCl and De-ionized water	
Sodium bisulfate	ACS reagent grade, granular	

* Or Equivalent

For other regents and standards refer to the determinative method.

11. CALIBRATION AND STANDARDIZATION

11.1. For requirement and procedure refer to the determinative method.

12. PROCEDURE

12.1. **Sample Preparation- Aqueous:**

12.1.1. All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Suspended particulates in volatile organic samples should be allowed to settle and are not subsampled.

12.1.2. Assemble the purge-and-trap device. The operating conditions for the GC and GC/MS are given in the specific determinative methods.

12.1.3. When using the Archon autosampler, the 40mL VOA vial can be placed directly into the autosampler tray. The autosampler will then follow its program as to what order to sample the vials and what volume is to be removed. Dilutions may be done automatically by the autosampler, or diluting the sample into another VOA vial and placed into the tray. The Archon will add a programmed internal standard to the sample as the aliquot is being removed. The autosampler removes an aliquot of the sample, adds the internal standard and surrogates and delivers the aliquot to the concentrator to begin the purge cycle. Upon completion of the purge cycle, the autosampler will rinse the entire sample pathway with water to remove any potential carry-over contamination.

12.2. **Sample Preparation- High Concentration Samples:**

12.2.1. Refer to Method 5035A for preparation of high concentration samples.

12.2.2. See section 12.1 for procedure preparation, after sample is prepared by Method 5035A.

12.3. **Sample Analysis**-

12.3.1. For requirement and procedure refer to the determinative method.

13. QUALITY CONTROL

13.1.1. For requirement and procedure refer to the determinative method

14. DATA ANALYSIS AND CALCULATIONS

14.1.1. For requirement and procedure refer to the determinative method

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

15.1.1. For requirement and procedure refer to the determinative method

16. Corrective Actions for Out-of-Control Data

16.1.1. For requirement and procedure refer to the determinative method

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1.1. For requirement and procedure refer to the determinative method

18. Method Performance

18.1.1. For requirement and procedure refer to the determinative method

19. Instrument/Equipment Maintenance/Troubleshooting

19.1.1. For requirement and procedure refer to the determinative method

20. Safety

20.1. All in-house safety regulations have to be observed during sample preparation and analysis.

20.2. **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.

20.2.1. Read information and follow warnings listed on the labels of the containers of the chemicals.

20.2.2. In addition, the reference file of material safety data sheets (MSDS) should be consulted for properties of the chemicals used, to determine handling precautions.

20.3. Safety glasses, a lab coat, and disposable gloves must be worn when handling chemicals and samples.

20.4. Sample handling should be conducted in fume hoods.

20.5. The company wide Chemical Hygiene and Safety Manual contains additional information on safety.

20.6. **Equipment:** Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones. The purge and trap concentrator and autosampler use gas under pressure to purge samples and, in some cases, drive the robotic assemblies. These high pressures introduce the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

21. Waste Management

21.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste.

21.2. Refer to the lab's *Sample and Waste Management* SOP.

22. POLLUTION PREVENTION

22.1. For requirement and procedure refer to the determinative method

23. REFERENCES

23.1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

23.2. Bellar, T., "Measurement of Volatile Organic Compounds in Soils Using Modified Purge-and-Trap and Capillary Gas Chromatography/Mass Spectrometry ", U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH, November, 1991.

23.3. USEPA OSW, "Development and Evaluation of Methods for the Analysis of MTBE," September 17.2001.

23.4. USEPA OUST, *Environmental Fact Sheet: Analytical Methods for Fuel Oxygenates,* EPA 510-F-03-001, April, 2003 http://www.epa.gov/OUST/mtbe/omethods.pdf.

23.5. White, H., Lesnik, B., and Wilson, J. T., “Analytical Methods for Fuel Oxygenates”, *LUSTLine* (Bulletin #42), October, 2002, http://www.epa.gov/oust/mtbe/LL42Analytical.pdf

23.6. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 5030C - Purge-and-Trap for Aqueous Samples, Revision 3 May 2003

23.7. National Environmental Laboratory Accreditation Conference (NELAC) Constitution, Bylaws and Standard, approved June 5, 2003, (EPA/600/R-04/003)

23.8. 2009 TNI Laboratory Accreditation Standards, EL-V1-2009.

24. TABLES AND FIGURES

For requirement and procedure refer to the determinative method

25. REVISIONS

Document Number	Reason for Change	Date
S-LI-O-013-rev.00	Transition to PACE format. Separated preparation method from determinative methods.	04/17/2015

APPENDIX P3-2
SOP FOR THE SEPARATORY FUNNEL EXTRACTION BY SW-846 METHOD 3510C
(S-LI-O-004-REV.00)

STANDARD OPERATING PROCEDURE
SEPARATORY FUNNEL EXTRACTION
Reference Methods: EPA SW-846 METHOD 3510C AND EPA 625

Local SOP Number:	S-LI-O-004-rev.00
Effective Date:	Date of Final Signature

APPROVALS

Joann M. Slavin	5/9/15
General Manager	Date
Nicole L. Crespin	5/9/15
Quality Manager	Date
James Bidas	5/9/15
Department Manager	Date

PERIODIC REVIEW

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1. Identification of Test Method

1.1. This Standard Operating Procedure (SOP) documents the procedures used by PASI – LI for extracting non-volatile and semi-volatile organic compounds from aqueous samples in a separatory funnel while meeting the requirements specified in EPA SW-846 Method 3510C and EPA 625.

1.2. For work governed by the NYS DEC Analytical Service Protocol (ASP), the requirements for analysis and reporting of the DEC ASP have to be met.

1.3. In addition to the requirements of this SOP, the guidelines in the *Quality Assurance, Quality Control Manual* have to be observed.

2. Summary of Method

2.1. A measured volume of sample, usually about 1 liter, is serially extracted with Dichloromethane in a separatory funnel. 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 or analysis.

3. Scope and Application

3.1. This procedure is for extracting water insoluble or slightly water soluble organic compounds from aqueous samples using Dichloromethane as the extraction solvent.

3.2. This procedure is applicable for the extraction of Semi-Volatile Compounds (BNAs), Pesticides, PCBs, and petroleum hydrocarbons.

3.3. 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 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 extraction of aqueous samples.

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.

6.3. Extracts that exhibit interferences can be run through a cleanup procedure (see EPA method 3600). Before using a cleanup method, the analyst should 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, Shipment and Storage

Table 7.1 – Sample Collection, Preservation, Storage, and Hold time

Sample Type	Collection per Sample	Preservation	Storage	Holding Time
Aqueous	1.0 L Amber glass Teflon-lined cap	None	4 ± 2°C	Extraction – 7 days from collection date. (ASP Protocol: 5 days from VTSR*) Analysis – 40 days from extraction date.
Aqueous PCBs	1.0 L Amber glass Teflon-lined cap	None	4 ± 2°C	Extraction – 1 year from collection date. (ASP Protocol: 5 days from VTSR*) Analysis – 1 year from extraction date.

*VTSR = Verified Time of Sample Receipt

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. ·ALIQUOT: A measured portion of a field sample taken from analysis.

8.2. ·ANALYTE: The element or ion an analysis seeks to determine; the element of interest.

8.3. ·BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For Q. C. purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

8.4. ·CONTROL SAMPLE: A Q. C. sample introduced into a process to monitor the performance of the system.

8.5. ·FIELD DUPLICATES: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

8.6. ·LABORATORY CONTROL SAMPLE: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

8.7. ·MATRIX: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

8.8. ·MATRIX DUPLICATE: An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

8.9. ·MATRIX SPIKE: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

8.10. ·MATRIX SPIKE BLANK: An aliquot of reagent water fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the spiking solution used for the MS/MSDs.

8.11. ·MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

8.12. ·METHOD BLANK: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process. For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern should not be higher than the highest of either:

8.12.1. The method detection limit, or

8.12.2. Five percent of the regulatory limit for that analyte, or

8.12.3. Five percent of the measured concentration in the sample.

8.13. ·QUALITY ASSURANCE PROJECT PLAN (QAPjP): An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives for a specific analyte above the MDL, and may involve the use of matrix spikes.

8.14. ·RCRA: The Resource Conservation and Recovery Act.

8.15. ·REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

8.16. ·REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water.

8.17. ·RECOVERY: A determination of the accuracy of the analytical procedure made by comparing measured values for a spiked sample against the known spike values. Recovery is determined by the following equation:

8.17.1. $\% \text{ recovery} = \frac{\text{measured value}}{\text{known value}} \times 100\%$

8.18. ·SAMPLE: A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

8.19. ·SPLIT SAMPLES: Aliquots of sample taken from the same container and analyzed independently. In cases where aliquots of samples are impossible to obtain, field duplicate samples should be taken for the matrix duplicate analysis. These are usually taken after mixing or compositing and are used to document intra- or interlaboratory precision.

8.20. ·STANDARD ADDITION: The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.

8.21. ·SURROGATE: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

9. Equipment and Supplies

Table 9.1 - Instrumentation

Equipment	Vendor	Model / Version
N-EVAP	Organomation	11155
Water Bath	Boekel/Grant	PB-2800
TurboVap	Biotage	103187/07

Table 9.2 - Glassware

Glassware	Vendor	Catalog #
2 L Separatory Funnels	Fisher	10-437-25E
1L Graduated Cylinder		
250 mL Glass Beakers	Fisher	10-322E
Glass Funnels	Fisher	10-346-5C
250 mL Kuderna Danish Concentrators	Fisher	570035-0250
500 mL Kuderna Danish Concentrators	Fisher	570035-0500
250 mL Kuderna Danish Flasks	Fisher	570011-0250
500 mL Kuderna Danish Flasks	Fisher	570037-0500
Snyder Columns	Fisher	503000-0121
Concentrator Tubes	Fisher	570051-1025
250 mL Turbovap Tubes	Atlas	www.atlasglassworks.com

Table 9.3 - General Supplies

Supply	Vendor	Catalog #
2 mL crimp-top Amber Vials	Restek	24386
2 mL crimp-top Clear Vials	Restek	24384
Crimp Caps	Restek	24370
Clips for KD flasks	Fisher	05-884D
Boiling chips	Fisher	09-191-20
5 ¾ inch Pasteur Pipets	Fisher	22-230-482
9 inch Pasteur Pipets	Fisher	22-230-490
pH Test Strips	Fisher	M1095350007
11 mL Clear Vials	Fisher	B69308
40 mL Amber Vials	SciSpec	B75741

Supply	Vendor	Catalog #
10 µL Syringe	Hamilton	80000
25 µL Syringe	Hamilton	80200
50 µL Syringe	Hamilton	80920/80900
100 µL Syringe	Hamilton	81020/81000
250 µL Syringe	Hamilton	81120
500 µL Syringe	Hamilton	81220
1000 µL Syringe	Hamilton	81320

10. Reagents and Standards

Table 10.1 – Reagents

Reagent	Vendor	Catalog #
Reagent Water, Organic-free	N/A	N/A
Sodium Sulfate	Fisher	S-415-200LB
Dichloromethane (Methylene chloride)	Fisher	D151-4
Acetone	Fisher	A949-4
Hexane	Fisher	H306-4
Sulfuric acid (conc)*	Fisher	A300-212
Sodium Hydroxide**	Fisher	S-318-3

*1:1 Sulfuric Acid – add 500 mL conc. H_2SO_4 to 500 mL H_2O. Add acid slowly to avoid bumping. Always add acid to the water, NOT the opposite.

**10N Sodium Hydroxide – add 400 g NaOH to 1.0 L of H_2O. Use a Teflon bottle to make the solution and cool bottle in a bucket of cold water as the dissolution of NaOH is highly exothermic.

All solvents must be pesticide quality or equivalent.

Table 10.2 – 8270/STCLP/SIM Stock Standards

Standard	Description	Concentration	Vendor	Catalog #
QC8270_1	1,3,5-Trinitrobenzene	1000 µg/mL	Restek	568614
QC8270_2	8270 Megamix	1000 µg/mL	Restek	31850
QC8270_3	Appendix IX Mix 2	1000 µg/mL	Restek	568635
QC8270_4	Decane	1000 µg/mL	SPEX	S-1112
QC8270_5	Dinoseb	1000 µg/mL	Restek	32251
QC8270_6	Octadecane	1000 µg/mL	SPEX	S-2850
QC8270_7	Appendix IX Mix 1	2000 µg/mL	Restek	32459
QC8270_8	Benzidine Mix	2000 µg/mL	Restek	31834
QC8270_9	Benzoic Acid	2000 µg/mL	Restek	31879
QC8270_10	Methapyrilene	2000 µg/mL	Restek	32460
QC8270_11	Organophosphorous Pesticides Mix	2000 µg/mL	Restek	32419
QC625_1	Acid Composite Mix	2000 µg/mL	Accustandard	CLP-HC-A-R
QC625_2	Base/Neutral Composite Mix	2000 µg/mL	Accustandard	CLP-HC-BN-R

Standard	Description	Concentration	Vendor	Catalog #
QC625_3	Composite Mix 3	2000 µg/mL	Accustandard	Z-014F
QC625_4	Benzidines Mix	2000 µg/mL	Accustandard	Z-014E-R3
QC4.2_1	Acetophenone	5000 µg/mL	Restek	30621
QC4.2_2	Atrazine	1000 µg/mL	Restek	32208
QC4.2_3	Benzaldehyde	2000 µg/mL	Restek	33017
QC4.2_4	Biphenyl	2000 µg/mL	Supelco	4-8161
QC4.2_5	Caprolactam	2000 µg/mL	Restek	31833
SS952_1	BNA Surrogates, Acid/Base Indicator	100-150 µg/mL	Ultra	ISM-336XC-500
SS952SIM_1	SOM 1.0 SIM	2000 µg/mL	Restek	33913
QCSTCLP_1	TCLP Semivolatile Mix	2000 µg/mL	Ultra	TCLP-512

Table 10.3 – DRO Stock Standards

Standard	Description	Concentration	Vendor	Catalog #
QCDRO_1	Diesel Fuel 2	50,000 µg/mL	Accustandard	DRO-AK-102-LCS-10X-R1
SSDRO_1	1,4-Dichlorobenzene-d4	2000 µg/mL	Accustandard	Z-014J-3-M-0.5X

Table 10.4 – 8081/8082/PTCLP Stock Standards

Standard	Description	Concentration	Vendor	Catalog #
QC8081_1	alpha-Chlordane	1000 µg/mL	Accustandard	P-134S-A-10X
QC8081_2	gamma-Chlordane	1000 µg/mL	Accustandard	P-135S-A-10X
QC8081_3	Isodrin	100 µg/mL	Accustandard	P-471S
QC8081_4	Semi-Volatile Calibration Mix 6	2000 µg/mL	Restek	31012
QC8081_5	Chlordane	100 µg/mL	Accustandard	P-017S
QC8081_6	Toxaphene	1000 µg/mL	Accustandard	AS-E0111
QC8081_7	Mirex	100 µg/mL	Accustandard	P-066S
QC8082_1	Aroclor 1016	1000 µg/mL	Ultra	EPA-1282
QC8082_2	Aroclor 1260	1000 µg/mL	Ultra	EPA-1362
SS953_1	Pesticides Surrogate Mix	200 µg/mL	Supelco	CRM48460

Table 10.5 – 8141 Stock Standards

Standard	Description	Concentration	Vendor	Catalog #
QC8141_1	Organophosphorous Pesticide Mix	2000 µg/mL	Ultra	US-119
QC8141_2	Organophosphorous Pesticide Mix	200 µg/mL	Ultra	SPM-614
QC8141_3	Chloropyrifos	100 µg/mL	Ultra	PST-480M100A01
SS8141_1	Methidathion	100 µg/mL	Ultra	PST-1520A100A01

Table 10.6 - Standard Definitions

Standard	Description
Surrogate Standard	Surrogates are added to each sample and QC sample to monitor extraction efficiency.
QC Standard	This solution contains all targeted analytes and is added to the LFB and MS/MSD.

Table 10.7 - Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	§ Reference solution purchased directly from approved vendor	§ Unopened - Expiration date listed on standard ampule. § Opened - 6 months from date opened or listed expiration date (whichever is sooner)	§ Storage conditions listed on standard ampule.
Intermediate and Working Standard Solutions	§ Solutions prepared by diluting stock solution	§ 6 months from preparation or stock standard expiration date (whichever is sooner) § Replace solution sooner if degradation or evaporation is suspected.	§ Amber vials with Teflon lined screw caps § Method required storage conditions.

Table 10.8 – Working Level Standard Preparation

Solution Name	Concentration	Final Volume	Stocks	Solvent
QC8270	50 µg/mL	20 mL	1000 µL QC8270_1 1000 µL QC8270_2 1000 µL QC8270_3 1000 µL QC8270_4 1000 µL QC8270_5 1000 µL QC8270_6 500 µL QC8270_7 500 µL QC8270_8 500 µL QC8270_9 500 µL QC8270_10 500 µL QC8270_11	Methanol

Solution Name	Concentration	Final Volume	Stocks	Solvent
QC625	100 µg/mL	20 mL	1000 µL QC625_1 1000 µL QC625_2 1000 µL QC625_3	Methanol
BENZIDINES	2000 µg/mL	1 mL	Undiluted QC625_4	Methanol
QC4.2	100 µg/mL	20 mL	400 µL QC4.2_1 2000 µL QC4.2_2 1000 µL QC4.2_3 1000 µL QC4.2_4 1000 µL QC4.2_5	Methanol
SS952	100 µg/mL 150 µg/mL	500 mL	Undiluted SS952_1	Methanol
QC625SIM	10 µg/mL	5 mL	500 µL QC625	Methanol
SS952SIM	10 µg/mL	20 mL	100 µL SS952SIM_1	Methanol
QCSTCLP	100 µg/mL	20 mL	1000 µL QCSTCLP_1	Methanol
QCDRO	50,000 µg/mL	1 mL	Undiluted QCDRO_1	Acetone
SSDRO	2000 µg/mL	1 mL	Undiluted SSDRO_1	Methanol
QC8081	20 µg/mL	10 mL	QC8081_1 QC8081_2 QC8081_3 QC8081_4	Acetone
CHLORDANE	100 µg/mL	N/A	Undiluted QC8081_5	Methanol
TOXAPHENE	1000 µg/mL	N/A	Undiluted QC8081_6	Methanol
MIREX	0.4 µg/mL	25 mL	100 µL QC8081_7	Acetone
QC8082	10 µg/mL	100 mL	1000 µL QC8082_1 1000 µL QC8082_2	Acetone
SS953	0.2 µg/mL	1000 mL	1000 µL SS953_1	Acetone
QC8141	200 µg/mL	10 mL	1000 µL QC8141_1	Acetone
SPM614	20 µg/mL	20 mL	2000 µL QC8141_2	Acetone
CHLOROPYRIPHOS	100 µg/mL	1 mL	Undiluted QC8141_3	Acetone
SS8141	10 µg/mL	20 mL	2000 µl SS8141_1	Acetone

11. Calibration and Standardization

11.1. Not applicable to this SOP.

12. Procedure

12.1. Using a 1 L graduated cylinder, measure two 1.0 L aliquots of nanopure water and transfer to a 2 L Separatory Funnel. These will be used for batch Method Blank and Laboratory Fortified Blank.

12.2. Equilibrate samples at room temperature.

12.3. Determine sample volume.

Procedure A:

12.3.1. For samples received in bottles provided by the lab, which will be uniform, use a calibrated 1 L bottle to compare to sample and use spare sample if additional volume is needed to make up 1L (note that removal of sample will not be necessary, as the 1L mark of lab provided bottle comes to the neck of the bottle). Transfer to a 2 L separatory funnel.

Procedure B:

12.3.2. If samples are **not** received in bottles provided by the lab, use entire volume supplied. For this procedure the actual sample volume needs to be recorded. Mark the meniscus of the sample, and pour the sample into a 2L separatory funnel. Determine the volume at a later time by filling water to the mark.

12.3.3. Alternatively, measure the sample volume in a Teflon graduated cylinder and add the appropriate amount from spare to make up 1000 mL. Use a small volume of methylene chloride to rinse the cylinder and add the rinsate to the sample separatory funnel. Note: Measuring cylinders have to be cleaned between samples.

12.4. If multiphase samples are received, notify the client to discuss how to proceed.

12.5. Select one sample from the batch and measure two additional 1.0 L aliquots for Matrix Spike and Matrix Spike Duplicate. If there is insufficient spare volume, prepare two 0.5 L aliquots.

12.6. Spike each Field Sample and QC sample with the volume of Surrogate Solution indicated in table 12.2.

12.7. Spike the LFB, MS and MSD with the volume of QC Solution indicated in table 12.3.

12.8. Test the pH of each Field Sample. Using a Pasteur Pipet, place a small aliquot of sample onto the pH test strip. Do not dip the pH test strip in the sample.

12.9. Adjust the pH of the sample to the range listed in table 12.1. Use 1:1 Sulfuric Acid to decrease the pH and 10N Sodium Hydroxide to increase the pH.

12.10. Add 60 mL of Methylene chloride to the empty sample bottle and shake for ~30 seconds. Transfer the 60 mL of Methylene chloride to the corresponding Separatory Funnel for a quantitative transfer.

12.11. Ensure that the screw cap of the Separatory Funnel is tightly sealed. Shake sample ~5 seconds and vent stopcock to relieve pressure. Shake sample vigorously for 1-2 minutes, venting periodically.

12.12. Allow the two layers to separate for at least 10 minutes. If an emulsion is present use a clean 9 inch Pasteur Pipet to break up the emulsion. If Emulsion persists centrifugation or filtration through glass wool may be necessary.

12.13. Drain the extract through a drying funnel (prepare a drying funnel by plugging a glass funnel with glass wool, fill with Sodium Sulfate and pre-rinse with Methylene chloride). Drain into either a 250 mL KD Concentrator or a TurboVap concentrator tube. (For 8270D/625 a 500 mL KD Concentrator must be used – see section 12.15). Avoid transferring the aqueous layer onto the Sodium Sulfate. Rinse the sodium sulfate with ~20-30 mL of Methylene chloride.

12.14. Perform two additional extractions using 60 mL of Methylene chloride and repeating steps 12.10 to 12.13.

12.15. For 8270D/625 samples, the pH must be adjusted to 11 or greater. Three additional 60 mL extractions are performed, repeat steps 12.10 to 12.13.

12.16. If the volume of sample was marked on the outside of the sample bottle (as in Section 12.3.2), refill the bottle to the mark with water and then measure the volume using a graduated cylinder and record in prep log.

12.17. Extract Concentration with KD Concentrators

12.17.1. Remove the drying funnel and discard the used sodium sulfate.

12.17.2. Add ~3 boiling chips, attach a Snyder Column to the top of the KD Flask.

12.17.3. Pre-wet the column by adding one mL MC to the top.

12.17.4. Place each sample in a hot water bath. The temperature of the water bath should be ~20°C above the boiling point of the solvent, however it must be adjusted as required to complete the concentration in 10-20 minutes.

12.17.5. Concentrate until the extract volume is ~3 mL. Do not allow the samples to go to dryness.

12.17.6. Remove the KD Concentrator from the water bath. Add a squirt of Methylene chloride into the Snyder column to rinse. Remove the clips and rinse the outside of the flask with Acetone to remove water. Allow the sample to sit for ~10 minutes.

12.17.7. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1 - 2 mL of methylene chloride. Detach the concentrator tube, place on the N-EVAP, as described below in Section 12.19. If solvent exchange is required, transfer the extract from the concentrator tube to a 11 mL vial, making sure to rinse the tube with Methylene chloride for a quantitative transfer, and then proceed to Section 12.19 for N-EVAP concentration.

12.18. Extract Concentration on TurboVap

12.18.1. The TurboVap provides automated solvent concentration with nitrogen and a sensor controlled endpoint.

12.18.2. Remove the drying funnel from the extracts collected in the TurboVap concentrator tubes and discard the used sodium sulfate.

12.18.3. Set temperature to 50°C and N2 pressure to 7 psi. Select end volume of 1 mL. Load tubes with sample extracts and close cover.

12.18.4. When extracts reach ~ 10 mL, it is necessary to rinse the glassware walls throughout the remainder of the concentration process. The number of rinse steps will be dependent on the sample matrix.

12.18.5. When “ready” light flashes, the end point is reached.

12.18.6. For Tests that need solvent exchange (8081, TCLP PEST, 8082, 8141) see Section 12.19.7.

12.18.7. For Tests that do not need solvent exchange (8270D, 8270D-SIM, 625, 8015D DRO, TCLP BNA) see Section 12.19.6

Note: Carefully rinse the walls and particularly the tip of the concentrator tubes for quantitative transfer of the samples. The rinsing process is very critical to obtain adequate recovery. Great care has to be taken to rinse the walls and especially the lower cone very thoroughly.

12.19. N-EVAP Concentration

12.19.1. Adjust the temperature of the N-EVAP to 30°C -35°C. Install fresh Pasteur pipets as nozzles for each new set of samples to avoid cross contamination.

12.19.2. Load tubes or vials with sample extracts on the manifold and lower nozzles until they almost touch the surface of the extracts.

12.19.3. Turn on the nitrogen supply at 10-20 psi and adjust the individual valves to evenly supply each position with a GENTLE stream of clean dry nitrogen (Observe distortion of solvent surface to gauge intensity.)

12.19.4. Submerge the tubes in the warm bath. This will prevent water condensation.

12.19.5. DO NOT allow sample to evaporate too low or lightweight compounds may be lost.

12.19.6. For Tests that do not need solvent exchange (8270D, 8270D-SIM, 625, 8015D DRO, TCLP BNA):

- When the volume reaches ~0.5 -1 mL, transfer to a 2 mL amber vial and adjust volume according to table 12.1 using final solvent rinse of concentrator tube.

12.19.7. For Tests that do need solvent exchange (8081, TCLP PEST, 8082, 8141):

- When the volume reaches ~0.5-1 mL, add ~3 mL of hexane and concentrate to slightly below 1 mL.
- Repeat hexane solvent exchange two additional times.
- Adjust volume according to table 12.1 using final solvent rinse of concentrator tube.
- Transfer a 1 mL aliquot to a 2 mL vial.

12.20. Additional cleanup procedures may be necessary:

12.20.1. For samples containing high concentrations of high molecular weight non-targeted analytes, Gel Permeation Chromatography by EPA 3640A may be performed.

12.20.2. For Pesticide and PCB samples Sulfur Cleanup by EPA 3660B is required.

12.20.3. For PCB samples Acid Cleanup by EPA 3665A is required.

Table 12.1 – Method Initial Volume, pH, Final Volume and Final Solvent

Analytical Method	Initial Volume	Sample pH	Final Volume*	Final Solvent
8270D	1.0 L	2 then 11	1.0 mL	Methylene chloride
8270D SIM	1.0 L	2 then 11	1.0 mL	Methylene chloride
625	1.0 L	2 then 11	1.0 mL	Methylene chloride

Analytical Method	Initial Volume	Sample pH	Final Volume*	Final Solvent
TCLP BNA	1.0 L	2 then 11	1.0 mL	Methylene chloride
8015D DRO	1.0 L	As received	1.0 mL	Methylene chloride
8081B	1.0 L	Between 5 and 9	10.0 mL	Hexane
8082A	1.0 L	Between 5 and 9	10.0 mL	Hexane
8141B	1.0 L	As received	10.0 mL	Hexane
TCLP PEST	0.5 L	Between 5 and 9	10.0 mL	Hexane

*If ½ the normal Initial Volume is used (i.e. 0.5 L for 8270D or 625) the Final Volume must also be ½ the value listed in the above table.

Table 12.2 – Surrogate Spiking Chart

Analytical Method	Solution	Spike Volume*
8270D	SS952	500 µL
8270D SIM	SS952SIM	100 µL
625	SS952	500 µL
TCLP BNA	SS952	500 µL
8081B	SS953	1000 µL
8082A	SS953	1000 µL
8141B	SS8141	1000 µL
8015D DRO	SSDRO	75 µL
TCLP PEST	SS953	1000 µL

*If ½ the normal Initial Volume is used (i.e. 0.5 L for 8270D and 625) the Spike Volume must also be ½ the value listed in the above table.

Table 12.3 – QC Spiking Chart

Analytical Method	Solution	Spike Volume*
8270D	QC625	500 µL
8270D	BENZIDINES	25 µL
8270D	QC4.2	500 µL
8270D	QC8270**	1000 µL
8270D SIM	QC625SIM	100 µL
625	QC625	500 µL
625	BENZIDINES	25 µL
8015D DRO	QCDRO	50 µL
TCLP BNA	QCSTCLP	500 µL
8081B	QC8081	20 µL
8081B	CHLORDANE***	20 µL
8081B	TOXAPHENE***	20 µL
8081B	MIREX	1000 µL
8082A	QC8082	500 µL
8141B	QC8141	5 µL
8141B	SPM614***	250 µL
8141B	CHLOROPYRIPHOS***	100 µL

Analytical Method	Solution	Spike Volume*
TCLP PEST	QC8081	20 µL
TCLP PEST	TOXAPHENE***	20 µL

*If ½ the normal Initial Volume is used (i.e. 0.5 L for 8270D and 625) the Spike Volume must also be ½ the value listed in the above table.

**QC8270 contains all currently certified analytes and may be used in place of QC625, BENZIDINES and QC4.2, which contain a smaller TCL list of compounds.

***Prepare an additional LFB for each of these standards.

13. Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
MB	Reagent water	One per batch of up to 20 samples	All targeted analytes less than PQL	Re-extract batch if there are positives greater than PQL
LFB	All targeted analytes	One per batch of up to 20 samples	See Analytical SOPs	Re-extract batch if method specified LFB limits are not met.
MS/MSD	All targeted analytes	One set per batch of up to 20 samples	See Analytical SOPs	Add appropriate qualifiers for all MS/MSD compounds that fail QC requirements. No re-extract necessary.
Surrogates	Method specified Surrogate analytes	All samples and QC samples	See Analytical SOPs	Re-extract sample if method specified surrogate limits are not met.

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. The suitability of the method for the analytes tested was determined when the method was developed. During "method startup" in the laboratory, the capability of the analysts, suitability of equipment, materials and instrumentation was tested.

18.2. Internal method performance is established and monitored with use of the following (where applicable):

18.2.1. Method Detection Limit studies

18.2.2. Demonstration of Capability

18.2.3. Precision and accuracy

18.2.4. Positive and negative controls

18.2.5. Measurement of sample matrix effects

18.2.6. Quality Control Samples (Proficiency Testing)

19. Instrument/Equipment Maintenance/Troubleshooting

19.1. For information and guidance related to instrument/equipment maintenance, troubleshooting and computer hardware and software refer to the following documents:

Document # (latest revision)	Document Title
QAM	*Quality Assurance Quality Control Manual*
ADMIN002	*Computers and Programs*

19.2. Troubleshooting procedures also come from experience and training. Problems that the analyst encounters may be elevated to the department supervisor. Other means of problem resolution may come from review of prior related corrective action reports, instrument maintenance records, internal and external audits and lab notes.

20. Safety

20.1. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by personal protection and engineering measures. MSDS are available for all chemicals used in the lab and are available for review.

20.1.1. Read information and follow warnings listed on the labels of the containers of the chemicals.

20.1.2. In addition, the reference file of material safety data sheets (MSDS) should be consulted for properties of the chemicals used, to determine handling precautions.

20.2. Safety glasses, a lab coat, and disposable gloves must be worn when handling chemicals and samples.

20.3. Sample handling should be conducted in fume hoods.

20.4. The company wide Chemical Hygiene and Safety Manual contains additional information on safety.

21. Waste Management

21.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste.

21.2. Refer to the lab's *Sample and Waste Management* SOP.

22. Pollution Prevention

22.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Use pollution prevention techniques in laboratory operation whenever feasible.

22.2. Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

22.3. Control releases from hood and bench operations and comply with discharge permits and regulations, hazardous waste identification rules, and land disposal restrictions.

22.4. The generated waste has to be disposed in a manner not to cause pollution.

22.5. All wastes deemed toxic are collected in the laboratory and are disposed by professional companies licensed in waste disposal.

22.6. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

23. References

23.1. "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update III, Dec. 1996.

23.2. "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update IV, February 2007

23.3. "New York State Department of Environmental Protection Analytical Services Protocol," Update IVa, January 1998

23.4. "New York State Department of Environmental Protection Analytical Services Protocol," April 2005

23.5. National Environmental Laboratory Accreditation Conference (NELAC) Constitution, Bylaws and Standard, approved June 5, 2003, (EPA/600/R-04/003).

23.6. 2009 TNI Laboratory Accreditation Standards, EL-V1-2009.

24. Tables and Figures

24.1. Not applicable to this SOP.

25. Revisions

Document Number	Reason for Change	Date
S-LI-O-004-rev.00	Taking prep methods out of analytical methods and transitioning to PACE format. Removed 8270 DRO.	4/17/15

APPENDIX P3-3

SOP FOR THE SAMPLE PREPARATION AND ANALYSIS OF CHLORINATED PESTICIDES BY SW-846 METHOD 8081B (8081B_R3)

STANDARD OPERATING PROCEDURE

METHOD 8081B

SAMPLE PREPARATION AND ANALYSIS OF CHLORINATED PESTICIDES

Prepared by: Ursula Middel NRC Date: 1/14/14

Approved by: [signature] Date: 1/14/14
Quality Assurance Manager

Approved by: [signature] Date: 1/14/14
Laboratory Manager

SOP No.	8081B
Revision No.:	3
Revision Date	1/14/14
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1.0 IDENTIFICATION OF THE TEST METHOD AND INTERNAL AND NELAC REQUIREMENTS

1.1 The procedure incorporates the following EPA methods, found in the SW 846 Test Methods for Evaluating Solid Waste, Revision 1, Dec. 1996. **(References 1 and 2).**

DETERMINATION:

METHOD #
8081 B

SAMPLE EXTRACTION:

MATRIX	METHOD #	PROCEDURE
Aqueous	3510C	Liquid-Liquid Extraction (Separatory Funnel)
Aqueous	3520C	Continuous Liquid Liquid Extraction
Solids	3545A	Pressurized Fluid Extraction
Non-aqueous Solvent	3580A	Solvent Dilution

CLEANUP:

INTERFERENCE	METHOD #	PROCEDURE
Polar Analytes	3620B	Florisil Cleanup
High Molecular Weight Analytes, Sulfur	3640A	Gel Permeation Cleanup
Sulfur	3660B	TBA Sulfite Cleanup

1.2 For work governed by the NYS DEC Analytical Service Protocol (ASP), the requirements for analysis and reporting of the DEC ASP **(References 3, 4, 5)** have to be met.

1.2.1 The analytical method of the DEC ASP is followed in this SOP.

1.2.2 For reporting of data packages with full documentation according to ASP requirements, all raw data have to be included, and summary tables of calibrations and Q. C. data have to be submitted on forms as specified in the DEC ASP.

1.3 The work can only be performed by analysts trained in the procedure who are qualified according to the standards set by NELAC (National Environmental Laboratory Accreditation Conference).

1.3.1 In addition to the requirements of this SOP, the guidelines in the "*Quality Assurance, Quality Control Manual*" have to be observed.

1.4 For (additional) information and guidance related to instrument/equipment maintenance, troubleshooting and computer hardware and software refer to the following documents:

Document # (latest revision)	Document Title
QAM	*Quality Assurance Quality Control Manual*
ADMIN002	*Computers and Programs*
GC001	*HP 6890 Series Gas Chromatograph Operating Manual Volume 2.Inlets*
GC002	*HP 6890 Series Gas Chromatograph Operating Manual Volume 3. Detectors*
GC003	*HP 6890 Series Gas Chromatograph Operating Nanual Volume 1. General Information*
GC021A	*Perkin Elmer Instruments Total Chrom Workstation Chromatography Software Application Manager's Guide (Book 1of 5)*
GC021B	*Perkin Elmer Pe Nelson Division Turbochrom Workstation Application Manager's Guide (Book 2 Of 5)*
GC021C	*Perkin Elmer Instruments Total Chrom Chromatography Software Tutorial (Book 3 Of 5)*
GC021D	*Perkin Elmer Instruments Total Chrom Chromatography Software Users's Guide Volume Ii (Book 4 Of 5)*
GC021E	*Perkin Elmer Instruments Total Chrom Chromatography Software Users's Guide Volume I (Book 5 Of 5)*

Troubleshooting procedures also come from experience and training. Problems that the analyst encounters may be elevated to the department supervisor. Other means of problem resolution may come from review of prior related corrective action reports, instrument maintenance records, internal and external audits and lab notes.

2.0 APPLICABLE MATRICES

2.1 The method is intended for analysis of pesticides in water, soil and sediment samples, oil, sludges, and solid waste materials.

2.2 Multi-phase samples must undergo phase separation. Which phases are to be analyzed, depends on the analysis objective, and the client has to be consulted.

2.3 Extraction in solid matrices by Accelerated Solvent Extraction (ASE). Method 3545A, is most effective for dry samples with small particle sizes. Soil/sediment samples may be air-dried and ground or mixed with anhydrous sodium sulfate or pelletized diatomaceous earth to absorb moisture before utilizing ASE extraction procedure.

3.0 REPORTING LIMITS AND DETECTION LIMITS

3.1 The quantification limits for water and soil are presented in **Table 1**, "Practical Quantification Limits."

3.1.1 Concentration levels can be measured from low to medium, i.e. from below practical quantification limits (PQL) to one million times these limits. PQL limits are based on sample sizes of 15 g and 1 L. For high levels, extracts are diluted if the calibration range is exceeded.

3.1.2 Smaller sample sizes can only be extracted, if the method is to be used for other media which dictate smaller aliquots or if high concentrations of targeted analytes are known to be present. In these cases smaller sample aliquots need to be approved by the client since they affect the reporting limits.

3.2 Detection limits that can be achieved by this method are presented in **Tables 7 and 8**. In presence of sample interferences, the practical detection limits (PDL) that can be reported for samples is generally about ½ PQL.

3.3 The results between practical quantification limits (PQLs) and the practical detection limit of ½ PQL are reported with the qualifier "J" as estimated values, if "CLP like" reporting is required. .

3.4 If the Accelerated Solvent Extractor is used, reporting limits equivalent to those required for 30 g sample weights are achieved by reducing the final extract volume. (The GPC eluate, which represents an aliquot of half of the extract, is concentrated to 2.5 mL instead of 5 mL.)

3.5 A Limit of Detection and a Limit of Quantification are established and verified quarterly.

3.5.1 Refer to the QAM for LOD/LOQ procedures and definitions.

3.5.2 Current limits are recorded and stored on the server in O/QC/LOD_LOQ.

3.5.3 Reporting to these limits (as apposed to standard PQL, which may differ from LOQ) must be determined at the start of a project and reporting conventions must be established with the client. Project specific requirements must be communicated to analysts prior to sample analysis.

4.0 SCOPE AND APPLICATION

4.1 The method is intended for the analysis of the target compound list (TCL) presented in **Table 1**.

4.1.1 Of the two isomers of heptachlor epoxide, only the exo isomer (isomer B) of environmental significance is tested for. (The two isomers are separated on the chromatographic system.) Calibration and reports for "heptachlor epoxide" imply only the exo isomer.

4.2 Other analytes that are listed in the EPA method can be added to the scan. Accordingly, the compounds have to then be included in the calibrations and Q. C. check solutions. Acceptable method performance for those analytes has to first be documented by multiple analyses as required for initial documentation of capability (IDC).

4.2.1 Kepone generates very wide peaks that cannot be identified by retention time criteria and should therefore be analyzed by GC/MS.

5.0 SUMMARY OF THE TEST METHOD

5.1 Sample Preparation

5.1.1 Water samples can be extracted by separatory funnel or continuous extraction.

5.1.2 15 g of soil samples are processed by Pressurized Fluid Extraction according to EPA method 3545A.

5.1.3 The extracts are dried and concentrated.

5.1.4 GPC cleanup must be performed on all soil sample extracts to avoid instrument contamination. Florisil cleanup is performed as needed. Additionally, residual sulfur is routinely removed by TBA sulfite.

5.2 Sample Analysis

5.2.1 The sample extracts are analyzed by GC/ECD. Samples are injected simultaneously onto two wide bore capillary columns with a splitter and analyzed on two ECD detectors.

5.2.2 The samples are analyzed in “analytical sequences”. Each analytical sequence comprises multi-level calibrations, performance checks and calibration verifications.

5.2.3 The analyses are evaluated and the data reported, taking into account the results from both analytical columns.

5.2.4 If full documentation is required, data packages are generated according to the NYS DEC Analytical Service Protocol.

6.0 DEFINITIONS

- ACCURACY: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will

be a combination of a random component and of a common systematic error (or bias) component.

- ALIQUOT: A measured portion of a field sample taken from analysis.
- ANALYTE: The element or ion an analysis seeks to determine; the element of interest.
- BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For Q. C. purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.
- BIAS: The deviation due to matrix effects of the measured value (x - x) from a known spiked amount. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).
- BLANK: See Equipment Rinsate, Method Blank, Trip Blank
- CALIBRATION: The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of concentration of reagents as used in the sample preparation.
- CONTROL LIMITS: Arrange within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that non-compliant data be flagged.
- CONTROL SAMPLE: A Q. C. sample introduced into a process to monitor the performance of the system.
- CORRELATION COEFFICIENT: A number (r) which indicates the degree of dependence between two variables (concentration – absorbance). The more dependent they are the closer the value to one. Determined on the basis of the least squares line.
- DATA QUALITY OBJECTIVES (DQOs): A statement of the overall level of uncertainty that a decision-maker is willing to accept in results.
- DATA VALIDATION: The process of evaluating the available data against the requirements of the protocol and project plan in regard to quality measurements such as precision, bias, and detection limit. Data validation may be very rigorous, or cursory, depending on project DQOs. The available data reviewed will include analytical results, field Q. C. data and lab Q. C. data, and may also include field records.
- DRY WEIGHT: The weight of a sample based on percent solids. The weight after drying in an oven.
- DUPLICATE: See Matrix Duplicate, Field Duplicate, Matrix Spike Duplicate.

- ESTIMATED DETECTION LIMIT (EDL): The estimated level at which an analyte is detectable based on standard injections: The level should be at least 5 times the signal/noise ratio and less or equal to the quantification limit.
- EQUIPMENT BLANK: See Equipment Rinsate.
- EQUIPMENT RINSATE: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.
- ESTIMATED QUANTITATION LIMIT (EQL): The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected as the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs in SW-846 are provided for guidance and may not always be achievable.
- FIELD DUPLICATES: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.
- HOLDING BLANK: An aliquot of reagent water that is stored with environmental samples to demonstrate that the samples have not been contaminated during storage.
- INITIAL CALIBRATION VERIFICATION (ICV): Verification of the average response factor of the initial calibration with a solution of a different source. Acceptance criteria of the calibration verification are used.
- INSTRUMENT DETECTION LIMIT (IDL): For inorganics it is determined by multiplying by the Students t-Test value the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x the estimated IDL on three days with a minimum of seven measurements per day.
- INTERNAL STANDARDS: Compounds added to every standard, blank, matrix, spike, matrix spike duplicate, matrix spike blank, sample for VOAs), and sample extract (for semivolatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.
- LABORATORY CONTROL SAMPLE: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.
- LIMIT OF DETECTION (LOD): An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte-and

matrix-specific and may be laboratory-dependent. There is a major difference between NELAC and DoD definitions of LOD. According to NELAC, the LOD equates with the MDL. Under the DoD QSM standard, the LOD is greater than the MDL and the concentration of the LOD verification sample establishes the LOD.

- LIMIT OF QUANTITATION (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The lowest concentration that produces a quantitative result within specified limits of precision and bias. The LOQ shall be set at or above the concentration of the lowest initial calibration standard.
- MATRIX: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.
- MATRIX DUPLICATE: An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.
- MATRIX SPIKE: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.
- MATRIX SPIKE BLANK: An aliquot of reagent water fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the spiking solution used for the MS/MSDs.
- MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.
- METHOD BLANK: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process. For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern should not be higher than the highest of either:
  - (1) The method detection limit, or
  - (2) Quantification limit (PQL) or
  - (3) Five percent of the regulatory limit for that analyte, or
  - (4) Five percent of the measured concentration in the sample.
- METHOD DETECTION LIMIT (MDL): The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the MDL levels.

- METHOD DETECTION LIMIT STUDY: The analysis and statistical evaluation of seven duplicates of blanks spiked with the level of the analytes of interest at estimated detection limits, for the purpose of determining the MDL levels.
- ORGANIC-FREE REAGENT WATER: For volatiles, all references to water in the methods refer to water in which an interferant is not observed at the method detection limit of the compounds of interest.
  - Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water.
  - Organic-free reagent water may also be prepared by boiling water for 15 minutes and, subsequently, while maintaining the temperature at 90EC, bubbling a contaminant-free inert gas through the water for 1 hour. For semivolatiles and nonvolatiles, all references to water in the methods refer to water in which an interferant is not observed at the method detection limit of the compounds of interest.
  - Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water.
- PERCENT DIFFERENCE (%D): As used in this analytical method and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison (i.e., the %Difference may be either negative, positive or zero).
- PERCENT MOISTURE: An approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105°C, including water. Percent moisture may be determined from the decanted samples and from samples that are not decanted.
- PERCENT SOLIDS: The proportion of solid in a soil sample determined by drying an aliquot of the sample.
- PERFORMANCE EVALUATION (PE) SAMPLE: A sample of known composition provided by NYSDEC for contractor analysis. Used by NYSDEC to evaluate laboratory performance.
- PREPARATION BLANK: An analytical control that contains distilled deionized water and reagents, which is carried through the entire analytical procedure (digested and analyzed). An aqueous method blank is treated with the same reagents as a sample with a water matrix. A solid method blank is treated with the same reagents as a soil sample.

- PRECISION: The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses.
- PROJECT: Single or multiple data collection activities that are related through the same planning sequence.
- PRIMARY ANALYSIS: One of two types of pesticide/PCB analysis by GC/EC techniques, the other being the Confirmation of Analysis. If the two analyses are run at separate times, the Primary Analysis is the first analysis chronologically, and is used to establish the tentative identification of any pesticides/PCBs detected. The identification is then confirmed in the confirmation analysis. If the two analyses are run simultaneously, either may be considered the Primary Analysis.
- QUALITY ASSURANCE PROJECT PLAN (QAPjP): An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives for a specific analyte above the MDL, and may involve the use of matrix spikes.
- QUANTIFICATION LIMIT (PQL) The quantification limit (or "reporting limit") is defined as the lowest calibration level.
- RCRA: The Resource Conservation and Recovery Act.
- REAGENT BLANK: See Method Blank.
- REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
- REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.
- RECOVERY: A determination of the accuracy of the analytical procedure made by comparing measured values for a fortified (spiked) sample against the known spike values. Recovery is determined by the following equation:

$$\% \text{ Rec} = \frac{\text{measured value}}{\textit{known} \text{ value}} \text{ x } 100\%$$

- REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.
- RELATIVE PERCENT DIFFERENCE (RPD): As used in this Protocol and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference above.)

- RESOLUTION: Also termed separation, the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

$$\% \text{ Resolution} = \frac{A}{B} \times 100$$

- RESPONSE FACTOR (RF): The response factor or "calibration factor" for an analyte as peak area per amount (ng) analyzed.

$$RF = \frac{A_{std}}{Conc_{std} \times V_i}$$

Where:
RF = Calibration factor as area per ng
A_{std} = Peak area of standard
$Conc_{std}$ = Concentration of standard in ng per uL
V_i = Volume injected in uL

- RELATIVE RESPONSE FACTOR (RRF): The response factor or "calibration factor" for an analyte as peak area per amount (ng) analyzed relative to the response factor of the internal standard which is set to 1. Since the injection volumes cancel out, the concentrations are entered in the equation.

Relative Response Factor (RRF)

$$RRF = \frac{A_x \times C_{is}}{A_{is} \times C_x}$$

Where:
A_x = Area of characteristic ion for compound measured
C_{is} = Concentration of internal standard (ng/uL)
A_{is} = Area of the characteristic ion for the specific internal standard
C_x = Concentration of compound to be measured (ng/uL)

- ROUNDING RULES:
  - If the figure following those to be retained is less than 5, the figure is dropped and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.
  - If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.
  - If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44 while 11.425 is rounded off to 11.42.

- If a series of multiple operations is to be performed (add, subtract, divide, multiply) all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

- SAMPLE: A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.
- SAMPLE DELIVERY GROUP (SDG): A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer samples within a Case, received over a period of up to 7 calendar days. Data from all samples in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, whichever occurs first:
  (1) SDG; or
  (2) Each 20 samples within a SDG; or
  (3) Each 7-day calendar period during which samples in a SDG are received, beginning with receipt of the first sample in the SDG.

  Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the Laboratory.
- SPLIT SAMPLES: Aliquots of sample taken from the same container and analyzed independently. In cases where aliquots of samples are impossible to obtain, field duplicate samples should be taken for the matrix duplicate analysis. These are usually taken after mixing or compositing and are used to document intra- or interlaboratory precision.
- STANDARD ADDITION: The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.
- STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.
- SURROGATE: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.
- TENTATIVELY IDENTIFIED COMPOUNDS (TIC): Compounds detected in samples that are not target compounds, internal standards or surrogate standards. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass special library searches for tentative identification.

- TRIP BLANK: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.
- VALIDATED TIME OF SAMPLE RECEIPT (VTSR): The date on which a sample is received at the Laboratory's facility, as recorded on the shipper's delivery receipt and Chain of Custody.
- WET WEIGHT: The weight of a sample aliquot including moisture (undried).

7.0 INTERFERENCES

7.1 Analysis is performed on ECD detectors, which are semi-selective. Not only other chlorinated organic compounds interfere with the analysis, but other analytes that "capture" electrons also show responses.

7.1.1 Phthalates contain carbonyl groups which respond on the ECD. Phthalates represent the most commonly found interferences.

7.1.2 Contact of samples or reagents with any plastic materials has to be avoided to prevent phthalate contaminations.

7.2 In presence of interferences, analysis on two different analytical columns aids in distinguishing the targeted analytes from interferences.

7.3 To reduce the number of potentially interfering organics, the extract is subjected to cleanup procedures designed to isolate the targeted analytes as much as possible.

7.4 Apart from interferences stemming from the sample itself, interferences can also be introduced into the extract. These secondary contaminations have to be minimized.

7.5 All glassware has to be scrupulously cleaned as described in the SOP for glassware preparation.

7.6 The reagents utilized during the sample preparation have to be pure or have to be treated to remove interferences.

7.6.1 High purity solvents are purchased. Blanks for solvents are not generally analyzed, but the method blanks would alert the analyst, if a bad batch of solvent had been obtained.

7.6.2 Sodium sulfate, used for drying of extracts, has to be purified by baking in the kiln for at least four hours at 400°C - 450°C and (or) extraction solutions with ether.

7.7 To demonstrate that no secondary contaminations are introduced by the glassware or reagents, method blanks are run with each batch.

7.7.1 If new reagents are used within a batch, a new method blank has to be included.

7.8 The level of interferences encountered in the extract will determine the practical detection limit (PDL). Empirically a level of 1/2 the quantification limit (PQL) has been found to be a practical limit for reporting for most types of samples based on historical data. Values above the PDL and under the quantification limit can be reported as estimated values.

7.9 For highly contaminated samples the detection limit may be above the PQL where sample have to be diluted and the reporting limit will be raised.

8.0 SAFETY

8.1 All in-house safety regulations have to be observed during sample preparation and analysis.

8.1.1 Consult material handed out during orientation for handling of corrosives, flammable and toxic materials.

8.1.2 Use common sense and specially regard safety of coworkers.

8.2 Observe safety rules for working with chemicals in posted Code of Federal Regulations Section 29, Part 1910.1450.

8.3 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest level possible.

8.3.1 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, the BHCs, and the PCBs. Primary standards of these toxic compounds should be prepared in a hood.

8.3.2 Read information and follow warnings listed on the labels of the containers of the chemicals.

8.3.3 In addition, the reference file of material safety data sheets (MSDS) should be consulted for properties of the chemicals used, to determine handling precautions.

8.3.4 Some pesticides have been tentatively classified as known or suspected, human or mammalian carcinogen. High level standard have to be treated accordingly as toxic materials.

8.4 Particular safety measures apply to the use of the Accelerated Solvent Extractor. The use of organic solvents, elevated temperatures, and high pressures in Method 3545A present potential safety concerns.

8.4.1 Extraction cells in the oven are hot enough to burn unprotected skin. Allow the cells to cool before removing them from the oven or use appropriate protective equipment (e.g., insulated gloves or tongs), as recommended by the manufacturer.

8.4.2 During the gas purge step, some solvent vapors may exit through a vent port in the instrument. Follow the manufacturer's directions regarding connecting this port to a fume hood or other means to prevent release of solvent vapors to the laboratory atmosphere.

8.4.3 The instrument may contain flammable vapor sensors and should be operated with all covers in place and doors closed to ensure proper operation of the sensors. Follow the manufacturer's directions regarding replacement of extraction cell seals when frequent vapor leaks are detected.

9.0 EQUIPMENT AND SUPPLIES

9.1 Equipment and Supplies for Sample Preparation

- Sample bottle - amber glass, 1 liter with Teflon lined cap
- Liquid-liquid extractor - ROT-X-TRACT-L from Organomation or equivalent with Hershberg - Wolf type continuous extractors or “one-step” extractors
- Separatory funnel - 2000 mL Teflon with Teflon stopcock
- Drying oven
- Weighing pans - aluminum
- Glass jars - 8 oz. wide mouth
- Kuderna - Danish apparatus, comprising:
  - Receiving vial - 10 mL
  - Evaporative flask - 500 mL
  - Snyder column - three ball, macro
  - Snyder column - three ball, micro (optional)

- Powder funnel - 10 cm
- Boiling chips - Teflon, solvent rinsed
- Water bath
- Balance - Sartorius, top loading
- Nitrogen evaporator - 12 position N-EVAP from Organomation or equivalent
- Vials - calibrated for 5 mL
- Vials - calibrated for 10 mL
- Auto-sampler vials - 2 mL, amber or clear, with Teflon lined caps
- Automated GPC System - AccuPrep with AccuChrome chromatography software from J2 Scientific, or equivalent
- GPC column - 700 mm x 25 mm with S-X3 beads
- Syringe - 10 mL with Luer lock fitting
- Syringe filter - disposable, 25 mm, 5 micron (or less) from Gelman or equivalent
- Florisil cartridges, 1000 mg with SS or Teflon frits from Supelco or equivalent
- Vacuum manifold - with Teflon Luer fittings and valves by Supelco or equivalent
- Test tubes - 16 x 100 mm
- Glass wool - Pyrex, solvent rinsed
- Drying column or powder funnel
- Spatula - stainless steel
- Pasteur pipets - disposable, 5.5 inch
- pH Paper - wide range
- Syringe - 1 mL
- Centrifuge - clinical
- Vortex mixer
- pH Meter - with glass electrode
- Stirrer - magnetic
- Graduated cylinder - 1000 mL
- Kiln operated at 400°C, - Firemate FE27 from Cress or equivalent
- Nitrogen instrument grade, with dual stage regulator to provide 10 - 20 psi
- Bottle - 500 mL with Teflon lined cap for extract storage

- Automated N2 evaporator - Turbo Vap II from Zymark
- Concentrator vials - 200 mL for Turbo Vap II
- Kuderna - Danish apparatus:
  Receiving vial - 10 mL
  Evaporating flask - 500 mL
  Snyder column - three ball, macro
  Snyder column - three ball, micro (optional)
  Clips - for 19/22 and 24/40 ground glass joints
- Nitrogen concentrator 12-position nitrogen concentrator, N-EVAP from Organomation, or equivalent

9.2 Supplies and Materials for Accelerated Solvent Extraction

- Pressurized fluid extraction device - Accelerated Solvent Extractor ASE200 or equivalent
- Stainless steel extraction cells- 11, 22, 33 mL, capable of withstanding 2000+ psi
- Drying oven- adjustable at 100 +/- 5°C
- Crucibles - porcelain or disposable aluminum
- Apparatus for grinding - capable of reducing particle size to < 1 mm
- Analytical balance - capable to weighing to 0.01 g.
- Vials for collection of extracts - 40-mL or 60-mL, pre-cleaned, open top screw-cap with PTFE-lined silicone septum (Dionex 049459, 049460, 049461, 049462 or equivalent)
- Filter disk - 1.91 cm, Type D28 (Whatman 10289356, or equivalent)
- Cell cap sealing disk (Dionex 49454, 49455, or equivalent)
- Kiln- capable for heating of diatomaceous earth and sodium sulfate to 400oC, from Cress or equivalent
- Powder funnel- 10 cm, to be used for drying with sodium sulfate
- Glass wool- fiberglass 8 micron, purified by heating at 400oC for 4 hours in kiln, or equivalent
- Drying apparatus (optional)- Dry-Disk Apparatus for removal of water in extract from Horizon, or equivalent
- Dry-Disks for Horizon drying apparatus
- Nitrogen concentrator (optional)- multi-position automated nitrogen concentrator, Turbovap VI from Zymark, or equivalent
- Kuderna - Danish apparatus:

Receiving vial - 10 mL
Evaporating flask - 500 mL
Snyder column - three ball, macro
Snyder column - three ball, micro (optional)
Clips - for 19/22 and 24/40 ground glass joints

- Nitrogen concentrator 12-position nitrogen concentrator, N-EVAP from Organomation, or equivalent
- Automated N_2 evaporator - TurboVap II from Zymark or equivalent (optional for GPC extracts)
- Concentrator vials - 200 mL for Turbo Vap II

9.3 Equipment and Supplies for Instrument Analysis

9.3.1 GC System:

- GC with three stage programmable temperature program, gas controls, inlet system suitable for capillary columns, nitrogen-phosphorous detector, auto-sampler
- 0.32 mm x 30 m fused silica column, RTX-CLP from Restek or equivalent
- 0.32 mm x 30 m fused silica column, RTX-CLP2 from Restek or equivalent
- Alternatively wide-bore columns of 0.53 mm may be used

9.3.2 Data acquisition and processing/reporting system:

- Computer
- Monitor
- Printer
- TotalChrom from Perkin Elmer or equivalent
- Omega from Khemia, or equivalent.

10.0 REAGENTS AND STANDARDS

10.1 Reagents and Standards for Sample Preparation

- Reagent water - generated by High Purity Water System from Aries or equivalent

  For quality specifications of reagent water used in the lab, refer to the *Laboratory Water Supply* section in the Quality Assurance Manual and/or the *Reagent Water* section in the Materials SOP, latest revisions.

- Sodium sulfate - granular anhydrous reagent grade, heated in kiln for at least four hours

- Hydromatrix - Diatomaceous earth-based material rinsed with methylene chloride and dried at 400°C for 4 hours in a shallow tray, cooled in a desiccator, and stored in a glass bottle.
- Methylene chloride - pesticide grade (MC)
- Hexane - pesticide grade
- Acetone - pesticide grade
- 2,4,5-trichlorophenol
- Methanol - pesticide grade
- TBA - sulfite - 3.39 g of tetrabutyl ammonium hydrogen sulfate and 25 g sodium sulfite, dissolved in 100 mL reagent water. (Use equivalent weights for different solution volume.) Extract solution with three portions of appr. 20 mL hexane and store in amber bottle with Teflon lined cap. Date solution and discard after one month.
- Sodium hydroxide solution - 10N, dissolve 40 g of reagent grade NaOH in 100 mL reagent water
- Concentrated sulfuric acid - 18N
- 1:10 Acetone/hexane mixture - 10 mL acetone mixed with 90 mL hexane
- GPC calibration solution

Analyte	mg/mL
corn oil	25
bis-2-ethylhexyl phthalate	1
methoxychlor	0.2
perylene	0.02
sulfur	0.08

Prepare by diluting purchased intermediate solution with methylene chloride.

- Surrogate solution

Analyte	ug/mL
tetrachloro-m-xylene	0.2
decachlorobiphenyl	0.2

Dilute purchased intermediate solution with acetone.

- Surrogate solution for medium level soils

Analyte	ug/mL
tetrachloro-m-xylene	2
decachlorobiphenyl	2

Dilute purchased intermediate solution with acetone.

Prepare dilution from manufacturer's solution in methanol or acetone.

- **Q. C. check spiking solutions for GPC check**

Analyte	ug/mL
AR1016	10
AR1260	10

Prepare dilution from manufacturer's solution in methanol or acetone.

- **Florisil cartridge performance check solution (FCPC)**

  2,4,5-trichlorophenol at 0.1 µg/mL in acetone
  Q. C. check spiking solutions:

- **Q. C. spiking solution containing all single peak targeted pesticides at 20 ng/µL in methanol. As long as ICP check was performed, solutions (or parent solutions) purchased from same vendor as the calibration solutions may be used to eliminate possible discrepancies.**

- **TCLP spiking solution**

Analyte	ng/µL
gamma-BHC	1.0
endrin	1.0
heptachlor	1.0
heptachlor epoxide	1.0
methoxychlor	10
alpha-chlordane	1.0
gamma-chlordane	1.0

- **Solution of toxaphene at 500 ng/µL in methanol**

  Note that solutions of toxaphene from different manufacturers may have different patterns due to dechlorination of the highly chlorinated constituents.

- **Solution of technical chlordane at 40ng/µL in methanol.**

10.2 Reagents and Solutions for Accelerated Solvent Extraction

- **Organic-free reagent water- from High Purity Water System from Aries or equivalent**

  For quality specifications of reagent water used in the lab, refer to the *Laboratory Water Supply* section in the Quality Assurance Manual and/or the *Reagent Water* section in the Materials SOP, latest revisions.

- **Pelletized diatomaceous earth- purified by heating at 400°C for 4 hours in kiln**
- **Nitrogen cylinder - high-purity used to pressurize and purge the extraction cells**
- **Ottawa sand - purified by heating in kiln for 4 hours at 400°C**

- Anhydrous sodium sulfate- purified by heating in kiln for 4 hours at 400°C
- Acetone- pesticides grade
- Methylene chloride - pesticides grade
- Acetone/methylene chloride 1:1v/v-

10.3 Standards for Instrument Analysis

All standards for instrument analysis are prepared in hexane.

- Individual Standard Mixture A (INDA)

Analyte	Concentration (ng/mL)				
alpha-BHC	5	10	20	40	80
Heptachlor	5	10	20	40	80
gamma-BHC	5	10	20	40	80
Isodrin	10	20	40	80	160
Endosulfan I	5	10	20	40	80
Dieldrin	10	20	40	80	160
Endrin	10	20	40	80	160
p,p'-DDD	10	20	40	80	160
p,p'-DDT	10	20	40	80	160
Methoxychlor	50	100	200	400	800
Tetrachloro-m-xylene	5	10	20	40	80
Decachlorobiphenyl	10	20	40	80	160

- Individual Standard Mixture B (INDB)

Analyte	Concentration (ng/mL)				
beta-BHC	5	10	20	40	80
delta-BHC	5	10	20	40	80
Aldrin	5	10	20	40	80
Heptachlor epoxide	5	10	20	40	80
alpha-Chlordane	5	10	20	40	80
gamma-Chlordane	5	10	20	40	80
p,p'-DDE	10	20	40	80	160
Endosulfan sulfate	10	20	40	80	160
Endrin aldehyde	10	20	40	80	160
Endrin ketone	10	20	40	80	160
Endosulfan II	10	20	40	80	160
Tetrachloro-m-xylene	5	10	20	40	80
Decachlorobiphenyl	10	20	40	80	160

Note: For method 608, the individual mixes can be combined, if adequate resolution of all targeted analytes is achieved on both analytical columns. (Good resolution is generally achieved on the new non-polar columns for the targeted analytes.) Solutions have been kept separately in order to be able to use the same calibrations as for ASP.

- Solution of toxaphene at 2 ng/µL and DCB/TCX at 0.040 ng/µL.
- Breakdown Check / Performance Evaluation Mix (PEM)

Analyte	Concentration (ng/mL)
gamma-BHC	10
alpha-BHC	10
4,4'-DDT	100
beta-BHC	10
Endrin	50
Methoxychlor	250
Tetrachloro-m-xylene	20
Decachlorobiphenyl	20

This mix may be used to check the decomposition of 4,4'-DDT and endrin or a mix of only the two analytes may be used instead.

- Initial Calibration Verification Standard Mixes

Solutions for all targeted analytes at medium level from a different source for calibration verification.

- Instrument Blank Solution

Analyte	ng/mL
TCX	20
DCB	20

This solution can be prepared by diluting the surrogate spiking solution with hexane.

10.4 Purchase certified solutions as intermediate or stock solutions and retain the certificates of the manufacturers to present upon request and record solution in the log book and Omega system.

10.5 Solution in sealed ampoules can be held up to two years or shorter/longer as indicated by the manufacturer's expiration date. After opening of the ampoule the six months of expiration times apply.

10.6 Make appropriate dilutions for secondary and working standards with the solvent indicated.

10.7 Each solution preparation has to be entered in the standard log and Omega system.

10.8 Standard solutions have to be properly labeled and stored in amber bottles with Teflon lined caps under refrigeration at 4°C ± 2°C. It is recommended to divide solutions into portions and store in small bottles to minimize headspace, prevent frequently opening of the bottles, and thus preserve integrity of the solution and extend the time when new solutions have to be prepared.

10.9 Intermediate and working calibration standards are prepared by diluting the purchased solutions every two to six months or if changes of concentrations are observed or suspected. The solution for the instrument blank and other solutions that are used frequently, have to be prepared on a weekly basis.

10.10 Before it can be used, each new surrogate, Q. C. and MS spiking solution has to be tested for composition (it should be +/- 20% of true value) in the analytical department and approved by signature in the standard log.

10.11 All solutions have to be equilibrated at room temperature for one hour before they can be used. Solutions that contain analytes that are poorly soluble and may have come out of solution, e. g. DCB, may have to be sonicated or vortexed for about 20 seconds before usage, to make sure that the correct concentration is reached.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 Cleaned glassware is provided by the laboratory. Water samples are collected in amber 1 L bottles and soil samples in amber jars both fitted with Teflon lined caps.

11.2 The samples have to be held at 4 ± 2°C from time of collection until delivery to the laboratory and once received must be stored at 4°C ± 2°C in an environment free of potential contaminants.

11.3 Extraction of water by separatory funnel or continuous extraction has to be completed within seven days of sample collection. Soil samples have to be extracted within 10 days of collection. Analysis must be performed within 40 days after samples were extracted, but due dates for data packages have to be observed.

11.4 Water samples should be re-extracted within 7 days of collection and soil within the mandatory maximum holding time of 14 days from collection.

11.4.1 If re-extraction is necessary, complete a non-conformance memo (NCM). From the NCM the scan is reentered on the extraction work sheets with the new extraction due date.

12.0 QUALITY CONTROL

During “method startup”, the suitability of the method for the targeted analytes is tested by analyzing four spiked blanks at medium level and seven low level spikes to determine the sensitivity of the method. The method performance is then continuously checked by analyzing quality control samples on an ongoing basis. All surrogate recoveries, spike recoveries for targeted compounds, and blanks have to be monitored, and specific Q. C. requirements have to be met. Minimum detection limits (MDL) are determined on a yearly basis.

If the ASP protocol is required, the Q. C. requirements of the CLP protocol apply. Refer to the Q. C. criteria in the SOP of the CLP method.

12.1 Surrogate Recoveries

12.1.1 Tetrachloro-m-xylene and decachlorobiphenyl are spiked into all samples and Q. C. samples to monitor the efficiency of sample preparation for all extracts. (Concentration and spike volumes are given in section “Reagents and Solutions” and in section “Sample Preparation”)

12.1.2 Recovery results are computed according to this equation:

Recovery for Spikes (% Rec)

$$\% \text{ Rec} = \frac{X}{T} \text{ x } 100\%$$

Where:
X = Measured result
T = Targeted value of concentration spiked, in ug/L or ug/kg

12.1.3 Report recovery for both columns on Form II for CLP like reporting.

12.1.4 Determine in- house surrogate limits and update as the LFB limits.

12.1.5 Alternately a limit of 30 to 150% may used to be consistent with CLP reporting. (Usually computed in-house limits were wider.)

12.1.6 If sample recoveries are not within the surrogate recovery limits, the following procedures are necessary:

- Check to be sure that there are no errors in the calculations, surrogate solutions. If errors are found, recalculate the data accordingly.
- Examine chromatograms for interfering peaks and mis-integrated peak areas.
- Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the extract.

12.1.7 If no instrument problem is found, the sample has to be re-extracted.

12.1.8 If surrogate recoveries are outside limit in the re-extract as well, provide both sets of data to document matrix interference.

12.1.9 No re-injections or re-extractions are necessary for the sample used for MS/MSD spiking, if the surrogate recoveries of the MS and MSD extracts fail the requirements. Also, associated samples, i.e. of the same matrix, that show the same recovery pattern, need not be repeated. Discuss the similar recoveries in the narrative.

12.1.10 If the recovery is within the limits in the re-extract, provide the re-analysis data to the data user. If the holding time for the method has expired prior to the re-extraction and/or re-analysis, provide both the original and re-analysis results to the data user, and note the holding time problem.

12.1.11 If a sample required dilution and the surrogates are diluted out, the surrogate recoveries from a lesser dilution or the undiluted run should be reported. The results of both the diluted and undiluted (or less-diluted) analyses should be provided to the data user.

12.2 Accuracy and Precision

To develop precision and accuracy data for each of the spiked compounds, the analyst has two choices: Analyze the original sample and an MS/MSD pair; or analyze the original sample, a duplicate sample, and one spiked sample. Which option is used depends on the sample concentration, as discussed below.

12.2.1 For accuracy determination, include a spiked sample (MS) and a lab fortified blank (LFB) with each extraction batch of up to 20 samples.

12.2.1.1 The LFB and the MS/MSD samples are spiked with the Q. C. solution with all targeted analytes.

12.2.1.2 The concentration of the sample spike should be a multiple of the sample concentration, otherwise Q. C. limits do not apply.

12.2.1.3 No Q. C. limits are developed for the matrix spikes, but the LFB limits are used for MS and MSD samples.

12.2.2 For precision data, one duplicate per batch up to 20 samples must be analyzed on positive samples, or a matrix spike duplicates may be analyzed instead, if no positives are found.

12.2.2.1 If possible select positive samples, if they have already been analyzed.

12.2.2.2 Instead of developing limits, set limits for RPD to 30 %.

12.2.2.3 If RPD limits are exceeded, the data have to be flagged with a qualifier.

12.2.3 Follow the spiking procedures at the levels provided in the sample preparation section.

12.2.4 Compute the LFB, MS and MSD recoveries by the formula below.

Percent Recovery (% Rec)

$$\% \text{ Rec.} = \frac{X - B}{T} \times 100$$

Where:
% Rec. = Recovery as percent
X = Value of measurement as ug/L or ug/kg
T = Targeted concentration as ug/L or ug/kg
B =Background concentration of unspiked sample in ug/L or ug/kg

12.2.5 In-house Q. C. limits for LFBs for method accuracy are developed once enough data points are obtained. Use the recovery data and determine limits in "Control Charting" of the Omega software found under category "Quality Control", or perform statistical evaluations using the equations below.

12.2.6 Calculate the accuracy of the procedure from the last 30 data points (if available):

Relative Average Recovery (Accuracy) ($\%\overline{X}$)

$$\%\overline{X} = \frac{\sum x_i / n}{T} \times 100$$

Where:
$\%\overline{X}$ = Relative average recovery as percent
X_i = Value of individual measurement
n = Total number of value
T = Targeted value (concentration)

12.2.7 Compute relative standard deviation (% RSD) with the following equation:

Relative Standard Deviation (%RSD)

$$\% \text{ RSD} = \sqrt{\frac{\sum (\overline{X}_i - \overline{X})^2 / n - 1}{\overline{X}}} \times 100$$

Where:
% RSD = Relative standard deviation
etc. as above

Determine in-house Q. C. limits according to the equation

Warning Limit (WL) and Control Limit (CL)

$WL = \% \overline{X} \pm 2 \times \% RSD$

$CL = \% \overline{X} \pm 3 \times \% RSD$

Where:
WL = warning limit
CL = Control limit
etc. as above

12.2.8 Typical Q. C. limits for spike recoveries are presented in **Table 6**. Updated limits are kept in the Omega data system.

12.2.9 Limits should be reviewed on an on-going basis. Long standing established limits are generally not updated as long as they are confirmed, to maintain consistent Q. C. To update, compute new Q. C. limits with the Omega control charting, using the last 30 measurements. In-house Q. C. limits must be examined for reasonableness. The Q. C. limits are entered into the Omega system.

12.2.10 Compare LFB results with the Q. C. limits in Omega for compliance. If the recoveries for analytes in the lab fortified blank (LFB) show recoveries outside the Q. C. limits, the responses for these analyte are out of control, and the causes need to be investigated and corrected.

12.2.10.1 Samples with positives for analytes with noncompliant recoveries have to be re-extracted and reanalyzed or have to be flagged. The results are regarded estimated and cannot be used for regulatory purposes.

12.2.10.2 Samples that have no positives may be reported if responses were too high or if sensitivity is still sufficient to report PQLs in spite of the low recovery.

12.3 Method Blank

12.3.1 With each batch of samples extracted together for a particular matrix, a method blank is extracted and analyzed with the samples on both columns.

12.3.2 The blank consists of either reagent water or 30 grams of sodium sulfate or 15 g of Hydromatrix (Diatomaceous earth). The blank extracts undergo the same treatment as the sample extracts, including all cleanup procedures.

12.3.3 If not all samples are subjected to sulfur cleanup, hexane spiked with the surrogate standard is cleaned as a separate “sulfur blank” or an aliquot of the method blank is cleaned up for the sulfur blank.

12.3.4 For a method blank to be acceptable, it cannot contain any targeted analyte above PQL.

12.3.5 Extractions should be halted until the source of the contamination is found and eliminated.

12.3.6 If a blank contains more than the acceptable level of a targeted analyte, re-extract all samples that were extracted in that extraction batch.

12.3.7 No re-extraction is required, if no positives are found in the samples or if the level of positives is not critical for the usability of the data. To establish this, the acceptability of the results needs to be discussed with the data user. (Compare Section 21.)

12.3.8 If **surrogate recoveries for the blank** do not meet the limits, the associated samples from the extraction batch may have to be re-extracted.

12.3.8.1 If recoveries of the blank are too high, samples do not have to be re-extracted.

12.3.8.2 Effect of low recoveries on the associated samples needs to be investigated, and affected samples should to be re-extracted or qualified.

12.3.8.3 Since blanks are extracted to show interferences, sample result would not be affected, if interferences for the analytes could still be detected in the blank at a sufficient level at the recovery found.

12.3.8.4 Recoveries for at least one surrogate compounds must be between 30 percent and 150 percent. (It has been found that selective losses of DCB are caused by adsorption on the auto-extractor on new glassware, and if silanization wears off by usage. Selective loss of TCX may be caused during the concentration step, which would also affect early eluting pesticides.

12.4 Method Detection Limits (MDL)

12.4.1 The MDLs define the lowest levels, where positives will be found with 99 percent confidence with the particular analytical method in clean media. The MDLs should be at or below ½ PQL.

12.4.2 The “method” includes the sample preparation, (including cleanup), and analysis.

12.4.3 Every 12 months, perform MDL studies with seven parallel extractions and analyses of low level lab fortified blanks (LFB), i.e. reagent water or sodium sulfate or 15 g of Hydromatrix (Diatomaceous earth) (instead of soil) respectively.

12.4.4 For the MDL study for mixed pesticides spike each LFB analysis with half of the quantification limit (PQL) of all single pesticides in mixes INDA and INDB. Separately analyze seven extracts of toxaphene at or above the estimated detection limit, i.e. at about half PQL.

12.4.5 Analyze the extracts on both analytical columns on a calibrated instrument that meets all performance check criteria for analytical sequences. Tabulate the results for the seven parallel analyses and statistically evaluate the standard deviations according to the equation below.

12.4.6 From the obtained S calculate the MDL as follows:

Method Detection Limit (MDL)

$MDL = t_{n-1} \times S$

Where:
S = Standard deviation
t_{n-1} = Students t-Test value (for seven replicates $t_{n-1} = 3.14$)

12.4.7 Updated MDLs are kept on file in the laboratory and/or Omega data system. In order to update the MDLs in the Omega, enter the data into an excel spread sheet and transfer them into the method test-code.

12.4.8 Representative detection limits for this method are presented in **Tables 7 and 8**.

12.5 Quality Assurance

12.5.1 Analyze certified reference material, obtained from another source than the calibration standards, to check method performance. State proficiency samples may be used for that purpose.

12.5.2 In-house accuracy limits must be met.

13.0 CALIBRATION, SYSTEM PERFORMANCE CHECKS, AND RETENTION TIME WINDOWS

13.1 Analytical Sequence

13.1.1 If an instrument has not been used for a prolonged period, injection a priming solution is recommended of at least 10 times the high calibration standard to prevent absorption problems. Run a blank after that to avoid carryover.

13.1.2 Calibration standards, performance checks and sample injections are run in an analytical sequence as follows.

13.1.3 The "initial sequence" comprises an instrument blank, multipoint calibrations for the pesticides mixes INDA and INDB and single point calibrations for toxaphene.

13.1.4 Following the initial calibration, inject samples. Intersperse calibration verifications (CCV) after every 20 samples (or less). Use medium level mixes, or alternate with the low- and high- level pesticides mixes.

13.1.5 With each CCV inject an instrument blank.

13.1.6 Breakdown has to be checked with an injection of the endrin/DDT mix at the beginning of every 12 h shift. The PEM solution may be used.

13.1.7 The sequence is terminated with an instrument blank and the standard mixes. If a positive multi-peak compound is found, also inject that standard. All sample injections are therefore bounded by calibrations and instrument blanks.

13.1.8 Terminated or interrupted sequences can be restarted with an instrument blank and a calibration verification, if they meet the acceptance criteria.

13.1.9 Once the performance and calibration requirement can no longer be met, corrective action must be taken, and a new sequence must be started

13.2 Initial Calibration

13.2.1 New RFs / curves are established for multipoint calibrations, whenever changes have been made to the instrument, or when the requirements for the calibration verifications (discussed below) can no longer be met.

13.2.2 Generally external calibration is performed.

13.2.3 A minimum of 3 levels have to be analyzed for the initial calibration.

13.2.4 Multipoint calibrations are generally performed at the five suggested levels presented under solutions in exhibit 10.2. for average response factors in the linear range,

13.2.5 For secondary fit curves, a minimum of 6 calibration points are needed, and a sixth level has to be added.

13.2.6 Calculate the individual response factors for areas according to the following equation. Use area counts for the computation.

Response Factors (RF)

$$RF = \frac{A_{std}}{Conc_{std} \times V_i}$$

Where:
RF = Calibration factor as area per ng
A_{std} = Peak area of standard
$Conc_{std}$ = Concentration of standard in ng per uL
V_i = Volume injected in uL

13.2.7 Evaluate the relative standard deviation for the multipoint calibration as follows:

Relative Standard Deviation of Response Factors (% RSD)

$$\% \text{ RSD} = \frac{\sqrt{\left[\sum (RF_i - RF_{avg})^2\right] / (n-1)}}{RFavg} \times 100\%$$

Where:
% RSD = % Relative standard deviation
RF_i = Response factor from the ith calibration run
RF_{avg} = Average response factor for the analyte (as below)
n = Number of calibration points for the analyte

13.2.8 For RSDs under 20%, linearity through the origin may be assumed. Average response factors are determined from a minimum of five data points (including level 1) with the following equation:

Average Response Factor (RF_{avg})

$$RF_{avg} = \frac{\sum RFi}{n}$$

Where:
RF_{avg} = Average response factor
RF_i = Response factor for ith calibration run
n = Number of calibration points

13.2.9 If RSDs are over 20%, data have to be examined, whether the response is truly nonlinear or just sporadic due to some inaccurate data points. If a certain level indicates a poor analysis/bad injection, reanalyze that level. If the response is nonlinear, the concentration range can be reduced in order to be able to use average factors.

13.2.10 Linear Regression Function not through the Origin

13.2.10.1 If the RSD of any target analyte is greater than 20%, linearity through the origin cannot be assumed. A regression equation that does NOT pass through the origin is employed. A linear regression of instrument response versus standard concentration with, response as a dependent variable (y) and concentration as an independent variable (x) is used.

13.2.10.2 This is most easily achieved by performing a linear least squares regression of the instrument response versus the mass of the analyte chromatographed. Make certain that the instrument response is treated as the dependent variable (y) and the amount as the independent variable (x). This is a statistical requirement and is not simply a graphical convention. For external standard calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument and y is the area of the response. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

13.2.10.3 The regression calculation with a weighting factor of 1 ("unweighted") will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. R has to be 0.995 for the function. If the software calculates r^2 which is the coefficient of determination, it has to be >0.99.

13.2.10.4 The problem with the application of the linear regression with an intercept often yields highly inaccurate results for low levels. To avoid a significant bias, the representativeness of the calibration model for low levels has to be checked by "re-fitting" the low data point as described below.

13.2.11 Linear Regression Functions forced through the Origin

13.2.11.1 For projects that can be analyzed according to EPA method 8000C (**Reference 2**), the linear regression "curve" may be forced through the origin by the instrument software.

13.2.11.2 This has the advantage of better accuracy for low concentrations

13.2.11.3 The coefficient of determination r^2 calculated by the software must be greater or equal to 0.99

13.2.11.4 However, the use of a linear regression or forcing the regression through zero may NOT be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards.

13.2.12 Secondary order functions

13.2.12.1 **Second order functions generally give more accurate data than linear regression not through the origin and** are preferred for RSDs over 20%. They can also be used for lower RSDs at the discretion of the analyst. A minimum of six

data points is needed to establish the curve, and the coefficient of determination r^2 must be equal or larger than 0.99.

13.2.12.2 For secondary curves, use the acquisition software to establish secondary order functions and compute data.

13.2.12.3 Since Omega cannot perform calculations with curves, positives for these analytes have to then be entered manually from the data obtained in the acquisition software.

13.2.12.4 Plot the standard and sample chromatograms at a scaling factor that will give the medium level standard at 50 percent to 75 percent of full scale.

13.2.12.5 The plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The calibration model must be continuous and monotonic. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

13.2.13 "Refitting" the calibration model

Refitting is recommended to check each calibration model for it's representativeness. **Even if the RSD is below 20% or r or r2 is above 0.0995 or 0.99**, the high or low level may not be represented by the response factor or linear regression. Especially if the calibration levels progress geometrically (2,4,8,16,32) the average factor may not be suitable for the upper end, and the linear regression may give inaccurate data for the low level if the formula has a high intercept.

13.2.13.1 This check is performed by comparison of the calculated amount with the known value. Each level may be checked, but mostly the low level and possibly the high level is generally affected by inaccuracies.

13.2.13.2 Calculate the % difference by using the following equation:

$$\% \text{ Difference} = \frac{Cc - Ce}{Ce} \times 100$$

where:

Cc = Calculated amount of standard,

Ce = Expected amount of standard,

13.2.13.3 The absolute value of the percent difference between these two amounts for every calibration level should be less than or equal to 20%. The recalculated concentration of the low level must be within ± 30% of the known concentration standards against the expected amount.

13.2.13.4 If the criteria cannot be met, the range has to be reduced. (The model is usable for the range for which the data points were within 20%.) Alternatively, if the high or low level that does not comply has to be included, a different model must be used.

13.3 Initial Calibration Verification (ICV)

13.3.1 To verify the calibration solution, an initial calibration verification (ICV) with a standard from a different source must be analyzed immediately following the initial calibration.

13.3.2 The ICV is analyzed at the medium level, and should meet the acceptance criterion of 20 %.

13.4 .Retention Time Windows

13.4.1 Retention time windows are determined for all analytes by adding and subtracting the "variances" to the retention times of the medium level standards. For the multi-peak analytes, windows are computed for all calibration peaks (or highest peaks in groups of peaks with summed areas).

13.4.2 "Custom" windows with statistical variances should be used in order to avoid false positives in particular in samples containing high levels of interferences.

13.4.3 The "custom" retention time window variances for the instrument are determined by retention time studies with statistical evaluation as follows:

13.4.4 Select at least three standard injections over a 72 hour period. If the variance is calculated for a particular analytical sequence, include standard retention times for beginning, middle and end of sequence.

13.4.5 Calculate the retention time window variances as three times the standard deviation.

Retention Time Window Variance (RTWV)

RTWV = 3S

$$S = \sqrt{\sum (RT_i - RT_{avg})^2 / (n-1)}$$

Where:
RTWV = Retention Time Window Variance
S = Standard deviation
RT_i = Retention time of the ith standard analysis
RT_{avg} = Average retention time of the analyte
n = Number of runs

13.4.6 If a window variance is less than 0.03 minute, replace it by the default value of 0.03.

13.4.7 Examine the values and modify them based on experience if necessary. ("The experience of the analyst should weigh heavily in the interpretation of chromatograms.")

13.4.8 Establish new window variances with three standard injections, if major modifications are made to the instrument, e.g. a new column is used or operating parameters are changed substantially.

13.4.9 Compute the retention time window (RTW) for identification for the analyses from the medium level (not average) standard retention time plus/minus the variance.

13.4.10 If retention time shifts of more than half the variance are observed, update the windows, using the retention times of the calibrations at the beginning of the day as the new midpoints for the daily windows.

13.4.11 Check that all continuous calibrations fall within the calculated RTWs. Samples that are not bracketed by standards within the RTW, need to be reanalyzed.

13.5 Technical Acceptance Criteria for System Performance Checks

13.5.1 Breakdown

13.5.1.1 Breakdown of endrin and 4,4'-DDT has to be computed from the analysis of the performance mix PEM in the initial sequence and every 24 hours thereafter. Breakdown for each analyte cannot exceed 15 percent.

13.5.1.2 Calculate % DDT breakdown as area of 4,4'-DDD plus area of 4,4'-DDE, divided by sum of areas of 4,4'-DDT, 4,4'-DDD and 4,4'-DDE . Equally, sum the area found of the breakdown products of endrin aldehyde and endrin ketone and divide by the sum of areas of breakdown product and endrin injected.

13.5.1.3 Note: According to methods 8081B, the breakdown is computed from the areas of the breakdown analytes. Since our Omega system cannot perform that calculation, computation is performed manually.

13.5.2 Instrument Blank

13.5.2.1 Analyze instrument blanks at the frequency indicated in the analytical sequence. As a blank, inject hexane containing the surrogate standards.

13.5.2.2 Check instrument blanks for compliance: A blank does not meet acceptance criteria, if more than one half CRQL of any analyte is found. If contamination is found, corrective action must then be taken.

13.5.2.3 All samples, which were analyzed after the last compliant instrument blank, have to be reanalyzed, after acceptable system performance has been re-established.

13.5.2.4 To prevent many re-injections, increase the frequency of instrument blanks, if samples with high contamination levels are analyzed.

13.5.3 Chromatography and Retention Time Shifts

13.5.3.1 Monitor the baseline of the chromatograms: In any standard or blank injection the baseline must return to 25% of full scale after the alpha BHC and before the DCB peaks.

13.5.3.2 Check that no retention time shifts outside the daily RTWs exist: The peaks for the targeted analytes and surrogate compounds must all elute in the established retention time windows.

13.5.3.3 Samples that are not bracketed by standards within RTWs need to be reanalyzed.

13.5.4 If at any time any of the technical acceptance criteria are no longer met, stop the analytical sequence and take corrective action. Restart the sequence with a compliant calibration verification and instrument blank, or run a new initial sequence.

13.5.5 Any samples analyzed after the last compliant check have to be reanalyzed in a compliant sequence.

13.6 Continuous Verification of Calibration (CCV)

13.6.1 In the analytical sequence inject calibration standards in the mandated intervals (after every 20 samples or less). Use medium level mixes, or alternate with the low- and high- level pesticides mixes.

13.6.2 If no toxaphene standards are injected as CCV, any samples with positive toxaphene have to be re-analyzed with a calibration standard. The concentration of that standard has to be at a level within 20% of the sample.

13.6.3 Compute the percent difference (%D) for the response of the continuous injections of the individual standards relative to the average response of the initial calibration, using the following equation:

<u>Percent Difference for Continuous Calibrations (%D)</u>

$$\%D = \frac{RF_c - RF_{avg}}{RF_{avg}} \%$$

Where:
RF_{avg} = Average response factor
RF_c = Response factor of continuous calibration (INDA or INDB)

13.6.4 The acceptance criterion for the continuous calibration verification (CCV) is 20% for medium and high levels. For a low level CCVs the acceptance limit is 40%.

13.6.5 Minor corrective action may be taken to return the GC to the conditions of the initial calibration by cutting a small piece of the column, replacing the y-connector, the septum, or the injection port liner, or by adjusting the helium flow. The corrective action is followed by reanalysis of the continuous calibration.

13.6.6 If the reanalysis does not meet acceptance criteria, a new initial calibration has to be performed.

13.6.7 Concentrations in samples analyzed during the analytical shift for compounds which exceed 20% D for the CCV are regarded estimated. Samples with positives for those analytes should therefore be re-analyzed after acceptance criteria can again be met. If this is not possible, then the concentrations have to be fagged as estimated with a qualifier.

13.6.8 If one of the two analytical columns meets CCV criteria and the variability criterion is exceeded on the other column, the compliant column should be used as quantification column. This has to be noted in the narrative. However, if the concentration in the <u>compliant</u> column is significantly higher (above 20% difference) it indicates the presence of an interference. In that case the lower value from the noncompliant column should be reported. That value has to then be flagged as estimated.

13.6.9 However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., >20%, and the analyte was not detected, samples do not need to be reanalyzed, as the verification standard has demonstrated that the analyte would have been detected were it present.

13.6.10 The sequence has to be terminated with a calibration verification check (CCV). For external calibration, all samples have to be bracketed with compliant CCVs.

14.0 SAMPLE PREPARATION PROCEDURE

14.1 Sample Extraction

If less than 90% of the required amount of sample material is received, notify client. Also discuss how to proceed with multiphase samples.

14.1.1 Separatory Funnel Extraction by EPA Method 3510

14.1.1.1 Calibrate vials to 10 mL: Pipet 10.0 mL of hexane into a vial and mark the bottom of the meniscus. Discard the solvent. Dry vial under nitrogen stream. (Instead of using calibrated vials, extract volumes may be adjusted by holding a vial containing 10 mL of hexane next to the sample vials in order to measure 10 mL.)

14.1.1.2 Equilibrate samples at room temperature.

14.1.1.3 Determine sample volume.

Procedure A:

Use entire volume supplied. For this procedure the actual sample volume needs to be recorded. Mark the meniscus of the sample, and pour the sample into a two liter separatory funnel. Determine the volume at a later time by filling water to the mark. Use different volume as available or as needed for required reporting limits.

Procedure B:

In order to obtain 1000 mL for all samples, the following two methods are acceptable:

- As long as sample bottles are uniform, i. e. supplied by the lab, bring volume of sample to 1000 mL level by comparing to a bottle with measured 1000 mL. Bring volume to 1000 mL mark by discarding some sample or filling up with spare bottle.
- Alternatively, if samples were supplied in different bottles, measure the sample volume in a Teflon measuring cylinder and add the appropriate amount from spare to make up 1000 mL. Note: Measuring cylinders have to be cleaned between samples. Use a small volume of methylene chloride and some Reagent water to rinse the cylinder and add the rinsates to the sample separatory funnel.

14.1.1.4 Rinse the bottle with 60 mL of methylene chloride and add the rinsate to the separatory funnel as the first solvent portion for extraction.

14.1.1.5 Measure the pH of the samples and, if necessary, adjust the pH between five and nine with either 10N NaOH or 1:1 sulfuric acid. If an adjustment was necessary, record in extraction log.

14.1.1.6 Prepare a method blank and a lab fortified blank (Q. C. check) by adding approximately one liter of Reagent water to a separatory funnel.

14.1.1.7 Spike the funnel intended for the lab fortified blank with 20 µL of the Q. C. spiking solution. Spike 500 µL of the TCLP solution instead for TCLP scan.

14.1.1.8 In addition, alternating, spike a lab fortified blank with 20 µL of the toxaphene Q. C. solution or 40 µL of the chlordane Q. C. solution.

14.1.1.9 With clean (preferably dedicated) syringe, add 1 mL of surrogate standard to the method blank and all samples, and Q. C. samples.

14.1.1.10 For sample aliquots selected for matrix spike/matrix spike duplicates, also add 20 µL of the Q. C. spiking solution, using clean syringe. (Spike 500 µL TCLP solution instead for TCLP scan.) To obtain data points for toxaphene and chlordane, rotate the Q. C. spiking. Use clean syringes!

14.1.1.11 Extract sample with the added 60 mL methylene chloride (MC) rinsate by shaking for two minutes, venting periodically. (Methylene chloride builds up pressure fast, therefore vent after sealing and shaking just once.)

14.1.1.12 Allow solvent layer to separate from water layer for a minimum of ten minutes and drain MC.

14.1.1.13 Repeat extraction two more times for a total of three extractions.

14.1.1.14 For emulsions of more than one third of the solvent layer that cannot be sufficiently separated in the separatory procedure, the extraction of the sample should be restarted in a continuous extractor.

14.1.1.15 Pour combined extracts through funnel with approximately 1-1/2 inch anhydrous granular sodium sulfate. Collect in KD concentrator or Zymark concentrator tube. Rinse with 20 to 30 mL of MC.

14.1.2 Extraction with Continuous Liquid/Liquid Extractor by EPA Method 3520

14.1.2.1 Assemble Rot-X-Tract according to manufacturer's instructions.

14.1.2.2 Fill about 150 mL MC into extractor vessels and add 1-2 boiling chips.

14.1.2.3 Measure sample volume as in Separatory Funnel procedure above.

14.1.2.4 Measure pH of samples with pH paper and if needed adjust sample pH between five and nine with either 10 N NaOH or 1:1 sulfuric acid. Note any adjustments in extraction log.

14.1.2.5 Slowly pour sample on top of the MC. Avoid mixing with the MC layer in order not to introduce water into the overflow, and do not bring meniscus any closer than 0.5 cm above the overflow.

14.1.2.6 Rinse the bottle with about 50 mL of methylene chloride and slowly add the rinsate to the extractor body. Add more MC if needed. The volume in the receiving flask has to be at least 100 mL.

14.1.2.7 Adjust the solvent/sample interface in the extractor to be approximately one cm above overflow, by adding Reagent water.

14.1.2.8 Fill two extractors with approximately one liter of Reagent water for method blank and lab fortified blank (LFB).

14.1.2.9 Spike the extractor intended for the lab fortified blank with 20 μL of the Q. C. spiking solution. (Spike 500 μL of the TCLP solution instead for TCLP scan.)

14.1.2.10 In addition, alternately spike a lab fortified blank with 20 μL of the toxaphene Q. C. solution or 40 μL of the chlordane Q. C. solution.

14.1.2.11 For sample aliquots selected for matrix spike/matrix spike duplicates and the matrix spike blank (MSB), if required for ASP protocol, also add 1 mL of the MS spiking solution, using clean syringe. (Spike 500 μL TCLP solution instead for TCLP scan.)

14.1.2.12 With clean (preferably dedicated) syringe, add 1 mL of surrogate standard to the method blank and all samples, and Q. C. samples.

14.1.2.13 Raise the temperature of the water-bath to achieve a steady

boil of the MC. Adjust to condense 10-15 mL per minute.

14.1.2.14 Reflux the MC for 12 hours. If the condensation rate was less than 10 mL/min, then the extraction period has to be extended to 18 hours.

14.1.2.15 Cool down the system and remove the round bottom flasks.

- Filter extract through a layer of approximately 1-1/2 inches of Na_2SO_4 in powder funnel, if needed. (The Na_2SO_4 has to be baked in a kiln for at least two hours at 400°C and rinsed with MC after packing in the funnel before usage.)
- Concentrate extracts by any of the methods described in the concentration section.

14.1.3 Low Level Extraction of Soil/Sediment Samples by Pressurized Fluid Extraction, EPA Method 3545A, with Accelerated Solvent Extractor

14.1.3.1 As needed, the particle size of dry sediment/soil and dry waste samples is reduced by grinding to a size of 1 mm.

14.1.3.2 Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. The analyst may add palletized diatomaceous earth or other clean, dry reagents to the sample to make it more amenable to grinding.

14.1.3.3 % moisture is determined separately on 5-10g of the sample in the Wet Chemistry Department. If the sample contains a water layer, the water has to be decanted, before % moisture is determined.

- % moisture is measured separately on 5-10 g of the sample in the Wet Chemistry Department.
- For samples with a % moisture between 30 and 50%, the sample bottle from the organic department has to be used. (For dry samples the weight does not change significantly, and concentrations are regarded estimated for samples above 50% moisture.) If the sample was decanted, the particular sample has to be submitted to the wet chem. department for analysis of % moisture.

14.1.3.4 The weighed portion of the sample is heated in aluminum weighing dishes in the oven at 100 °C for 24 hours.

14.1.3.5 The samples are cooled in a desiccator until constant weight is reached.

14.1.3.6 % solid and % moisture are computed from the weight difference before and after drying.

14.1.3.7 A sample portion, to be extracted by method 3545A, is weiged in an aluminum weighing dish. Generally 15 g are used.

14.1.3.8 Typically, an equal volume of pelletized diatomaceous earth is added. More or less of the adsorbent may be used depending on % moisture. Make sure quantity used fits into the extraction cell.)

14.1.3.9 The sample and sorbent are mixed with a narrow stainless steel spatula in the weighing dish.

14.1.3.10 The mix is then transferred into the 33 mL stainless steel cylinder. Wipe adhering sample material from the spatule onto a small piece of filter paper and add the paper to the extraction cell.

14.1.3.11 For small sample weights load into smaller cells of 22 mL. Additional adsorbent may be added to the cell, to fill any void volume in the extraction cells to minimize amount of solvent used

14.1.3.12 More or less of the adsorbent may be used depending on % moisture. Additional adsorbent may be added to fill any void volume in the extraction cells to minimize amount of solvent used.

14.1.3.13 Fill two cells with diatomaceous earth for the method blank and the lab fortified blank.

14.1.3.14 Weigh 2 extra aliquots of the matrix spike sample for the MS and MSD.

14.1.3.15 Spike the MS, MSD, MSB (if required for ASP protocol) with 0.5 mL of the matrix spike solution. (Since only 15 g are extracted the spike amounts are half of those for sonication with 30 g due to a higher concentration factor.)

14.1.3.16 Spike the lab fortified blank (LFB) with 10 µL of Q. C. spiking solution. In addition, alternately also spike a LFB with 10 µL of the toxaphene Q. C. solution or 20 µL of the chlordane Q. C. solution.

14.1.3.17 With clean (or dedicated) syringe, spike all samples and Q. C. samples with one mL surrogate solution.

14.1.3.18 Place filter disk on the top of the cells. Wipe outside threads of the cells with soft tissue to remove all particles, to assure leak-free closure. Tightly seal screw cap.

14.1.3.19 Load up to 24 extractor cells containing samples and Q. C. samples onto the instrument auto-sampler tray.

14.1.3.20 Fill solvent bottle with 1:1 methylene chloride / acetone. For best results, with very wet samples (e.g. 30% moisture), reduce or eliminate the quantity of hydrophilic solvent used.

14.1.3.21 Consult manufacturer's instructions about solvent volume to be used. The volume is not a function of the sample size but the cell volume.

14.1.3.22 Load cleaned and dried receiving vials. For the 33 mL cells load 60 mL receiving vials and 40 mL vials for smaller cell sizes.

14.1.3.23 Start the automated extraction process.

14.1.3.24 The extraction solvent is introduced under pressure and the cells are heated to 100°C. They are held at that temperature under pressure of ca. 2000 psi for 5 minutes.

14.1.3.25 The solvent is then expelled with nitrogen into the collection vessels.

14.1.3.26 To maximize extraction efficiency, the cells are flushed for several minutes with small aliquots of solvent.

14.1.3.27 The fractions are collected in the receiving vials.

14.1.3.28 After cool-down, the extracts have to be dried and concentrated.

14.1.5 Medium Level Extraction of Soil/Sediment Samples with Accelerated Fluid Extractor (Pressurized Fluid Extraction Method 3545A)

14.1.5.1 In all following steps be very careful to avoid cross contamination. Wipe neck of container, re-close containers immediately and do not reuse any utensils that contacted sample.

14.1.5.2 Perform preparatory steps of decanting, mixing, pH measurement and determination of % moisture as in the low level extraction procedure.

14.1.5.3 2 g of sample are weighed into the 11 mL stainless steel cylinder. Approximately 5 g of pelletized diatomaceous earth is added and mixed with a narrow stainless steel spatule. Wipe adhering sample material from the spatule onto a small piece of filter paper and add the paper to the extraction cell.

14.1.5.4 Additional adsorbent may be added to fill any void volume in the extraction cells to minimize amount of solvent used.

14.1.5.5 Fill two cells with equivalent amounts of diatomaceous earth for the method blank and the lab fortified blank.

14.1.5.6 Weigh 2 extra aliquots of the matrix spike sample for the MS and MSD.

14.1.5.7 Double all spiking solution volumes for Q. C. and surrogate spiking solutions of the low level extraction. (Final extract volume is double.)

14.1.5.8 Place filter disk on the top of the cells. Wipe outside threads of the cells with soft tissue to remove all particles, to assure leak-free closure. Tightly seal screw cap.

14.1.5.9 Load up to 24 extractor cells containing samples and Q. C. samples onto the instrument auto-sampler tray.

14.1.5.10 Fill solvent bottle with 1:1 methylene chloride / acetone. For best results, with very wet samples (e.g. 30 % moisture), reduce or eliminate the quantity of hydrophilic solvent used.

14.1.5.11 Load cleaned and dried 40 mL receiving vials.

14.1.5.12 Start the automated extraction process.

14.1.5.13 The extraction solvent is introduced under pressure and the cells are heated to 100°C. They are held at that temperature under pressure of ca. 2000 psi for 5 minutes.

14.1.5.14 The solvent is then expelled with nitrogen into the collection vessels.

14.1.5.15 To maximize extraction efficiency, the cells are flushed for several minutes with small aliquots of solvent.

14.1.5.16 The fractions are collected in 40 mL receiving vials.

14.1.5.17 After cool-down the extract have to be dried and concentrated to 10 mL.

14.1.6 Dilution Procedure by EPA Method 3580A for Oil and Non-aqueous Liquids

14.1.6.1 Calibrate vials (that can be capped) to 10 mL and 5 mL: Pipet 10.0 mL and respectively 5.0 mL of hexane into a 12 mL vial and mark the bottom of the meniscus. Discard this solvent. Dry vials. (Alternatively hold a calibrated vial or a vial containing

10 mL (or 5 mL) of hexane **next** to the sample vial that is being brought to the final volume.)

14.1.6.2 Cap vials and determine the tare weights.

14.1.6.3 Transfer approximately 1 g sample to the vial and wipe the mouth of the vial with a tissue to remove any sample material. Cap the vial immediately to avoid any cross-contamination.

14.1.6.4 Weigh to the nearest 0.01 g. Record the weight.

14.1.6.5 Spike liquid matrices with the Q. C. spikes and surrogates as above for water samples.

14.1.6.6 For oil samples, a high dilution is necessary, and high spiking levels are needed. Spike samples with five times the amounts used in the water extractions.

14.1.6.7 Bring the volumes to 10 mL by adding hexane to the 10 mL marks. Agitate (vortex) vials to achieve homogenous concentration.

14.1.6.8 Using disposable tips, pipet 1 mL of each vial into vials, calibrated to 5 mL.

14.1.6.9 For oil samples dilute to 5 mL with hexane.

14.2 Extract Concentration

14.3.1 KD Concentration

14.2.1.1 Add 1-2 boiling chips to evaporative flask, containing combined dried extract portions, and attach a macro three ball Snyder column.

14.2.1.2 Pre-wet the column by adding one mL MC to the top.

14.2.1.3 Place the KD on a hot water bath (60°C to 80°C) so that the concentrator tube is partially immersed in hot water and the lower rounded surface on the flask is swept by steam. In uncovered water baths it is necessary to also immerse the flask partially.

14.2.1.4 Concentrate to 1-2 mL and allow to drain and cool at least 10 minutes. (Make sure samples do not go to dryness.)

14.2.2 Extract Concentration on Turbo Vap II

14.2.2.1 The Turbo Vap II provides automated solvent concentration with nitrogen with sensor controlled endpoint.

14.2.2.2 Collect extracts in 200 mL Turbo Vap concentrator tubes.

14.2.2.3 Set temperature to 50°C and N_2 pressure to seven psi. Select

end volume of 1 mL. Load 200 mL tubes with sample extracts and close cover.

14.2.2.4 When "ready" light flashes, the end point is reached, and the tubes are removed from the concentrator.

14.2.2.5 Diligently transfer concentrate to small tubes for solvent exchange on the N-EVAP as described below. Carefully rinse the walls and particularly the tip of the concentrator tubes for quantitative transfer of the samples.

14.2.3 Solvent Exchange with Micro KD

14.2.3.1 Rinse KD flasks and joints with several small portions of hexane and collect rinsates in receiving vial.

14.2.3.2 Add 5 mL hexane and new boiling chip.

14.2.3.3 Attach micro three-ball Snyder column. Pre-wet column by adding hexane through the top to bring to a total of about 10 mL.

14.2.3.4 Concentrate extract to approximately 1-2 mL. Add another 5 mL of hexane.

14.2.3.5 Concentrate again to approximately 1 mL.

14.2.3.6 Transfer concentrate to a calibrated 10 mL vial and bring final volume to 10 mL with hexane rinsings of the KD vials. For volume adjustment after GPC cleanup refer to that section.

14.2.3.7 Vortex samples to guarantee uniform solution before taking aliquot for Florisil cleanup or sulfur cleanup.

14.2.4 Nitrogen Evaporation with Solvent Exchange

14.2.4.1 Adjust the temperature of the N-EVAP to 30°C -35°C and install fresh Pasteur pipets as nozzles for each new set of samples to avoid cross contamination.

14.2.4.2 Load test tubes or receiving vials with sample extracts on the manifold and lower nozzles until they almost touch the surface of the extracts.

14.2.4.3 Turn on nitrogen supply at 10-20 psi and adjust the individual valves to evenly supply each position with a gentle stream of nitrogen. (Observe distortion of solvent surface to gauge intensity.)

14.2.4.4 When the volume in a vial reaches 0.5-1 mL, add 3 mL of hexane.

14.2.4.5 The previous step is repeated twice.

14.2.4.6 The hexane concentrate is transferred to a calibrated 10 mL vial with a Pasteur pipet, and the volume is adjusted to 10 mL with hexane rinsings of the concentrator vial.

14.2.4.7 For 15 g samples extracted by ASE bring end volume to 5 mL.

14.3 Extract Cleanup

Many samples contain contaminants that either interfere with the analysis or will cause problems for the analytical system. These contaminants have to be removed by appropriate cleanup steps.

The spiked blank, sample spikes, (if sample needed cleanup,) and the method blank have to be subjected to the same cleanup steps as the samples. The obtained **recoveries for the lab fortified blank (LFB) have to meet the accuracy Q. C. limits**. (If no historical data are available for recoveries obtained, **perform the LFB cleanup and check recoveries before any sample are subjected to the cleanup.**

14.3.1 Gel Permeation Chromatography (GPC) Cleanup by EPA Method 3640A

To remove high molecular weight interferences from sample extracts, GPC cleanup is performed on the methylene chloride concentrates. GPC cleanup has to be performed on all soil extracts and on water samples that contain high molecular substances that interfere with the analysis.

Cleanup is executed on the AccuPrep system with a 700 mm Bio Beads column.

14.3.1.1 Generating a Method for the AccuPrep GPC System with the AccuChrome Software

- To generate the programs, follow the examples supplied by the manufacturers.
- Enter "reagents", i.e. the solvents to be used, and their densities. These entries are not critical; the flow rates will be affected slightly, but will be the same in the calibration and the samples.
- Complete entries for the prompts of the setup parameters for system configuration in the "Defaults Tab", "Data Collection Tab", "Preferences Tab", "Hardware Tab" according to the manufacturer's manual. Consult the J2 instructions to set pressure limits, etc. Utilize default values for times, until data from the calibration can be entered.
- Set the pump speed in the method to the desired flow-rate. The nominal flow-rate is 5 mL/min. However, this flow-rate may be adjusted as long as the required peak resolutions

are obtained. (Once the flow-rate has been established, and the method is calibrated with that flow-rate, it may not be changed until a new calibration is performed, because it is critical for the correct collected fraction volumes, since times are entered from the calibration chromatogram.)

- Save method with method name in data system.

14.3.1.2 Packing of the GPC Column

GPC columns can be purchased already packed, or empty columns can be charged with Bio Beads by the following procedure.

- Soak 70 g Bio Beads SX-3 in 400 mL beaker with methylene chloride overnight. Maintain enough solvent to cover the beads at all times. Tightly cap with aluminum foil and leave hood running.
- Before filling column, make sure that top and bottom pistons fit tightly. If they slip too easily, replace the o-rings for tighter fit, otherwise gaps will occur in column later.
- Transfer content of 400 mL beaker to separatory funnel.
- With bottom inlet seals in place, add some methylene chloride to the column and then place funnel on top and fill beads into column. Drain the methylene chloride into a beaker under the inlet.
- Add methylene chloride to the slurry as needed, keeping slurry “wet”. It is important to fill continuously to achieve a uniform packing without gaps for efficient chromatographic separation.
- When solvent is drained, rinse sides of funnel and column with methylene chloride. Wait until all excess solvent above the beads is drained and surface begins to look dry. Do not allow the beads to drain dry! If this happens, re-wet the beads.
- Wipe beads from inner wall of column, loosen the seal on the plunger assembly and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed, but loose enough so that the plunger can be moved.
- Push the plunger until it meets the gel, then compress the column bed about 3-4 cm until it reaches the desired bed length.

- Tighten the plunger and re-install the end caps and tighten the nylon screws.
- Connect column to J2 System. (See J2 instructions). Pump methylene chloride through the column at five mL per minute for several hours, until all air bubbles are removed.
- Observe the column pressure. If pressure exceeds 15 psi, temporarily exchange flow direction until gel is uniformly wetted and swollen, and pressure drops to 7-12 psi. The bed-length may have to be adjusted with slightly loosened inlet plunger to achieve a final pressure of 7 -12 psi.

14.3.1.3 Calibration of the GPC Column

- Calibration has to be performed weekly and after any maintenance steps affecting elution of compounds or if elution parameters can no longer be matched.
- Manually verify the pump flow rate with a volumetric flask. The displayed pump speed may not accurately reflect the flow rate. To adjust the flow-rate, enter the time used to collect 25 mL in the prompt under "Pump Control" and enter "update flow-rate calibration".
- Warm up and zero the UV detector.
- Follow the J2 user guide to start a run and upon completion import the data into AccuChrome.
- Use the AccuChrome software to label peaks and check the resolution.
- Evaluate peak resolution of the UV chromatogram. All peaks have to be resolved at least 85 percent and perylene and sulfur 90 percent. If this is not achieved, the system may include air or require cleaning or repacking of column.
- Determine "dump" and "collect" cycles for pesticides / PCBs: Fraction I ("dump" cycle) contains corn oil and most bis(2-ethylhexyl)phthalate (BEHP); Fraction II ("collect" cycle) must contain more than 95 percent of methoxychlor and less than 15 percent of BEHP. The fraction is terminated after perylene and before the sulfur peak; and Fraction III ("wash" cycle) includes the sulfur eluate and also provides wash time with MC between injections.
- Check that retention times for bis(2-ethylhexyl)phthalate and perylene are within ± 5% of previous calibration Use the "sample comparison report" of AccuChrome to compare the calibration with the previous calibration. If acceptance

criteria are exceeded, take corrective action according to the "Maintenance" section below and recalibrate.

- AccuChrome will mark the time at the lowest point of the valley between the peaks. Use these times to enter the collection times for Fraction II in the method in the AccuChrome data system and save.

14.3.1.4 Calibration Check of the GPC Column

- Within seven days prior to analyzing samples, the column performance (calibration) has to be checked, by running the matrix spike solution and an aroclor solution through the column, and analyzing the extracts by GC.
- Prepare pesticide GPC calibration check solutions by concentrating 1 mL of the matrix spike solution and 1 mL of surrogate solution to less than 1 mL and diluting to 10 mL with MC.
- Perform GPC cleanup using the same method, i.e. the same "dump" and "collect" cycles as for samples.
- Concentrate the collected Fraction II and solvent exchange to 10 mL hexane.
- Omit Florisil cleanup.
- Repeat procedure with MC solution containing 0.1 µg/mL each of AR1016 and AR1260 in methylene chloride. (Spike 500 µL of the Q. C. solution and bring to 10 mL.)
- Note: To simulate samples processed on the ASE instrument, the concentrations of the two GPC check solutions can be reduced to half. To compensate for that, the collected fractions have to be concentrated to 2.5 mL instead of 5 mL.
- Perform GC analyses of the concentrated and solvent exchanged eluates of the calibration checks for pesticides and PCBs.
- Determine recoveries for the pesticides GPC check. Recoveries must be 80 to 110 percent
- Examine aroclor pattern for AR1660 injection. The ratio of peaks should not be changed from standard pattern.

14.3.1.5 GPC Blank

At the same frequency as the calibration check (weekly), and after particularly dirty samples, a GPC blank has to be run through the system:

- The GPC blank consists of surrogate solution diluted 1:10 with MC.
- Fraction II has to be concentrated, solvent exchanged and analyzed by GC/ECD.
- Check the GPC blank for contaminations. No interferences with targeted analytes should be present, and no other large peaks should be found in the chromatograms.
- Positives cannot exceed half CRQL according to the ASP protocol. For internal quality no interferences larger than minimum detection limits (MDL) of pesticides (on either analytical column). Only if this criterion cannot be achieved by remedial action, the ASP limit of ½ CRQL should be applied.
- Note: The calibration solution contains a fairly high concentration of methoxychlor, which causes carryover. If a blank is run on the GPC instrument shortly after the calibration procedure, concentrations larger than ½ CRQL of methoxychlor will be found in the blank. Several runs with base-neutral sample extracts have to be processed first, or extensive rinsing with methylene chloride has to occur before the pesticides instrument blank can be processed.
- NO SAMPLE CAN BE CLEANED UP ON THE SYSTEM, UNTIL AN ACCEPTABLE GPC BLANK CAN BE OBTAINED.
- Any or all of the cleaning steps listed below in the maintenance section can be performed in order to achieve clean GPC blanks.

14.3.1.6 Determination of Organics Concentration in Samples

- Due to the loading capacity of the column, the sample extract should not contain more than 40mg/mL of extracted solids.
- For samples with high organic content, e.g. sludge, several aliquots have to be cleaned up and combined after cleanup. To achieve this, the sample extracts have to be diluted accordingly.

- If a sample appears more viscous than a 1:1 glycerol/water solution, this is an obvious indication that it requires dilution, before it can be run on the GPC apparatus.
- Check any suspected samples for organics content by evaporating an extract aliquot of 100 uL in aluminum weighing pan. The residue should not exceed 4 mg.

14.3.1.7 Sample Cleanup on the AccuPrep System

- Filter all samples through 5 micron filter (or smaller) to remove particles that can get lodged in the valves of the system and can cause scratches and leaking. Fill a 10 mL syringes with the extract, attach a filter cartridge, and push the sample through.
- Add the sample extracts, usually 10 mL, to 16 x 100 mm test tubes and tightly cover with aluminum foil to prevent evaporation. (A minimum of 8 mL should be loaded. The system uses 7.5 mL to fill the loop and perform rinsing steps.)
- Load all samples in the sequence as they are to be processed.
- Make sure that the methylene chloride reservoir is filled and the waste receptacle is empty.
- Charge the fraction collector with clean empty 200 mL receiving flasks.
- Enter a “sequence” that will designate how many samples will be processed. A maximum of 25 samples can be processed in one sequence. The number is limited by the number of positions in the collector.
- Assign which methods are to be used and ready the AccuChrome data system for acquisition.
- Start processing the samples.

14.3.1.8 Maintenance of the GPC System

To prevent retention time shifts and contamination of the samples by a dirty GPC system, the following maintenance steps have to be strictly adhered to.

- Any maintenance performed on the system has to be recorded in the GPC Calibration/Maintenance Log Book.
- The program for the J2 system includes “wash” steps of all parts of the system. If carryover is observed, the volumes of the rinses and amount of rinses may have to be increased.

- Elution times have to be monitored and have to remain within 5% for calibration checks.

  Constant room temperature needs to be maintained. Large fluctuations will cause retention time shifts. 22°C should not be greatly exceeded, to prevent out-gassing of the methylene chloride.

  Changes of peak elution times can also be caused by clogs, leaks, accumulation of particles in the column, deposits in the valves. Leaks may occur on scratched valves caused by deposited particles.

- Particularly dirty samples should be pre-filtered before the filtration through the 5 μ filter.
- For problems with the operation of the system consult the J2 Troubleshooting Guide.

  Clogs in the system can be isolated by by-passing the column. Dismantling and then sonicating components can be used to remove the clog.

- To ascertain that no contaminations are introduced into the samples during GPC cleanup, GPC blanks are run (at a minimum) every 7 days.
- To clean the system, if no acceptable GPC blank can be obtained, pump MC through for one to two hours, or flush several portions of 5 mL butyl chloride through the system.
- According to J2 recommendation, a mix of 5% toluene in methylene chloride can be pumped through the column to regenerate a column that no longer meets the separation criteria or shows contamination. Load 10 vials, each containing 10 mL of the solvent mix, and let them run through the system.
- Use of pre-columns ("guard" columns) will improve performance of the GPC column and extend the period until the column needs to be replaced.

14.3.1.9 Concentration of GPC extract

- The eluate of the GPC column of ca. 60 mL is collected in the collector in 200 mL Turbovap vials.
- Concentration is done in the Turbovap II according to the instructions under sample concentration.
- If the extraction was performed on 30 g, the volume of the extract is adjusted to 5 mL with hexane. If only 15 g of

sample were extracted, the volume of the extract is brought to 2.5 mL instead.

14.3.2 Sulfur Cleanup with TBA by EPA Method 3660B

Even though most of the sulfur is removed in the GPC procedure, residual amounts still sometimes cause interferences during analysis. To avoid time-consuming re-analyses, all soil extracts should therefore be subjected routinely to sulfur cleanup, even if presence is not obvious.

- Analyze endrin aldehyde before performing TBA cleanup or use different aliquots (cleaned and not cleaned), because it decomposes.
- Include separate sulfur cleaned blank (or aliquot of normal method blank), and subject an aliquot of the lab fortified blank (LFB) to TBA cleanup.
- Check volume of sample extracts and adjust if necessary. Vortex before taking aliquot.
- Perform this cleanup before the Florisil cleanup.
- Place aliquot larger than what is needed for Florisil into 10 mL screw cap vial.
- Add approximately one mL TBA sulfite (e. g. one disposable pipette) reagent and double that volume of 2-propanol, cap and shake for one minute. If sample is colorless or color unchanged, and if clear crystals are present, enough sodium sulfite is present. If the precipitated sodium sulfite disappears, add more crystalline sodium sulfite in approximate 100 mg portions until a solid residue remains after repeated shaking.
- Add five mL distilled water and shake for one minute. Allow to stand five to ten minutes.
- Transfer the hexane (top) layer to a small capped vial and mark the meniscus before storing in the refrigerator, or proceed with Florisil cleanup.

14.3.3 Sulfur Cleanup with Copper by EPA Method 3660B

- Check volume of sample extracts and adjust if necessary. Vortex for approximately 20 seconds.
- Perform sulfur cleanup before Florisil cleanup.
- Place aliquot larger than what is needed for Florisil cleanup into small screw cap vial.
- Clean copper powder by rinsing in succession with diluted nitric acid, water, and acetone and then dry with nitrogen.
- Add two grams (approximately to 0.5 mL level) to each vial.

- Shake capped vials for one minute.
- Remove an aliquot of this extract into clean vial. Note: Do not leave extract on copper to prevent degradation of analytes.
- The concentration of this extract aliquot represents 10 mL total extract volume. Mark the level of the meniscus on the vial. Store in refrigerator for analysis, or proceed with Florisil cleanup.

14.3.4 Florisil Cleanup by EPA Method 3620A

This cleanup procedure elutes all pesticides into one fraction with a moderately polar solvent mix that leaves more polar contaminations adsorbed on the cartridge packing. Cleanups are performed with 1 mL extracts on 1 g cartridges, using a Supelco manifold.

14.3.4.1 Florisil Cartridge Performance Check (FCPC)

Each batch or lot of cartridges has to be tested with the performance test mixture, to assure adequate pesticide recovery.

- Mount cartridge to be tested on Supelco manifold. Keep individual cartridge valves closed until mounting solvent.
- Mix 0.5 mL of the FCPC solution and 0.5 mL of the medium level individual standard mixture A and 4 mL of hexane. Blow solution down to a volume of 0.5 mL with nitrogen.
- Mount the concentrate onto a Florisil cartridge and elute according to the procedures described below. Concentrate eluates to 1 mL according to procedure for nitrogen concentration, but omit the steps with hexane addition.
- Analyze by GC. Concentration will be approximately half of the medium level INDA concentration. Recovery for the targeted compounds must be 80 to 120 percent, and TCP recovery must be under five percent.

14.3.4.2 Sample Cleanup on Florisil Cartridges

- Attach Supelco manifold to vacuum or use gravity feed. Adjust vacuum to approximately one psi.
- Mount 1g Florisil cartridge on manifold. Keep individual valves closed until rinsing. Rinse each cartridge with three portions of 5 mL 90:10 hexane / acetone mix, opening the valve to adjust to a flow-rate of approximately 3 mL per minute. DO NOT ALLOW CARTRIDGES TO GO DRY!
- Place labeled 10 mL collector tubes under cartridges.

- Before taking aliquots of the sample extracts, make sure they have the correct volume. (After solvent exchange 10 mL, after GPC 5 mL, and after sulfur cleanup at marked meniscus.)
- Adjust as necessary and briefly vortex to assure that solution is homogeneous, if an adjustment was made.
- Measure 1 mL of sample extract in a clean syringe and mount onto cartridge.
- Elute each cartridge with 9 mL 90:10 hexane / acetone mix and 2 mL hexane. Use elution solvent to rinse extract container for quantitative transfer.
- Concentrate each eluate to 1 mL according to the nitrogen concentration procedure, omitting steps 4 and 5, of hexane addition. Transfer concentrates to auto-sampler vials and store in refrigerator for analysis. This 1 mL extract represents 10 mL total extract volume.

15.0 INSTRUMENT ANALYSIS PROCEDURE

15.1 Setup and Parameters

15.1.1 Optimize the GC operating conditions to achieve best resolution for all peaks on both columns. Typical operating parameters for the analytical system are presented in **Table 2**.

15.1.2 The instrument parameters are entered into a method that is stored in the GC. Make sure that conditions are not changed once calibration is performed.

15.1.3 During setup, program the GC with a sequence for the auto-sampler and the method to be used. Consult the instrument manufacturer's manual for programming information.

15.1.4 Enter a "sequence" corresponding to the auto-sampler sequence in the TotalChrom software and specify the acquisition method for the analytical scan and samples to be analyzed.

15.1.5 Enter RT windows in the method as discussed in the calibration section.

15.2 Sample Analysis

15.2.1 The solutions of standards and extracts of samples and Q. C. samples are loaded onto the auto-sampler in the sequence discussed under "Analytical Sequence" in the calibration section and recorded in the log book.

15.2.2 All solutions are sequentially injected through a splitter onto two 0.32 mm fused silica capillary columns.

15.2.3 Compounds are segregated on the analytical columns with a temperature program to optimize resolution and detected on the two ECD detectors.

15.2.4 The signals from the detectors are recorded on the data system.

15.2.5 The acquired data are processed with the chromatography software and evaluated and reported as follows.

16.0 DATA ANALYSIS AND CALCULATIONS

16.1 Identification

16.1.1 Update the midpoints of the RT windows in the method and process the sequence of samples to obtain tentative identifications from the data system printouts.

16.1.2 Compounds eluting in the RT window are tentatively identified as positives by processing the acquired data with the method.

16.1.3 Edit computer identifications manually by comparing RTs with the actual RT windows and omit analytes that are only present on one column.

16.1.4 Chromatograms for samples cannot show large peaks interfering with targeted analytes. If retention times for targeted analytes are masked by interferences, further cleanup is necessary. Any or all of the steps in the cleanup section of the sample preparation could be repeated.

16.1.5 If further cleanup is unsuccessful, dilutions have to be analyzed. Report the steps taken in the narrative.

16.1.6 Report positives for pesticide if "hits" in the RT windows are found on <u>both</u> columns, and peaks are above ½ the CRQL.

16.1.7 The determination whether a multi-peak compound (toxaphene or technical chlordane) is present is based on pattern recognition. Match the major peaks by retention times and compare the ratios with those in the standard chromatogram. If a multi-peak compound is found in a sample, the standard for that compound has to be analyzed in the sequence within 72-hours from when it was found.

16.1.8 Note that fingerprints for toxaphene differ depending on origin due to dechlorination.

16.2 Quantification and Reporting of Results

16.2.1 Individual pesticides identified on both columns are quantified with the average calibration factor, determined in the initial calibration, according to the equation:

Analyte Concentration (Conc)

$$\text{Conc} = \frac{A * V * DF}{RFavg * Vi * W}$$

Where:

Conc = Concentration in sample as ug/L or ug/kg
A = Peak area
RF_{avg} = Average calibration factor for area per ng
Vi = Volume injected in uL
V = Extract volume in uL
W = Volume of sample in mL or dry weight in g
DF = Dilution factor

If GPC was performed and the volume after GPC is entered, calculate the concentration as:

$$\text{Conc (GPC)} = \frac{A * Vgpc * DF * 2}{RFavg * Vi * W}$$

Where:
Vgpc = Volume of extract in µL after GPC

16.2.2 Record results for both columns on Form X Pest-1 for reporting with full documentation.

16.2.3 For multi-peak compounds, individual peaks are quantified with the equation given above. The quantities computed are summarized on Form X Pest-2 for both columns.

- A minimum of three peaks is required for quantification. Four to six peaks should however be included for computation, because of the likelihood that peaks have to be omitted from the average because they do not exhibit the correct ratio to other peaks in the pattern.
- Concentrations computed for the individual peaks should be within a reasonable proximity of about 30 percent (or 50 percent for weathered patterns). Larger deviations either indicate severe weathering or co-elution with interferences. Do not include peaks with unresolved interferences in the average.
- The average amount for the peaks is calculated for each column on Form X.

- Due to inconsistencies of the peak ratios for toxaphene due to dechlorination in toxaphene from different origins, more accurate quantification is obtained by the total area approach unless many interferences are present. Great care has to be taken in the integration to assure that sample and standard receive the same baseline treatment. Otherwise large inaccuracies are introduced in the integration.
- Chlordane may also exhibit quite different ratios for the peaks and may have to be quantified by total area computation.

16.2.4 Report the lower result of the two columns for method 8081B by ASP B For CLP-like reporting, the qualifier "P" is used with the results to indicate discrepancies of results on the two columns above 25%.

16.2.5 Method 8081B stipulates in 7.5.5 that **unless otherwise stated in the project plan for the samples**, the more conservative **higher** result should be reported. If %D for the two values exceeds 40%, an interference is likely. The retention time of the peak should be checked and the peak shape examined for distortion to ascertain whether an interference is co-eluting. In presence of an obvious interference, the lower value can be reported.

16.2.6 However, most of our clients that request "routine" analysis are used to reporting of the lower result. New clients will have to consulted, whether lower result reporting is acceptable for their project.

16.2.7 In method 8000 C, exhibit 11.10.4.2 states that reporting of the lower result is appropriate. This would apply for 8081B.

16.2.8 In CLP-like reporting, quantities under the quantification limit (PQL), but which are "reportable", are reported as positives, but are flagged as estimated values with the qualifier "J". Whether a compound is "reportable" depends on the MDL level, the noise and the level of interferences. Generally 1/2 of the PQL has been found to be a reasonable cutoff, to eliminate interferences and contaminations to be reported as positives.

16.2.9 If levels of positive pesticides exceed the calibration range, samples have to be diluted. The dilution should show the diluted analyte in the upper half of the calibration range. Use the largest analyte to determine the dilution factor.

16.2.10 For CLP like reporting, submit both results and chromatograms for the original and dilution analysis in the data package.

16.3 GC/MS Confirmation

16.3.1 If the concentration of an analyte exceeds the MDL of the GC/MS instrument, this compound has to be confirmed by GC/MS analysis.

Generally, 1/3 (or lower) of the quantification limits of 100 ug/L, and respectively 3300 ug/kg, can be detected for an extract volume of ten mL.

16.3.2 For concentrations under these detection limits (30 ug/L or respectively 1000 ug/kg), concentrate the GC extract 10 times or analyze the GC/MS extract of the same sample. Together with the sample, analyze a reference standard containing the analyte at or above the quantification level and compare spectra and retention times.

16.3.3 To be identified as a positive, a peak must elute at the expected relative retention time (within ± 0.06 RRT units), and the three major ions must be present in the same ratios as in the standard (± 20%) and elute within one scan. To document the last criterion, extracted ion profiles (EIC) for the major ions can be submitted instead of spectra.

16.3.4 The major ions of the targeted analytes are presented in **Table 4**.

16.3.5 If the analyte is confirmed it is qualified with “C” on the report. If no confirmation was obtained, a laboratory-generated qualifier is noted on the report. Laboratory qualifiers that can be used are X, Y, and Z. (Use “X”, if not already otherwise designated.)

17.0 METHOD PERFORMANCE

17.1 The suitability of the method for the analytes tested was already determined, when the method was developed. During “method startup” in the laboratory, the capability of the analysts, suitability of equipment, materials and instrumentation was tested by analyzing four spiked blanks at medium level and seven low level spikes to check the sensitivity.

17.2 The method performance has to be continuously checked by monitoring system performance and analyzing quality control samples on an ongoing basis.

17.2.1 Requirements set forth in the sample preparation section for cleanups have to be met.

17.2.2 Limits for calibrations system performance have to be satisfied.

17.2.3 Q. C. requirements have to be met for all surrogate recoveries, spike recoveries for targeted compounds, and blanks have to be monitored.

17.2.4 Minimum detection limits (MDL) are determined on a yearly basis and have to satisfy the requirements for sensitivity of the method in respect to reporting requirements.

18.0 POLLUTION PREVENTION

18.1 Use pollution prevention techniques in laboratory operation whenever feasible.

18.2 Control releases from hood and bench operations and comply with discharge permits and regulations, hazardous waste identification rules, and land disposal restrictions.

18.3 The method is not a performance based method, and changes in regard to sample size, choice of solvent, etc. cannot be made to minimize the generation of waste.

18.4 The method allows the use of the Accelerated Solvent Extraction. This method offers a procedure with reduced sample weight and reduced amount of solvent consumption. This method should therefore be employed, if possible.

18.5 The generated waste has to be disposed in a manner not to cause pollution.

18.6 All wastes deemed toxic are collected in the laboratory and are disposed by professional companies licensed in waste disposal.

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

Before samples can be reported, the analytical data and quality control data have to be examined to assure that quality objectives are met.

19.1 Evaluation of Chromatographic Data

19.1.1 Check the chromatography of the analyses for baseline, resolution and interferences. (Refer to section 13)

19.1.2 Examine the surrogate compound retention times of all runs for retention time shifts.

19.2 Assessment of Calibration, Identification and Quantification

19.2.1 Check that the correct response factors were used for quantification of positives, i.e. the RFs of the current analytical sequence.

19.2.2 The compound has to elute in the expected retention time window.

19.2.3 Eliminate positive hits where the peak signal is under the practical detection limit (PDL).

19.2.4 Examine peaks for interferences and manually reintegrate peaks with poor integration. For reporting in data packages, manual integrations have to be signed by the analyst and reasons explained. Codes may be used if a list of explanations is included in the package.

19.2.5 Determine whether sample concentration is in the calibration range.

19.3 Acceptance Criteria of Quality Control Data

19.3.1 Make sure that data quality objectives are met for the quality control sample analyses and sample preparation efficiency is adequate.

19.3.2 Compare the surrogate recoveries of all extracts with the acceptance criteria in the quality control section to assure that preparation efficiency is acceptable for all analyses.

19.3.3 Check spike recoveries of the quality control samples against acceptance criteria listed in the quality control section.

19.3.4 Examine blanks for contaminations. Levels of targeted analytes cannot exceed the acceptance limits in the quality control section.

19.4 Once it has been established that the quality objectives are met, the finalized data are entered into the LIMS software and reported as described in the Quality Assurance Manual.

20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL DATA

20.1 Performance of Analytical System

20.1.1 If the analytical system is out-of-control according to the requirements outlined in section 13 for calibration and system performance, the samples after the last acceptable performance check are un-reportable and must be reanalyzed after remedial action is taken.

20.1.2 The system has to be recalibrated and the samples are reanalyzed.

20.2 Corrective Action for Noncompliant Surrogate Recoveries and Quality Control Samples

20.2.1 If surrogate recoveries for samples are not met, check for error and reanalyze. Re-extract positive sample with recoveries outside of Q. C. limits and negative samples with recoveries below the acceptance limits. If the spike recoveries for spiked samples are both outside the Q. C. limits the deviation is assumed to be matrix related and the samples do not have to be reanalyzed.

20.2.2 If the acceptance criteria established in the quality control section for lab fortified blanks (LFB) and method blanks cannot be satisfied even after reanalysis, the entire batch has to be re-extracted or proceed as outlined below.

21.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

21.1 Out–of–Control Analytical System

21.1.1 If the system fails after the second analysis (after remedial action was taken), the poor system performance is matrix related and will reoccur if the samples are injected again.

21.1.2 These sample analyses will have to be reported in spite of the deviations, and the problem has to be discussed in the narrative. Data have to be qualified and deemed "suspect" for the parameters for which acceptance criteria could not be met.

- In a limited amount of cases, acceptable date may be reportable from two analyses, if some analytes are acceptable in one run and the others in the other run. As an example, a group of samples may not have met the acceptance criteria for a pesticide that withstands sulfuric acid cleanup, and the rerun of the samples after acid cleanup could yield acceptable calibration checks for these particular analytes. These selected analytes could then be reported from the analysis after cleanup.

21.2 Out-of-Control Quality Control Data

21.2.1 If samples have to be re-extracted due to non-compliant quality control data, proceed as follow:

21.2.1.1 Report the re-extracted sample batch.

21.2.1.2 In the event that the re-extraction was performed out of holding time, both sets of data have to be reported, and the reasons explained in the narrative.

21.2.2 Exceptions for re-extractions may be made for sample batches with LFB recoveries outside Q. C. limits. The following applies:

21.2.2.1 Samples that do not have positives for any of the analytes for which recoveries are outside the limits, do not have to be re-extracted, unless the recoveries were very poor. The same applies to samples with positives below the reporting limits because those are regarded estimated.

21.2.2.2 If positive data are reported for noncompliant LFBs, this has to be communicated to the data user. The data for these analytes are not acceptable for regulatory purposes, unless the recoveries were too high and the analytes were not found. Data are regarded estimated.

21.2.3 If samples were extracted with a method blank that contained targeted analytes above acceptance limits the following applies:

21.2.3.1 The samples can be reported, if no positives are found in the samples.

21.2.3.2 If a blank contains more than the acceptable level of a targeted analyte, samples may only be accepted, if the level of positives in the samples are not critical for the usability of the data, and reporting of the data is approved by the data user.

21.2.3.3 As a guideline, if samples are at or above 20 times the blank level, they can be regarded to be genuine.

21.2.3.4 Regulatory limits can also be used as a guideline for acceptance. If the blank concentration is at or below 5% of the regulatory limit, data can be accepted.

21.3 Any non-compliances have to be addressed in the narrative, if data packages are submitted.

22.0 WASTE MANAGEMENT

Refer to the Quality Assurance Manual for proper collection procedures of toxic materials and their storage until pickup by a commercial Waste Management Company.

23.0 REFERENCES

Reference 1 For 8081B: "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update IV, February 2007

Reference 2 "New York State Department of Environmental Protection Analytical Services Protocol," Update IVa, January 1998

Reference 3 "New York State Department of Environmental Protection Analytical Services Protocol," April 2005

Reference 4 "New York State Department of Environmental Protection Analytical Services Protocol," April 2010

Reference 5 National Environmental Laboratory Accreditation Conference (NELAC) Constitution, Bylaws and Standard, approved June 5, 2003, (EPA/600/R-04/003)

Reference 6 2009 TNI Laboratory Accreditation Standards, EL-V1-2009.

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TABLE 1

PRACTICAL QUANTIFICATION LIMITS (PQL)

		Quantitation Limits (a)		
Analyte	CAS Number	Water µg/L	Soil µg/Kg (b)	On Column (pg)/µL
Alpha-BHC	319-84-6	0.05	1.7	5
Beta-BHC	319-85-7	0.05	1.7	5
Delta-BHC	319-86-8	0.05	1.7	5
Gamma-BHC (Lindane)	58-89-9	0.05	1.7	5
Heptachlor	76-44-8	0.05	1.7	5
Aldrin	309-00-2	0.05	1.7	5
Heptachlor epoxide	1024-57-3	0.05	1.7	5
Endosulfan I	959-98-8	0.05	1.7	5
Dieldrin	60-57-1	0.10	3.3	10
4,4'-DDE	72-55-9	0.10	3.3	10
Endrin	72-20-8	0.10	3.3	10
Endosulfan II	33213-65-9	0.10	3.3	10
4,4'-DDD	72-54-8	0.10	3.3	10
Endosulfan sulfate	1031-07-8	0.10	3.3	10
4,4'-DDT	50-29-3	0.10	3.3	10
Methoxychlor	72-43-5	0.50	17.0	50
Endrin ketone	53494-70-5	0.10	3.3	10
Endrin aldehyde	7421-36-3	0.10	3.3	10
Alpha-Chlordane	5103-71-9	0.05	1.7	5
Gamma-Chlordane	5103-74-2	0.05	1.7	5
Toxaphene	8001-35-2	5.0	170.0	500
Mirex	2385-85-5	0.1	3.3	10

(a) The quantification limits are matrix dependent.
(b) Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits for soil/sediment samples, are calculated on dry weight basis, and will be higher.

TABLE 2

GC OPERATING CONDITIONS FOR CHLORINATED PESTICIDES

(Recommended program; modify as needed to optimize)

Instrument:	HP6890
Column:	Column (A) RTX-CLP, fused silica 30 m, 0.32 mm ID or equivalent
Column (B):	RTX-CLP2, fused silica 30 m, 0.32mm ID or equivalent
Temperature Program:	Initial temperature 120°C; no hold ,
	Ramp I: 50°C/min to 200°C; no hold
	Ramp II: 7.0°C/min to 230°C; no hold
	Ramp III: 30°C/min to 300°C
	Final hold: 3 min
Total Run Time:	11 minutes
Injection Volume:	0.5 - 2 μL per column
Inlet Temperature:	250°C
Carrier Gas:	4 mL/min helium or hydrogen (each column)
Detector:	ECD
Detector Range:	------
Detector Temperature:	300°C
Detector Gas:	60 mL/min nitrogen
Processing Software:	Perkin Elmer TotalChrom 6.3, Omega from Khemia, or equivalent

TABLE 3

RETENTION TIME WINDOW VARIANCES (a)

Analyte	Variances (Minutes)
Alpha-BHC	0.05
Beta-BHC	0.05
Gamma-BHC	0.05
Delta-BHC	0.05
Heptachlor	0.05
Aldrin	0.05
Alpha-Chlordane	0.07
Gamma-Chlordane	0.07
Heptachlor epoxide	0.07
Dieldrin	0.07
Endrin	0.07
Endrin aldehyde	0.07
Endrin ketone	0.07
4,4'-DDD	0.07
4,4'-DDE	0.07
4,4'-DDT	0.07
Endosulfan I	0.07
Endosulfan II	0.07
Endosulfan Sulfate	0.07
Methoxychlor	0.07
Toxaphene	0.07
Mirex	0.03
TCMX	0.05
DCB	0.10

(a) These are CLP variances, which can be used based on the discretion of the analyst. (They may be used for clean samples, but for samples with more complex matrices, in-house variances should be determined with retention time studies.)

TABLE 4

MAJOR IONS FOR GC/MS CONFIRMATION

Analyte	Primary Ion	Secondary Ion(s)
a-BHC	183	181, 109
b-BHC	181	183, 109
d-BHC	183	181, 109
g-BHC	183	181, 109
Heptachlor	100	272, 274
Aldrin	66	263, 220
Heptachlor Epoxide	353	355, 351
Endosulfan I	195	339, 341
Dieldrin	79	263, 279
4,4'-DDE	246	248,176
Endrin	263	82, 81
Endrin Ketone	317	67, 319
Endrin Aldehyde	67	250, 345
Endosulfan II	337	339, 341
4,4'-DDD	235	237, 165
Endosulfan sulfate	272	387, 422
4,4'-DDT	235	237,165
Methoxychlor	227	228
a/g-Chlordane	373	375, 377
Toxaphene	159	231, 233
Mirex	272	237,274,270,239,235

TABLE 5

Q. C. LIMITS Surrogate Recovery Limits

Surrogate	Water - % Recovery	Soil - % Recovery
TCX	30 - 150	30 - 150
DCB	30 - 150	30 - 150

Q. C. Limits for Matrix Spikes Recoveries (a)

Analyte	WATER		SOIL	
	% Recovery	RPD	% Recovery	RPD
g-BHC	56 -123	15	35-135	50
Heptachlor	40-131	20	40-131	31
Aldrin	40-120	22	34 - 132	43
Dieldrin	52 - 126	18	31 - 134	38
Endrin	56 - 121	21	42 - 139	45
4,4'-DDT	38 - 127	27	23 - 134	50

(a) Limits are advisory for MS/MSD but mandatory for matrix spike blank (LCS)

TABLE 6

Q. C. LIMITS FOR LAB FORTIFIED BLANKS (a)

(Typical limits. Updated limits can be found in the Omega Data System.)

Analyte	Water-% Recovery	Soil -% Recovery
4,4´-DDD	51-138	54-132
4,4´-DDE	56-131	52-137
4,4´-DDT	56-130	55-130
Aldrin	30-147	35-147
alpha-BHC	59-127	35-146
alpha-chlordane	50-133	54-127
beta-BHC	62-122	54-131
delta-BHC	48-137	53-138
Dieldrin	56-131	47-134
Endosulfan I	51-130	28-150
Endosulfan II	52-143	61-135
Endosulfan sulfate	58-134	66-132
Endrin	68-140	57-148
Endrin aldehyde	23-163	61-136
Endrin ketone	61-141	72-135
gamma-BHC	59-128	37-146
gamma-chlordane	53-134	44-147
Heptachlor	44-136	44-139
Heptachlor epoxide	58-125	41-140
Methoxychlor	59-151	61-138
Toxaphene	58-153	50-150
Chlordane	45-167	50-150
Mirex	50-150	50-150

(a) Mandatory Limits

TABLE 7

METHOD DETECTION LIMITS FOR WATER

(Representative limits, updated limits on file)

ug/L

	MDL1	MDL2	MDL3	MDL4	MDL5	MDL6	MDL7	AVG	S	MDL
ALPHA-BHC	0.0411	0.04	0.042	0.039	0.039	0.045	0.04	0.041	0.0022	0.0069
GAMMA-BHC	0.0449	0.042	0.044	0.042	0.042	0.048	0.042	0.043	0.0022	0.0070
BETA-BHC	0.0618	0.046	0.048	0.041	0.045	0.051	0.046	0.048	0.0066	0.0208
DELTA-BHC	0.0538	0.041	0.043	0.04	0.042	0.047	0.042	0.044	0.0048	0.0150
HEPTACHLOR	0.0441	0.042	0.045	0.042	0.042	0.048	0.042	0.043	0.0023	0.0073
ALDRIN	0.0514	0.039	0.041	0.038	0.039	0.044	0.039	0.042	0.0047	0.0148
ISODRIN	0.0524	0.048	0.052	0.046	0.047	0.053	0.059	0.051	0.0046	0.0145
HEPT. EPOXIDE	0.0542	0.042	0.043	0.04	0.041	0.047	0.041	0.044	0.0049	0.0155
G. CHLORDANE	0.0613	0.049	0.052	0.048	0.049	0.055	0.049	0.052	0.0048	0.0149
A. CHLORDANE	0.0466	0.042	0.048	0.048	0.045	0.048	0.041	0.046	0.0029	0.0091
ENDOSULFAN I	0.0507	0.051	0.049	0.042	0.047	0.055	0.049	0.049	0.0040	0.0125
4,4'-DDE	0.0499	0.052	0.061	0.053	0.049	0.058	0.081	0.057	0.0111	0.0348
DIELDRIN	0.0456	0.047	0.051	0.045	0.044	0.053	0.055	0.049	0.0043	0.0136
ENDRIN	0.0658	0.063	0.068	0.063	0.066	0.07	0.063	0.065	0.0028	0.0088
4,4'-DDD	0.0596	0.058	0.06	0.056	0.057	0.065	0.063	0.060	0.0032	0.0102
ENDOSULFAN II	0.0519	0.052	0.055	0.05	0.05	0.058	0.052	0.053	0.0027	0.0085
4,4'-DDT	0.0583	0.057	0.059	0.055	0.056	0.066	0.056	0.058	0.0036	0.0114
ENDRIN ALDEHYDE	0.0634	0.058	0.063	0.056	0.058	0.071	0.058	0.061	0.0053	0.0166
METHOXYCHLOR	0.0653	0.064	0.071	0.062	0.068	0.073	0.065	0.067	0.0036	0.0115
ENDO. SULFATE	0.0619	0.06	0.061	0.057	0.059	0.066	0.058	0.061	0.0031	0.0096
ENDRIN KETONE	0.0664	0.063	0.07	0.063	0.064	0.072	0.064	0.066	0.0037	0.0117
MIREX	0.113	0.103	0.12	0.104	0.115	0.125	0.113	0.116	0.0079	0.0249

TABLE 8

METHOD DETECTION LIMITS FOR SOIL

(Representative limits, updated limits on file)

ug/kg

	MDL1	MDL2	MDL3	MDL4	MDL5	MDL6	MDL7	AVG	S	MDL
ALPHA-BHC	1.30	1.59	1.39	1.38	1.42	1.26	1.35	1.38	0.106	0.33
GAMMA-BHC	1.51	1.56	1.50	1.53	1.47	1.39	1.52	1.50	0.055	0.17
BETA-BHC	1.53	1.85	1.67	1.55	1.54	1.46	1.65	1.61	0.129	0.41
DELTA-BHC	1.36	1.41	1.37	1.25	1.31	1.35	1.33	1.34	0.051	0.16
HEPTACHLOR	1.44	1.47	1.46	1.40	1.44	1.27	1.45	1.42	0.069	0.22
ALDRIN	1.35	1.37	1.36	1.39	1.39	1.30	1.36	1.36	0.031	0.10
ISODRIN	1.72	1.76	1.65	1.73	1.71	1.64	1.75	1.71	0.047	0.15
HEPT. EPOXIDE	1.36	1.41	1.39	1.39	1.40	1.35	1.41	1.39	0.024	0.07
G. CHLORDANE	1.64	1.67	1.67	1.68	1.68	1.61	1.68	1.66	0.027	0.08
A. CHLORDANE	1.62	1.52	1.63	1.49	1.50	1.68	1.64	1.58	0.077	0.24
ENDOSULFAN I	1.47	1.64	1.54	1.65	1.68	1.31	1.56	1.55	0.129	0.40
4,4'-DDE	1.95	2.00	1.88	2.02	1.99	1.54	2.07	1.92	0.178	0.56
DIELDRIN	1.49	1.58	1.50	1.61	1.55	1.45	1.54	1.53	0.055	0.17
ENDRIN	2.02	2.04	2.07	2.05	2.07	2.01	2.09	2.05	0.029	0.09
4,4'-DDD	1.90	1.98	1.91	1.98	1.99	1.93	1.94	1.95	0.036	0.11
ENDOSULFAN II	1.73	1.86	1.81	1.82	1.88	1.78	1.78	1.81	0.051	0.16
4,4'-DDT	1.77	1.92	1.80	1.90	1.93	1.82	1.86	1.86	0.062	0.20
ENDRIN ALDEHYDE	1.87	1.92	2.32	2.29	2.36	2.32	2.29	2.20	0.207	0.65
METHOXYCHLOR	1.73	1.87	1.79	2.00	1.82	1.75	1.87	1.83	0.091	0.29
ENDO. SULFATE	1.90	1.96	1.93	1.99	1.98	1.90	1.98	1.95	0.038	0.12
ENDRIN KETONE	1.98	2.08	2.01	2.08	2.09	1.96	2.04	2.03	0.052	0.16
MIREX	1.48	1.52	1.52	1.59	1.53	1.45	1.25	1.476	0.1098	0.3448

APPENDIX P3-4
SOP FOR THE EXTRACTION OF AQUEOUS SAMPLES BY SOLID PHASE EXTRACTION BY SW-846 METHOD 3535A FOR PCB ANALYSIS
(S-NY-O-218-REV.08)

STANDARD OPERATING PROCEDURE

EXTRACTION OF AQUEOUS SAMPLES BY SOLID PHASE EXTRACTION (SPE)

Reference Methods: EPA Method 3535A

SOP Number:	S-NY-O-218-rev.08
Effective Date:	03/30/15
Supersedes:	S-NY-O-218-rev.07, S-NY-0178-rev.07

APPROVALS

[signature]	03/30/15
Assistant General Manager	Date
[signature]	03/30/15
Quality Manager	Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

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1. Purpose/Identification of Method

1.1. This is a Standard Operating Procedure (SOP) for the extraction of aqueous samples by Solid Phase Extraction (SPE) using US-EPA SW-846 Method 3535A.

2. Summary of Method

2.1. Prepare Sample: Warm to room temperature, spike and surrogate, check pH, acidify, foil, and cap.

2.2. Extractor Preparation: Turn on extractor, check gauges, check solvent and recovery bottles, purge.

2.3. Extract sample.

2.4. Solvent exchange to hexane and set to volume.

2.5. PCB analysis: The extract may be put through cleanup processes (see separate SOPs) and is then properly diluted and submitted for GC analysis.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in SPE extraction by method 3535A.

3.2. The following procedure is utilized by Pace Analytical Services, Inc. for the extraction of PCBs and Pesticides from aqueous samples using the solid phase extraction method. This method was developed to utilize an automated extraction system, which is the SPE-DEX® 4790 extractor from Horizon Technologies. The extraction disk that will be used is a 50mm Bakerbond Speedisk™ styrene divinyl benzene filter

3.3. **Parameters**: See SOPs for determinative methods for analyte lists.

4. Applicable Matrices

4.1. This method is applicable to surface water, groundwater, wastewater, and other aqueous samples.

5. Limits of Detection and Quantitation

5.1. Please consult determinative methods (or latest versions of SOPs for EPA Method 8082A, S-NY-O-314; EPA 8081B, S-NY-O-131; CSGB, S-NY-O-294; and CQCS, S-NY-O-133) for detection limits.

6. Interferences

6.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing equipment (such as the SPE-DEX 4790 extractor) that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials and equipment must be routinely verified to be free from interferences by running laboratory method blanks.

6.2. All equipment must be kept scrupulously clean. Clean all equipment as soon as possible after use, and store in jars away from environmental contaminants.

6.3. The use of high purity solvent and reagents will minimize interference problems. Purification of reagents by washing with solvent will also help to reduce interference problems.

6.4. Laboratory contamination can occur by introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing, gloves, pipette bulbs, etc. Samples and extracts should not be exposed to plastic materials. Phthalate esters exhibit a response on electron capture detectors, usually as late eluting peaks and can interfere in PCB quantification.

6.5. The sample matrix itself is also a potential source for method analyte interference. The clean-up procedures provided in this SOP can be used to overcome many of these interferences.

7. Sample Collection, Preservation, Shipment and Storage

7.1. The samples should be collected in unpreserved 1 liter glass bottles with a Teflon lined cap.

7.2. All samples must be placed on ice or refrigerated at 0-6°C from the time they are collected until delivery to the lab.

7.3. The samples must be protected from light and refrigerated at 0-6°C from time of receipt until they are removed from storage for extraction. Typically the entire water sample grab will be consumed at time of extraction and no sample is left for long-term storage.

7.4. The sample extraction hold time for Pesticides by EPA Method 8081B is seven days from the date the samples were collected. For 8082A, CSGB, and CQCS analyses, the hold time is up to one year from date of collection.

7.5. Sample extracts must be protected from light and stored refrigerated at 0-6°C. The hold time for sample extracts is 40 days. After analysis is complete, sample extracts will be discarded after 60 days or can be archived in a freezer at less than -20°C for longer periods of time depending on the program requirements.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. **Surrogate Standard Solution:** In chemical composition and chromatography similar to the analytes of interest. Usually not found in environmental samples. These compounds are spiked into all samples, blanks, and matrix spike samples prior to analysis. Percent recoveries are calculated for each surrogate.

8.3. **Matrix Spike Solution:** An aliquot of sample fortified with a known quantity of the analytes of interest, used to indicate the accuracy of the method by measuring the recovery.

8.4. **Laboratory Method Blank:** A laboratory derived sample consisting of reagent water or other blank matrix that consists of all reagents, internal standards, and surrogate standards that is carried through the entire analytical procedure. The laboratory method blank is used to define the level of laboratory analyte background or other interferences that exist in the laboratory environment, the reagents or the apparatus.

8.5. **Laboratory Control Sample (LCS):** Also known as the Quality Control (QC) Check Standard or Quality Control (QC) Check Sample. The LCS consists of an aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added. The LCS is extracted and analyzed exactly like a field sample and its purpose is to determine whether the analysis is in control and whether the laboratory is capable of making accurate and precise measurements.

8.6. **Matrix Spike/Sample Matrix Spike Duplicate (MS/MSD):** An aliquot of a field sample that is fortified with known quantities of the method analytes and subject to the entire analytical procedure. Its purpose is to assess the appropriateness of the method for the matrix by measuring the recovery.

8.7. **DI Water (De-ionized Water):** Water free from analytes that may interfere with the analytical test compounds.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Automated Solid Phase Extractor: Horizon# 4790-08. Consisting of:

9.1.1. Envision Platform;

9.1.2. SPE-DEX® 4790 Extractor;

9.1.3. 10” Solvent Vapor Exhaust Hose (2” Diameter);

9.1.4. Water and Solvent Waste Vacuum Lines;

9.1.5. Vacuum Supply Line Assembly;

9.1.6. 8’ Telco Communication Cable;

9.1.7. Solvent Delivery Line Kit;

9.1.8. Pressure Regulator Bracket Assembly;

9.1.9. Solvent Delivery Bottle Plug Kit;

9.1.10. Solvent Recovery Manifold Assembly;

9.1.11. Water Recovery Manifold Assembly;

9.1.12. Waste Solvent Recovery Bottle;

9.1.13. Waste Water Recovery Bottle;

9.1.14. Solvent Delivery Teflon Manifold Kit;

9.1.15. 2.5 Liter Safety Coated Solvent Bottles;

9.1.16. Vacuum Source – capable of 20 to 25” Hg;

9.1.17. Dry Trap for the Vacuum Source;

9.1.18. Dry Nitrogen Gas Supply – capable of minimum 60 PSI to maximum 80 PSI.

9.2. Glass Sample Containers: 1 liter amber glass bottle. The 1-liter sample bottles are available pre-cleaned and certified. The lab has an in-house verification procedure to test bottles at a rate of 1 per vendor lot number.

9.3. Bakerbond Speedisk™ DVB**:** Styrene Divinyl Benzene 50mm disk for sample extraction P/N 8059-06 or equivalent.

9.4. Disk Adapter.

9.5. Vials: glass, 40mL, 60mL, and 4 dram (with polyseal cap), for sample extracts (or equivalent.

9.6. Collection Vessel Adapter: 19/22 Taper.

9.7. Bottle Adapters: 38 x 400 adapters 33 × 430 adapter for 1 L amber glass bottle

9.8. Aluminum Foil Squares: purchased pre-cleaned.

9.9. pH indicator Strips: EM Science P/N 9590 or equivalent.

9.10. Pipettes**:** S/P Disposable Serological Borosilicate Pipettes (or equivalent):

9.10.1. 1mL x 1/100, Kimble #72120-1100;

9.10.2. 5mL x 1/10, Kimble #72120-5110;

9.10.3. 10mL x 1/10, Kimble #72120-10110;

9.10.4. Pasteur Glass Pasteur Pipettes: 9'', Krackeler-Brand #67-450-900.

9.11. Syringes:

9.11.1. 500µL Syringe, gas-tight, Hamilton #81217;

9.11.2. 1000µL Syringe, gas-tight, Hamilton #81317;

9.11.3. 250µL Syringe, gas-tight, Hamilton #81100.

9.12. Vial Rack: plastic rack used to hold vials during processing of extracts. Scienceware.

9.13. Beakers: assorted Pyrex 250mL, 600mL and 1000mL, used for liquid containment and pipette storage.

9.14. Graduated Cylinder: 2L capacity (Scienceware) and 4L capacity (Nalgene).

9.15. TurboVap LV Evaporator: Caliper.

9.16. Centrifuge: International Equipment Co., Model CL (or equivalent).

9.17. Wrist Shaker: Burrell wrist action shaker, Model 75 and 88 (or equivalent).

9.18. Flask: Chemglass, 125mL Erlenmeyer filter flask for purge recovery.

9.19. Keck Clip: to hold flask.

10. Reagents and Standards

10.1. 1:1 Sulfuric Acid: (H_2SO_4), J.T. Baker #9675-03, or equivalent. Preparation: Set up a small bucket with cold water and ice. Place a 1L beaker containing 500mL of DI water into the bucket. Slowly add 500mL concentrated H_2SO_4. Allow the mixture to cool after preparation. Then, transfer to a pre-cleaned 1L bottle for storage.

10.2. DI Water: (Reagent Water) 18 Megaohm water obtained from the laboratory's water purification system. Used for solid phase disk preparation, laboratory method blanks, laboratory control samples, MDL studies, and Demonstration of Capability studies.

10.3. Methanol: Pesticide residue quality. EM Science OmniSolv. P/N MX0488P-1 or equivalent.

10.4. Acetone: High purity solvent; (Burdick\Jackson) UN1090 or equivalent.

10.5. Hexane: High purity solvent; (Burdick\Jackson) UN1208 or equivalent.

10.6. Dichloromethane: High purity solvent; (Burdick\Jackson) UN1593 or equivalent.

10.7. Sodium Hydroxide: JT Baker, #5671-03 or equivalent.

10.8. Surrogate Standard (0.05ug/mL TCMX / 0.5ug/mL DCBP or 2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl at 0.2ug/mL): To every water sample and QC sample, surrogate standard is added before extraction is initiated. The surrogate standard must be replaced after six months.

10.8.1. To make 0.05ug/mL TCMX/0.5ug/mL DCBP: An ampule of Custom Standard CUS-4911 (Ultra Scientific) is brought to room temperature and shaken on a wrist action shaker for at least 30 minutes. Once the standard is room temperature 100uL is added to 1L of acetone for 8082A analysis, or methanol for 8081B analysis. All information is recorded in the standards logbook.

10.8.2. To make Nonachlorobiphenyl at 0.2ug/mL in acetone: Allow a stock standard of Nonachlorobiphenyl at 100ug/mL warm to room temperature. Take 0.2mL of this stock standard and add it to a 100mL volumetric flask. Fill to the meniscus with acetone. All information is recorded in the Standards logbook.

10.9. Aroclor Spiking Standard at 0.5ug/mL or 1.0ug/mL in Acetone: For 8082A analysis only. To every laboratory control spike, matrix spike, and matrix spike duplicate, add Aroclor spiking standard before extraction is initiated. The spiking standard must be replaced after six months.

10.9.1. To make an Aroclor at 0.5ug/mL spike: Allow a Calibration Stock Standard at 10.0ug/mL to warm up to room temperature. Using a class A volumetric pipette, pipette 5.0mL into a 100mL volumetric flask. Fill to 100mL with acetone. All information is recorded in the Standards logbook.

10.9.2. To make an Aroclor at 1.0ug/mL spike: Allow an Aroclor standard at 100ug/mL warm up to room temperature. Using a class A volumetric pipette, or a gastight syringe, add 1.0mL of the standard at 100ug/mL to a 100mL volumetric flask. Fill to 100mL with acetone. All information is recorded in the Standards logbook.

10.10. Mixed Pesticide with Hexachlorobenzene and Mirex at 1.0ug/mL in Methanol: For 8081B analysis only. To every laboratory control spike, matrix spike, and matrix spike duplicate, add spiking standard before extraction is initiated. The spiking standard must be replaced after six months.

10.10.1. To make Mixed Pesticide with HCB and Mirex at 1.0ug/mL: Follow the latest version of SOP S-NY-O-131 with the following change- use methanol as the solvent instead of hexane.

11. Calibration and Standardization

11.1. Please consult determinative methods (or latest versions of SOPs for EPA Method 8082A, S-NY-O-314 or EPA 8081B, S-NY-O-131, CSGB, S-NY-O-294, and CQCS, S-NY-O-133) for calibration information.

12. Procedure

12.1. **Sample Preparation** (**Note:** When rinsing or pre-rinsing is referred to in this SOP, it is performed using a pipette and beaker):

12.1.1. Throughout the entire process it should be noted that if the extraction technician encounters any problems or difficulties with any samples or steps involved, all work should **STOP!!!** Any problems should be brought to the attention of the supervisor and documented in LIMS.

12.1.2. Remove the samples for extraction from cold storage and allow them to warm up to room temperature (at least 30 minutes for a 1L sample). This can be accomplished first while the extraction equipment is prepared to extract samples.

12.1.3. Before any steps are taken, the extraction technician should first review the sample job folder and check the sample labels versus the original chain of custody. Any discrepancies should be brought to the attention of the supervisor and the sample login custodian.

12.1.4. Using 1L amber bottles prepare a method blank and laboratory control spike sample using reagent water equal to the sampling volume.

12.1.5. Mark the level of the sample on the outside of the container with a paint pen.

12.1.6. To the laboratory control sample, matrix spike, and matrix spike duplicate samples, add the matrix spike standard solution using a gas-tight syringe; see section 13.2 for spiking amounts. Then add the

surrogate standard solution to every sample and QC sample, also using a gas-tight syringe; see section 13.3 for surrogate amounts. This process must be done with a witness, and documented in LIMS.

12.1.7. Note: For those samples which require a 2L extraction volume, half the spike standard solution and surrogate standard solution are added to each bottle associated with the same sample.

12.1.8. Determine the pH of the sample by removing a small amount of sample with a Pasteur pipette (approximately 0.1mL) and wet a pH indicator strip.

12.1.9. Add 1.0mL of 1:1 sulfuric acid per liter of water to every sample and QC sample using a disposable pipette. Cap and invert the sample container several times to mix. Check the pH as in Section 12.1.7 to make sure it has been adjusted to a pH 2 or less. If not, add 0.5mL more of 1:1 sulfuric acid and test again. Continue to add 1:1 sulfuric acid in 0.5mL aliquots until the pH is 2 or less.

12.1.10. Cap and invert each sample and QC sample several times to mix.

12.2. **Extractor Preparation:**

12.2.1. Check solvent reservoir bottles, solvent recovery bottle and waste water recovery bottle. Fill solvent reservoirs if necessary. When filling the solvent reservoirs, care should be taken not to spill or drip solvent on the outside of the bottles as they are rubber coated, and some solvents may dissolve the rubber. Solvent reservoirs should never be filled past their rubber coating. The DI water reservoir should be emptied, rinsed with RO water, and re-filled weekly.

12.2.2. Empty the solvent recovery bottle into an approved waste container for proper disposal according to the latest version of the Waste Handling and Management SOP, S-NY-W-054.

12.2.3. If necessary, neutralize and empty the wastewater recovery jugs. Add sodium hydroxide until the pH reads between 5 and 9. Empty the jugs by turning the valve on the connected tubing which is connected to the drain to the open position.

12.2.4. Turn the vacuum pump on and adjust the main vacuum between 20" and 26" Hg. Verify the water recovery manifold assemblies on the waste water jugs are forming a good seal.

12.2.5. Turn on the pressurized gas source and adjust between 60 and 80 psi.

12.2.6. Turn on the controller.

12.2.7. Adjust the non-venting vacuum regulator attached to the solvent recovery manifold assembly on the solvent recovery bottle to no greater than 15" Hg. **NOTE:** DO NOT adjust the regulator greater than 15" Hg. Higher vacuum levels could cause an SPE disk to go dry during the methanol and reagent water pre-wet steps.

12.2.8. On the pressure bracket assembly, adjust the left regulator labeled SPE extractor pressure between 45 and 50 psi. Adjust the right regulator labeled solvent bottle pressure to approximately 18 psi.

12.2.9. Attach the disk adapter to a Bakerbond Speedisk™ designated for purges. Then place this set up on the disk holder platform.

12.2.10. Attach a 125mL flask designated for purges to the tapered joint on the bottom of the platform. Secure with a keck clip.

12.2.11. Invert a purge bottle and place into bottle holder of the extractor unit. (Note: A purge bottle is prepared by attaching a cap adapter to an empty 1L glass bottle. Since purge bottles can be reused, a set should be prepared and stored for reuse).

12.2.12. On the computer screen choose the icon corresponding to the Envision Platform controllers (Northeast Right Bank SPE or Northeast Left Bank SPE). The Operations Screen should load. The Operations screen is used to control and monitor the status of each Extractor. The screen is divided into

sections within the right and left pane. The right pane displays the status of all connected extractors; the left pane is used to control and monitor one extractor. The extractors now must be programmed for a purge.

12.2.13. On the left pane of the Operations Screen there will be "**Purge Methods**" with a drop-down menu beneath it. From the drop-down list select **NEA Purge**. Two options will begin to blink green: **Load** and **Load All**. The **Load All** option will load the method into all 8 extractors. The **Load** option will load the method into only the extractor displayed on the right. Since all 8 units will need to be purged prior to use, select the **Load All** option.

12.2.14. **Purge** and **Purge All** will begin to blink green. Choose **Purge All** to purge all 8 units at once, or choose **Purge** to purge only the unit displayed in the right pane. This will initiate the purge cycle sequence. The purge may also be started for each individual unit by pressing the **Purge** button on each unit. The purge cycle will clean the extractor unit by dispensing the pre-wet solvents and directing them to the solvent recovery bottle. Next the rinse solvents will be sprayed into the empty sample bottle and directed into the collection vessel. **NOTE:** Always use an empty bottle when running a purge cycle.

12.2.15. During the purge cycle, carefully observe the operation of the SPE-DEX® 4790 extractor unit. Make sure that it is functioning properly and that the check valve located in the disk holder assembly is operating correctly and solvents are being delivered to the correct locations.

12.2.16. Once the purge cycle is completed, the SPE-DEX® 4790 extractor unit is ready to process samples. **NOTE:** A purge cycle is required to be analyzed before each day, between sample extractions and before shutting down at the end of the day.

12.2.17. Detach the flask, and empty it into a properly labeled waste container.

12.2.18. Remove the purge bottle from the bottle holder.

12.2.19. Remove the Bakerbond Speedisk™ from the disk holder platform. Save the disk, since it may be reused for future purge cycles.

12.3. **Sample Extraction:**

12.3.1. Install a new Bakerbond Speedisk™ DVB onto the disk holder platform with the disk adapter.

12.3.2. Attach a vial adapter to the tapered joint on the bottom of the disk holder platform and place the keck clip on the adapter to secure it to the tapered joint. Attach a hexane pre-rinsed and properly labeled 40mL vial to the vial adapter.

12.3.3. Remove the bottle cap from the first sample. Place a 2" × 2" piece of pre-cleaned aluminum foil over the mouth of the bottle. Gently screw the amber bottle adapter (33 × 430) over the aluminum foil onto the screw threads of the bottle. When completed properly the aluminum foil will be taut and no rips will be evident. If a tear is detected, the adapter must be removed and a new piece of aluminum foil must be installed.

12.3.4. Invert the sample bottle and inspect for any leaks or rising bubbles from the seal of the bottle and the adapter. If no bubbles or leaks are detected, then a good seal has been made and the sample bottle can be installed onto the bottle holder assembly. If a leak or bubbles are observed, the adapter may need to be tightened or a new piece of aluminum foil may need to be installed. Test again until a good seal is formed.

12.3.5. With the bottle inverted, place the bottle into the bottle holder assembly ensuring that the solvent rinse stem is inside the bottle adapter. Gently lower and then firmly push the sample bottle into the bottle holder assembly. A bubble should escape into the bottle. Turn the bottle clockwise until a large bubble

floats up into the bottle. (**NOTE:** Do not turn more than three quarters of the way around). This breaks an opening in the aluminum foil and allows the sample to flow freely.

12.3.6. Once the sample bottle has been installed, the Envision Platform controller will need to be programmed with a sample method. To the left of the "**Purge Methods**" on the left pane of the Operations Screen there will be "**Current Method**" with another drop-down menu. Select **First Bottle** for PCB analysis. Select **Pesticide 8081** for Pesticide analysis.

12.3.7. The **Load**, **Load All** options will blink green. Just like the purge cycles, the **Load All** option will load the method into all 8 extractors and the **Load** option will load the method into only the extractor displayed on the left pane. Choose the **Load All** option.

12.3.8. Again the options **Start** and **Start All** blink green on the Operations Screen. Choose to either start all units at once by clicking **Start All** or each unit individually by choosing an extractor so that it is displayed in the left pane and then clicking **Start**. Each unit can also be individually started by pressing the **Start** button on the unit. The "Purge Method" and the "Current Method" will remain programmed in unless they are changed.

12.3.9. The SPE-DEX® 4790 extractor will automatically pre-wet the solid phase disk, extract the sample, air dry the disk post extraction, and extract the disk to recover the analytes of interest. See Attachment II. Each extractor will flash a green light while running: one blink for pre-wet, two for sample extraction (sample drop), three for wash (we don't use wash), four for air dry, five for rinse or disk extraction. The Envision Platform will also display the current status of each extractor unit.

12.3.10. Note: For those samples which require a 2L extraction follow steps 12.3.9 through 12.3.15 in SOP S-NY-O-208 for the second bottle extraction method.

12.3.11. Once the extraction process is completed the 40mL vial is removed from the vial adapter, capped with a pre-rinsed 40mL polyseal cap, and then the vial is placed in a rack. The sample is then ready to be stored in cold storage or taken through the required cleanup steps. The vial adapter is removed and placed in the hood to be cleaned.

12.3.12. Remove the sample bottle from the bottle holder assembly. Remove the cap adapter and place in the hood for cleaning.

12.3.13. Remove the Bakerbond Speedisk™ and disk adapter from the disk holder platform. Throw the used Bakerbond Speedisk™ in the garbage, and place the disk adapter in the hood to be cleaned.

12.3.14. Fill the sample bottle(s) with tap water to the mark made before the extraction. Measure the volume of the water using a 2L graduated cylinder to the nearest 10mL. Record the volume in LIMS.

12.3.15. Re-purge the units that were used as in Section 12.2. Shut down the system in the reverse order as startup; starting with the computer browser, controller, nitrogen source, and pump. Break the seal on the water recovery jug.

12.3.16. Clean all used equipment in the hood with acetone and then hexane. Leave in the hood to dry before putting away in the designated containers.

12.4. **Sample Extract Concentration:**

12.4.1. **Extract Solvent Reduction:**

12.4.1.1. The sample extract will have two layers, the top layer will be composed of the hexane/acetone used to elute components from the solid phase disk and the bottom layer will be composed of residual water and acetone from the extraction process. Carefully transfer the top layer of solvent to a hexane pre-rinsed and properly labeled 60mL vial using a hexane pre-rinsed disposable 10mL pipette.

12.4.1.2. Backwash the residual water/acetone in the 40mL with three Pasteur pipette volumes of hexane. Shake by hand in a chemical fume hood for 5 to 10 seconds. Allow to settle, forming two layers similar to the original sample. Transfer top hexane layer carefully into the vessel holding the rest of the extract. Repeat this two more times, for a total of three rinses.

12.4.1.3. After all the rinses have been transferred, rinse the outside of the 10mL pipette with hexane into the sample vial. Rinse the sides of the vial, and bring the volume of all the extracts up to just below the base of the vial neck.

12.4.1.4. Dump any remaining residual water from the water/acetone layer into a waste jar for evaporation in a hood. This jar will be saved for reuse.

12.4.1.5. The LV Turbo Vap Evaporator systems are used to reduce the sample volume. They use a heated water bath and positive pressure Nitrogen flow with vortex action. The units maintain a slight equilibrium imbalance between the liquid and the gaseous phases of the solvent extract, which allows for fractional reduction of the solvent without loss of higher boiling point analytes.

12.4.1.6. Turn on the LV Turbo Vap Evaporator and allow it to heat up to 40°C ± 2°C. Check the water level in the LV Turbo Vap and fill, if necessary, to the second stage from the top. On the top of the unit press the **START/PAUSE** key, a green light will light up to the right of it. Then press the Tube station keys corresponding to the rows needed to concentrate the samples.

12.4.1.7. As a precaution the Turbo Vap Evaporator system regulators should be checked to assure that there is no residual gas pressure within the system and that the gas pressure regulator is off before placing samples in the apparatus. Residual gas pressure may cause splashing and therefore cause cross-contamination of samples. Verify that the lid and regulator are closed, and that the cells or rows are turned on. Bleed any residual gas until the regulator gauge exhibits "0" psi.

12.4.1.8. Wipe down the inside of the LV Turbo Vap Evaporator with a hexane wetted paper towel including the top lid and pins. Close the lid and turn on the regulator to dry the LV Turbo Vap. Turn off the gas regulator before loading samples. Place the 60mL vials containing the sample extract into the LV Turbo Vap and close the lid.

12.4.1.9. Slowly open the pressure regulator, by turning it towards yourself. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the volume reduction.

12.4.1.10. The process of solvent (hexane) reduction takes approximately 20 – 30 minutes. DO NOT leave the unit unattended as extracts may be blown to dryness and loss of PCBs or Pesticides as well as surrogate and matrix spike may occur. Immediately notify the extraction supervisor if an extract is blown to dryness and note the incident in the sample extraction logbook.

12.4.1.11. Concentrate the extract to approximately 10mL. Remove the 60mL VOA vial from the Evaporator unit, being careful not to drip.

12.4.1.12. Fill the sample vial back up to just below the base of the neck of the vial with hexane.

12.4.1.13. Following the LV TurboVap Evaporator procedure described above, concentrate the extract to approximately 2mL or 5mL depending on the set volume. Remove the 60mL VOA vial from the Evaporator unit being careful not to drip water from the outside of the vial into any of the other samples.

12.4.1.14. Carefully transfer the extract into a pre-rinsed volumetric flask. The set volume is either 10mL or in the case of CSGB analysis 5mL.

12.4.1.15. Rinse the 60mL VOA vial with approximately 1.0mL of hexane and add this to the volumetric flask. Repeat this process until the bottom of the meniscus is just touching the line on the

volumetric. If you go over the set volume amount, the sample must be concentrated again and set to the correct volume.

12.4.1.16. Stopper with a pre-rinsed stopper and invert the volumetric several times to mix thoroughly.

12.4.1.17. Transfer the extract into a pre-rinsed and properly labeled 4 dram vial.

12.4.2. Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection, contain co-extracted interfering substances which must be removed before accurate chromatographic analysis can be performed. See separate cleanup SOPs for details (S-NY-O-337, S-NY-O-338, S-NY-O-339 and S-NY-O-340, as applicable).

12.4.3. **Final Extract Preparation:**

12.4.3.1. Transfer the extract to a hexane pre-rinsed and correctly labeled final 4-dram vial.

12.4.3.2. Complete all information in LIMS. Submit samples and project folder to GC Analyst.

13. Quality Control

13.1. The extraction technician should have completed an acceptable demonstration of capability before performing the method without supervision. The addition of spiking material to a sample or blank must be witnessed by another extraction chemist and noted in LIMS. All surrogates and matrix spikes must meet acceptable QC limits.

13.2. A method blank sample and lab control spike must be prepared per each extraction batch or 1 per 20 site samples, whichever is more frequent. A matrix spike/matrix spike duplicate (or lab duplicate) should be prepared for every 20 site samples or as per client specified quality assurance project plan (QAPP).

13.2.1. Spike default for 8082A analysis for LCS, MS, and MSD is 1.0mL A1242 at 0.5ug/mL in acetone for a 1L sample. Client and/or project specifications may dictate alternate amount or component mixture.

13.2.2. Spike default for CSGB analysis for LCS, MS, and MSD is 0.2mL A1242 at 1.0ug/mL in acetone for a 1L sample. Client and/or project specifications may dictate an alternate amount or component mixture.

13.2.3. Spike default for 8082A/CQCS analysis for LCS, MS, and MSD is 1.0mL A1242 at 1.0ug/mL in acetone for a 1L sample. Client and/or project specifications may dictate an alternate amount or component mixture.

13.2.4. Spike default for 8081B analysis for LCS, MS, and MSD is 0.2mL of Mixed Pesticide Cal. Standard at 1.0ug/mL in Methanol. Client and/or project specifications may dictate alternate amount or component mixture.

13.3. Surrogates are added to each sample prior to extraction to measure extraction/cleanup efficiency.

13.3.1. Default surrogate for 8082A analysis is 1.0mL 0.05ug/mL TCMX / 0.5ug/mL DCBP in acetone for a 1L sample. Client and/or project specifications may dictate alternate amount.

13.3.2. Default surrogate for CSGB analysis is 0.5mL Nonachlorobiphenyl at 0.2ug/mL in acetone for a 1L sample. Client and/or project specifications may dictate alternate amount.

13.3.3. Default surrogate for 8082A/CQCS analysis is 1.0mL Nonachlorobiphenyl at 0.2ug/mL in acetone for a 1L sample. Client and/or project specifications may dictate alternate amount.

13.3.4. For 8081B analysis the default surrogate is 0.4mL 0.05ug/mL TCMX/ 0.5ug/mL DCBP in methanol. Client and/or project specifications may dictate alternate amount.

14. Data Analysis and Calculations

14.1. Please consult determinative methods (or latest versions of SOPs for EPA Method 8082A, S-NY-O-314 or EPA 8081B, S-NY-O-131, CSGB, S-NY-O-294, and CQCS, S-NY-O-133) for calculations.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Please consult determinative methods (or latest versions of SOPs for EPA Method 8082A, S-NY-O-314 or EPA 8081B, S-NY-O-131, CSGB, S-NY-O-294, and CQCS, S-NY-O-133) for Data Assessment and Acceptance Criteria for Quality Control Measures.

16. Corrective Actions for Out-of-Control Data

16.1. Consult determinative methods (or latest versions of SOPs for EPA Method 8082A, S-NY-O-314 or EPA 8081B, S-NY-O-131, CSGB, S-NY-O-294, and CQCS, S-NY-O-133) for Corrective Actions for Out of Control Data.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Please consult determinative methods (or latest versions of SOPs for EPA Method 8082A, S-NY-O-314 or EPA 8081B, S-NY-O-131, CSGB, S-NY-O-294, and CQCS, S-NY-O-133) for Contingencies Actions for Out of Control Data.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. Please consult determinative methods (or latest versions of SOPs for EPA Method 8082A, S-NY-O-314 or EPA 8081B, S-NY-O-131, CSGB, S-NY-O-294, and CQCS, S-NY-O-133) for method performance.

19. Method Modifications

19.1. The SPE disks used by the lab (SDB), listed in section 9.3, are not the disks recommended for PCBs and organochlorine pesticides found in Section 11.3.1 and Table 1 of Method 3535A (C_{18}). Section 6.3 of Method 3535A does allow for alternate disks provided the lab can demonstrate adequate performance for the analytes of interest.

19.2. The lab adjusts the sample pH to <2 (see Section 12.1.8) instead of the recommended pH range of 5-9 as listed in Section 11.2 and Table 1 of Method 3535A.

19.3. The pre-wet, or washing, steps listed in Attachment II do not match exactly with the first and second washing steps listed in Sections 11.4.1 and 11.4.2 of Method 3535A. Both processes start with a pre-rinse using methylene chloride; however, Method 3535A states to use acetone next for OC pesticides and no second rinse for PCBs. The SOP states to use a second step rinse of hexane and then acetone for both OC pesticides and PCBs. The pre-wet steps used by the lab come from the SPE equipment manufacturer’s recommendations.

19.4. The rinse steps listed in Attachment II do not match exactly with the elution steps listed in Section 11.7 of Method 3535A. Both processes start with a rinse using acetone. However, for OC pesticides, Method 3535A states to use methylene chloride as the second rinse solvent, while the SOP states to use hexane as the second rinse solvent. For PCBs, Method 3535A states to use acetonitrile as the second rinse solvent, while the SOP states to use hexane as the second rinse solvent. The rinse/elution steps used by the lab come from the SPE equipment manufacturer's recommendations.

19.5. Method 3535A uses a 1L sample volume for extraction. On occasion the lab will use a 2L sample volume for the extraction to achieve a lower reporting limit.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. The extraction technician should have received in-house safety training and should know the location of first aid equipment and the emergency spill/cleanup equipment, before handling any apparatus or equipment.

22.2. Safety glasses, a lab coat, and gloves must be worn when handling glassware and samples.

22.3. Polychlorinated biphenyls and pesticides have been classified as a known or suspected carcinogen. The extraction technician must review the Safety Data Sheets (SDS) for PCBs and pesticides and all reagents used in the procedure before beginning the extractions. All equipment and solvents should be handled within a laboratory fume hood.

23. Waste Management

23.1. See latest version of SOP S-NY-W-054 for details regarding Waste Management.

24. Pollution Prevention

24.1. See the latest version of SOP S-NY-S-168 for pollution prevention measures.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. U.S. EPA SW-846 "Test Methods for Evaluation Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods," Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

25.5. "Guide to Environmental Analytical Methods," Fourth Edition, Genium Publishing Corporation, 1998.

25.6. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants," July 1988.

25.7. Horizon Technology, "SPE-DEX® 4790 Series Extractor: Automated Solid Phase Extractor System User's Guide," July 2001.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: SPE Extractor Diagram.

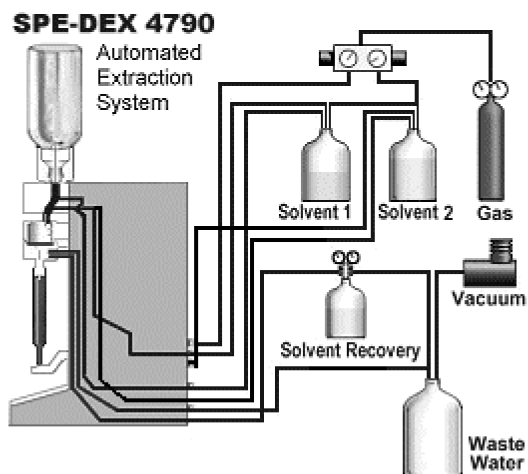
26.2. Attachment II: Extraction Methods.

26.3. Attachment III: Method Flow Chart.

27. Revisions

Document Number	Reason for Change	Date
S-NY-O-218-rev.07	General: converted to new format. Section 10: removed reagents involved in cleanup procedures. Section 12.1.2: added current practice for TCLP extracts. Section 12.4.5: removed cleanup procedures and added references for cleanup SOPs. Sections 19.1-19.4: added method modifications. Sections 25.1-25.3: added standard Pace references.	27Jan2015
S-NY-O-218-rev.08	General: removed information relating to extraction with volumes greater than 1L Sections 12.1.7, 12.3.10, and 19.5: added	30March2015

Attachment I: SPE Extractor Diagram



*NOTE: Limited diagram of the SPE-DEX® extractor set up. There are actually eight solvent bottles, five pre-wet solvents and three rinse solvents.

Attachment II: Extraction Methods

Extraction Method First Bottle

Step Number	Procedure
Step 1: Pre-wet Solvent: Dichloromethane	Soak Time: 1:00 minute Air Dry Time: 30 seconds
Step 2: Pre-wet Solvent: Hexane	Soak Time: 1:00 minute Air Dry Time: 30 seconds
Step 3: Pre-wet Solvent: Acetone	Soak Time: 1:00 minutes Air Dry Time: 30 seconds
Step 4: Pre-wet Solvent: Methanol	Soak Time: 1:30 minute Air Dry Time: 0:00 minutes
Step 5: Pre-wet Solvent: Reagent Water	Soak Time: 1:00 minute Air Dry Time: 0:00 minutes
Step 6: Pre-wet Solvent: Reagent Water	Soak Time: 1:00 minute Air Dry Time: 0:00 minutes
Step 7: Sample Extraction Solvent: None	Time depends on particulates and sample flow through the solid phase disk.
Step 8: Air Dry Disk Solvent: None	Air Dry Time: 5:00 minutes
Step 9: Rinse Solvent: Acetone	Soak Time: 1:30 minutes Air Dry Time: 1:00 minute
Step 10: Rinse Solvent Hexane	Soak Time: 1:30 minutes Air Dry Time: 1:00 minute
Step 11: Rinse Solvent Hexane	Soak Time: 1:30 minutes Air Dry Time: 1:00 minute
Step 12: Rinse Solvent Hexane	Soak Time: 1:30 minutes Air Dry Time: 1:00 minute

Extraction Method Second Bottle

Step Number	Procedure
Step 1: Sample Extraction Continued	Pressing Start on SPE-DEX 4790 Extractor will continue to process second bottle.
Step 2: Air Dry Disk	Air Dry Time: 5:00 minutes
Step 3 :Rinse Solvent: Acetone	Soak Time: 1:30 minute Air Dry Time: 1:00 minute
Step 4: Rinse Solvent: Hexane	Soak Time: 1:30 minute Air Dry Time: 1:00 minute
Step 5: Rinse Solvent: Hexane	Soak Time: 1:30 minute Air Dry Time: 1:00 minute
Step 6: Rinse Solvent: Hexane	Soak Time: 1:30 minute Air Dry Time: 1:00 minute

Attachment II: Extraction Methods (continued)

Extraction Method 8081 Pesticide

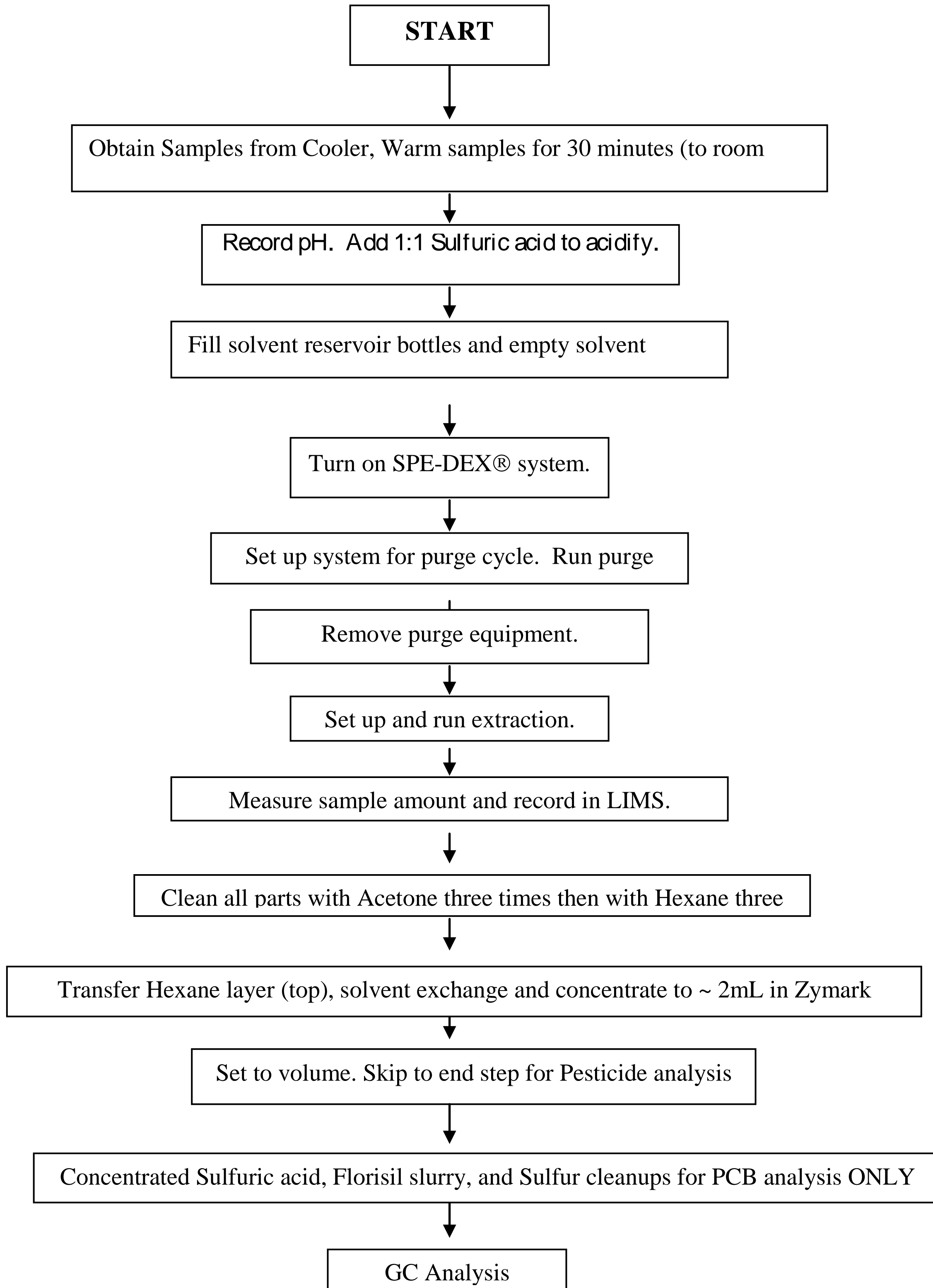
Step Number	Procedure
Step 1: Pre-wet Solvent: Dichloromethane	Soak Time: 1:00 minute Air Dry Time: 30 seconds
Step 2: Pre-wet Solvent: Hexane	Soak Time: 1:00 minute Air Dry Time: 30 seconds
Step 3: Pre-wet Solvent: Acetone	Soak Time: 1:00 minutes Air Dry Time: 30 seconds
Step 4: Pre-wet Solvent: Methanol	Soak Time: 30 seconds Air Dry Time: 0:00 minutes
Step 5: Pre-wet Solvent: Reagent Water	Soak Time: 10 seconds Air Dry Time: 0:00 minutes
Step 6: Sample Extraction Solvent: None	Time depends on particulates and sample flow through the solid phase disk.
Step 7: Air Dry Disk Solvent: None	Air Dry Time: 3:00 minutes
Step 8: Rinse Solvent: Acetone	Soak Time: 3:00 minutes Air Dry Time: 2:00 minutes
Step 9: Rinse Solvent: Hexane	Soak Time: 3:00 minutes Air Dry Time: 2:00 minutes
Step 10: Rinse Solvent Hexane	Soak Time: 1:00 minute Air Dry Time: 1:00 minute
Step 11: Rinse Solvent Hexane	Soak Time: 1:00 minute Air Dry Time: 1:00 minute
Step 12: Rinse Solvent Hexane	Soak Time: 1:00 minute Air Dry Time: 1:00 minute

Extraction Method Pest 2nd Bottle

Step Number	Procedure
Step 1: Sample Extraction Continued	Pressing Start on SPE-DEX 4790 Extractor will continue to process second bottle.
Step 2: Air Dry Disk	Air Dry Time: 3:00 minutes
Step 3 :Rinse Solvent: Acetone	Soak Time: 3:00 minutes Air Dry Time: 2:00 minutes
Step 4: Rinse Solvent: Hexane	Soak Time: 3:00 minutes Air Dry Time: 2:00 minutes
Step 5: Rinse Solvent: Hexane	Soak Time: 1:00 minute Air Dry Time: 1:00 minute
Step 6: Rinse Solvent: Hexane	Soak Time: 1:00 minute Air Dry Time: 1:00 minute

Step 7: Rinse Solvent: Hexane	Soak Time: 1:00 minute Air Dry time: 1:00 minute

Attachment III: Method Flow Chart



APPENDIX P3-5
SOP FOR THE DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBS) AROCLORS IN AQUEOUS SAMPLES BY SW-846 METHOD 8082A
(S-NY-O-314-REV.03)

STANDARD OPERATING PROCEDURE

DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBS) AROCLORS

Reference Methods: EPA Method 8082A

SOP Number:	S-NY-O-314-rev.03
Effective Date:	07/14/15
Supersedes:	S-NY-O-314-rev.02

APPROVALS

[signature]	07/14/15
Assistant General Manager	Date
[signature]	07/14/15
Quality Manager	Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose/Identification of Method

1.1. This Standard Operating Procedure (SOP) is used to determine Polychlorinated Biphenyl (PCB) Aroclors by gas chromatography with electron capture detection and total Aroclor quantification using EPA SW-846 Method 8082A- Polychlorinated Biphenyl (PCB) Aroclors by capillary column gas chromatography (GC).

2. Summary of Method

2.1. Samples are extracted with a pesticide analytical grade solvent. The extracts are further processed by concentration and a series of clean-up procedures. The sample extracts are then analyzed by injecting onto a gas chromatographic system equipped with an electron capture detector.

2.2. The purpose of this SOP is to provide a detailed written document for quantification of PCBs as Aroclors according to SW-846 Method 8082A specification.

2.3. This SOP provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCBs as Aroclors by gas chromatography. Sample extraction and cleanup procedures are described separately in additional laboratory Standard Operating Procedures.

2.4. Extensive knowledge of this SOP and EPA Method 8082A is required. The analysis portion of this method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analysis of PCBs by Method 8082A.

3.2. **Parameters**: The following PCB Aroclors can be determined by this method:

Compound	CAS Number
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

4. Applicable Matrices

4.1. This SOP is applicable in the determination and quantification of PCBs as Aroclors as outlined in EPA SW-846 Method 8082A. It is applicable to the following matrices: water, soil, sediment, sludge, oil, fuel oil, waste solvent, fish, other aquatic animals, tissue samples.

5. Limits of Detection and Quantitation

5.1. The following are default reporting limits based on the lowest calibration standard:

Matrix	Sample Mass/Volume Extracted	Calibration Curve Low Standard	Extract Volume	RL (PQL) (all Aroclors)
Soil/Sediment Solid	10g	20ng/mL	25mL	0.050mg/kg
Water	1 Liter	5ng/mL	10mL	0.050ug/L
Water	1 Liter	5ng/mL	5mL	0.025ug/L
Biota	10g (wet weight basis)	20ng/mL	25mL	0.050mg/kg
Waste Oil	0.5g	20ng/mL	25mL	1.00mg/kg
Wipe	1 Wipe	20ng/mL	25mL	0.500ug/wipe

5.2. Individual MDLs are determined based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. MDLs must be determined again whenever a major change in instrumentation or extraction methodology occurs.

5.3. MDLs are verified annually by the extraction and analysis of a low level MDL verification check sample. The Aroclor must be observed qualitatively in the MDL verification check sample.

5.4. Global MDL values can be obtained by request from the QA Department.

6. Interferences

6.1. Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters exhibit response on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Laboratory method blanks must be thoroughly reviewed for presence of non-target peaks and comparison of samples with blank chromatographic patterns.

6.2. Elemental sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur can be removed through the use of Method 3660.

6.3. Polychloroterphenyls (PCTs), polybrominatedbiphenyls (PBB), polychlorinated naphthalenes (PCN), as well as dioxins can co-elute with PCBs. Carry-over from these compounds, when in high concentration, is common if clean-up procedures are not followed. These materials may be removed through the use of specified clean-up procedures.

6.4. Pesticides can be a source of contamination through breakdown into components such as hexachlorobenzene (HCB). This chlorinated compound can carry-over on the GC column, and contaminate samples. Specified clean-up procedures should be followed to eliminate this as a source of contamination when analyzing PCBs. High concentrations of pesticides can cause carry-over on GC columns.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Sample Collection and Preservation:

7.1.1. Routine soil, sediment, sludge, solid, caulk, and concentrated liquid samples should be collected in 8 oz clear glass wide-mouth jars, fitted with a Teflon-lined cap. Aqueous samples should be collected in 1 liter amber glass bottles with a Teflon-lined cap. Project specific protocols may require that containers be pre-cleaned to EPA specification protocol A –. Protect samples from light.

7.1.2. All samples must be placed on ice or refrigerated at >0-6°C from the time they are collected until delivery to the lab. Samples that are collected within driving distance of the laboratory and delivered the

same day may not have reached temperature acceptance limits. These samples are deemed acceptable if evidence of cooling is present (i.e., they are received with ice in the cooler).

7.2. Sample Shipment:

7.2.1. Sample Shipment is accomplished through a carrier such as Federal Express or United Postal Service for overnight 1-day delivery to the lab. Shipment is normally handled by the field personnel collecting the samples and coordinated with sample receiving department at the lab. Samples can also be picked up by the lab courier service if samples are collected within driving distance to the lab.

7.3. Sample Storage:

7.3.1. The samples must be protected from light and refrigerated at >0-6°C from time of receipt until they are removed from storage for extraction. Remaining sample material will be stored protected from light and refrigerated at >0-6°C. Sample will be disposed of or stored / archived according to project specifications.

7.3.2. Routine soil, sediment, sludge, solid, liquid and concentrated liquid samples are stored in a refrigerator dedicated for this type of sample.

7.4. Sample Extract Storage:

7.4.1. Sample extracts must be protected from light and refrigerated at >0-6°C during the analysis. After analysis is complete, sample extracts will be discarded after 60 days or can be archived in a freezer at less than -20°C for longer periods of time depending on the program requirements.

7.4.2. Field samples, sample extracts, and calibration standards must be stored separately.

7.5. Required Hold Time:

7.5.1. Extraction of solid samples by appropriate technique must be completed within one year from sample collection. Extraction of aqueous samples by appropriate technique must be completed within one year from sample collection. Sample extracts must be analyzed within forty days from extraction.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. Accuracy – The nearness of a result or the mean of a set to the true value. Accuracy is assessed by analysis of references samples and percent recoveries.

8.3. Analytical Batch –The basic unit for analytical quality control is the analytical batch, which is defined as samples which are analyzed together with the sample method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar matrices (e.g. water, sediment, soil, etc.).

8.4. Blank – A blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix, however, a universal blank matrix does not exist for solid samples, but sometimes sodium sulfate is used as a blank matrix. The blank is taken through the appropriate steps of the process. A reagent blank is an aliquot of analyte-free water or solvent analyzed with the analytical batch. Field blanks are aliquots of analyte-free water or solvents brought to the field is sealed containers and transported back to the laboratory with the sample containers. Trip blanks and equipment blanks are two specific types of field blanks. Trip blanks are not opened in the field. They are a check on sample contamination originating from sample transport, shipping and from site conditions. Equipment blanks are opened in the field and the contents are poured appropriately over or through the

sample collection device, collected in a sample container, returned to the laboratory as a sample. Equipment blanks are a check on sampling device cleanliness.

8.5. Continuing Calibration Check Standard (CCCS) –The continuing calibration check standard contains all target analytes found in the calibration standards and is used to verify that the initial calibration is prepared correctly and that the instrument system is correctly calibrated. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards.

8.6. Calibration Standard (ICAL)– A series of known standard solutions used by the analyst for instrument calibration. Calibration standards are prepared from primary standard and/or stock standard solutions.

8.7. CAS Number – An assigned number used to identify a chemical. CAS stands for Chemical Abstracts Service, an organization that indexes information published in Chemical Abstracts by the American Chemical Society and that provides index guides by which information about particular substances may be located in the abstracts. Sequentially assigned CAS numbers identify specific chemicals, except when followed by an asterisk (*) which signifies a compound (often naturally occurring) of variable composition. The numbers have no chemical significance. The CAS number is a concise, unique means of material identification. (Chemical Abstracts Service, Division of American Chemical Society, Box 3012, Columbus, OH 43210: [614] 447-3600).

8.8. Duplicate– A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

8.9. Environmental Sample – An environmental sample or field sample is a representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination as requested or required.

8.10. Initial Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the analytical detector or method.

8.11. Instrument Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

8.12. Laboratory Control Sample (LCS) – Also known as the Quality Control (QC) Check Standard or Quality Control (QC) Check Sample. The LCS consists of an aliquot or reagent water or other blank matrix to which known quantities of the method analytes are added. The LCS is extracted and analyzed exactly like a field sample, and its purpose is to determine whether the analysis is in control and whether the laboratory is capable of making accurate and precise measurements.

8.13. Laboratory Method Blank – An analytical control consisting of all reagents and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

8.14. Matrix – The predominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

8.15. Matrix Spike – Aliquot of sample (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

8.16. Matrix Spike Duplicate – A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

8.17. Method Detection Limit (MDL) – The minimum constituent concentration that can be measured and reported with 99% confidence that the signal produced is different from the blank in a given matrix. The MDL is determined from a minimum of seven or eight replicate samples, taken through the entire preparation

and analysis procedure. The standard deviation, *s*, of those replicates is multiplied by a student's t factor in order to calculate the MDL.

8.18. SDS – Safety Data Sheet. OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs.

8.19. PCB- Polychlorinated biphenyls are a class of 209 individual chemical compounds (congeners), in which one to ten chlorine atoms are attached to biphenyl. Use of PCBs has made them a frequent environmental pollutant.

8.20. Precision – The agreement between a set of replicate measurements without assumption of knowledge of the true value. Precision is assessed by means of duplicate/replicate sample analysis.

8.21. Quality Control – Set of measures within a sample analysis methodology to assure that the process is in control.

8.22. Standard Curve – A standard curve is a curve which plots concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by diluting the stock analyte solution in graduated amounts which cover the expected range of the samples being analyzed. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

8.23. Stock Solution – Standard solution which can be diluted to derive the other standards.

8.24. Surrogate – Organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, calibration and check standards, samples (including duplicates and QC reference sample) and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

8.25. Surrogate Standard – A pure compound added to a sample in the laboratory just before processing so that the overall efficiency of a method can be determined.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Gas Chromatograph: Complete system for high resolution, capillary column capability and all required accessories. Pace Analytical Services, Inc. will use a Varian/Bruker Models 3800 and 450 (or equivalent) gas chromatograph (or equivalent), equipped with a Model 1177 split/splitless injector (or equivalent), temperature programmable oven, LEAP GC pal automatic sampler (or equivalent), and electron capture detector (or equivalent). A data system and integration of detector signal is interfaced to the gas chromatograph.

9.2. Chromatographic Data System: A data system for measuring peak height and peak area. An Empower computer network based workstation (Waters Corporation), will be employed to capture detector response and digitally store the chromatographic, electronic peak integration for precise calculations, database structuring of the analytical information, and archival capabilities.

9.3. Column (Primary Helium Carrier Gas): ZB-1, Phenomenex Cat. No. 7HG-G001-11; 30m x 0.25mm x 0.25um ; DB-1, J&W Part No. 122-1032; 30m x 0.25mm x 0.25um ; or equivalent.

9.4. Column (Secondary Helium Carrier Gas): ZB-5, Phenomenex Cat. No. ZB-5-G002-11; 30m x 0.25mm x 0.25um; DB-5, J&W Part No. 122-5032; 30m x 0.25mm x0.25um; or equivalent.

9.5. Column (Primary Hydrogen Carrier Gas): ZB-1MS, Phenomenex Cat. No 7FD-G011-08; 20m x 0.18mm x 0.18um.

9.6. Column (Secondary Hydrogen Carrier Gas): ZB-5, Phenomenex Cat. No 7FD-G002-08; 20m x 0.18mm x 0.18um.

9.7. Class A volumetric flasks: 5.0-100mL.

9.8. 8 dram vials and 4 vials dram for sample extract storage.

9.9. Pasteur pipettes.

9.10. 250mL and 100mL beakers, glass.

9.11. Disposable 1.0, 5.0, and 10.0mL pipettes.

9.12. Hexane, Burdick and Jackson-Pest Grade (or equivalent).

9.13. Acetone, Burdick and Jackson.-Pest Grade (or equivalent).

9.14. Toluene, Baker, (Cat.No. 9336-03) (or equivalent).

9.15. Methylene Chloride, Burdick and Jackson (Cat. No. 300-4) (or equivalent).

9.16. Ferrules: 0.4mm graphite/vespel, Restek 20229, and ¼" graphite ferrules, Restek 20210 or equivalent.

9.17. Injector septa: Thermolite Septa, Restek 20365 or equivalent.

9.18. Injector liner: Low Pressure Drop Liner w/Glass Wool, Restek 21033 or equivalent.

9.19. SGE Injector Syringe 10.0µL: SGE 002987 or equivalent.

9.20. Auto sampler vials: Snap vial 12x32mm Clear w/P, Microliter 11-5200 (or equivalent).

9.21. Snap Caps: 11mm Natural Snap Cap PTFE, Microliter 11-0051N-B (or equivalent).

10. Reagents and Standards

10.1. Aroclor Stock Standard Solutions:

10.1.1. Polychlorinated Biphenyls - Stock standards are prepared from individual Aroclor stock solutions from Accustandard. See Attachment 1 Table 1 for the exact preparation of each compound.

10.1.2. The stock standards are transferred into screw-cap boston bottles and stored in a freezer 0°C, protected from light. Stock standards should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards. Stock PCB standards must be replaced after one year, or sooner if a problem with instrument calibration is detected.

PCB Formulation	Supplier	Catalog #	Conc. (PPM)
A1016	Accustandard	C-216S-H-100x	10000.0
A1221	Accustandard	C-221S-H-100x	10000.0
A1232	Accustandard	C-232S-H-100x	10000.0
A1242	Accustandard	C-242S-H-100x	10000.0
A1248	Accustandard	C-248S-H-100x	10000.0
A1254	Accustandard	C-254S-H-100x	10000.0

A1260	Accustandard	C-260S-H-100x	10000.0
A1262	Accustandard	C-262S-H-10x	1000.0
A1268	Accustandard	C-268S-H-10x	1000.0
TCMX/DCBP (surrogate)	Ultra Scientific	CUS-4911	500/5000

*unless otherwise noted hexane is the solution used to make all dilutions.

10.2. Calibration Standards:

10.2.1. Calibration standards are prepared at five concentration levels using a prepared working standard. See Attachment I, Table 2 and for the preparation and exact concentrations of the working standards. The following five standards make up the initial calibration curve standard set for a High Level curve: 20ng/mL, 100ng/mL, 250ng/mL, 500ng/mL, 1000ng/mL. The following five standards make up the initial calibration curve set for a Low Level curve: 5ng/mL, 10ng/mL, 20ng/mL, 50ng/mL, 100ng/mL.

10.2.2. The two surrogates Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) are included in the A1254 calibration standards. The standard for TCMX/DCBP is prepared by diluting 1mL of TCMX/DCBP custom standard solution (ULTRA, cat.#CUS-4911, at 500/5000 ng/mL) into a 1000mL volumetric flask resulting in a solution of TCMX/DCBP at 0.5/5.0ug/mL.

10.2.3. Refer to Attachment I, Table 3 for instructions on preparation of the calibration standards containing A1254 and the surrogates. Refer to Attachment 1, Table 2 for instructions on preparing the remaining calibration standards.

10.2.4. Transfer all calibration standards to ASE vials and store in a refrigerator at 0-6°C, protected from light. Calibration standards must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

10.3. PCB Continuing Calibration Stock Standards:

10.3.1. The stock standards are transferred into a screw cap boston bottles and stored in a refrigerator protected from light. Stock standard should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards. Stock PCB standards must be replaced annually, or sooner if a problem with instrument calibration is detected.

PCB	**Supplier**	**Catalog #**	**Conc. (ug/mL)**
A1016	Chem Service	S-11086J	1000
A1221	Chem Service	S-11087J	1000
A1232	Chem Service	S-11088J	1000
A1242	Chem Service	S-11089J	1000
A1248	Chem Service	S-11090J	1000

A1254	Chem Service	S-11091J	1000
A1260	Chem Service	S-11092J	1000
A1262	Ultra Scientific	EPA-1372	1000
A1268	Ultra Scientific	EPA-1382	1000

10.4. Continuing Calibration Standards:

10.4.1. The surrogate compounds Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) are included in all Continuing Calibration Check Standards at a concentration near the mid-point of the surrogate calibration curve sequence. All continuing calibration standards are prepared independently from calibration standards, by using an alternate source purchased from standard vendors. Refer to Attachment II, Tables 1-3 for instructions on preparation of these standards.

11. Calibration and Standardization

11.1. Gas chromatographic operation parameters: See Attachment III.

11.2. Initial GC Calibration:

11.2.1. GC calibration is performed by the external standard calibration procedure. Prior to running samples the system must be calibrated and system performance must be verified.

11.2.2. Establish the gas chromatographic operating parameters outlined in the Procedure section and prepare the calibration standards at the five concentrations outlined in the Reagent and Standard section. Inject each calibration standard using the GC Autosampler and the parameters outlined in the Procedure section. Note: The same parameters are used for actual samples.

11.2.3. For each Aroclor, 5 peaks are selected to prepare calibration curves. The peaks selected from the multi-component Aroclor formulations were based on maximizing the separation for each Aroclor (i.e., minimizing peak overlap in retention time). Consideration was also given to selecting peaks that normally did not have problems with co-elution with interfering peaks or possible co-elution with organochlorine pesticides. The determined area of the five peaks selected for calibration is processed by the data workstation as a group, combining the area for calculations of the calibration factors. The following table lists the Aroclors that are included in the initial calibration and the peak numbers used.

Aroclor	Peak Numbers
A1016	6, 7, 8, 9, 10
A1221	1, 2, 3, 4, 5
A1232	5, 7, 8, 9, 10
A1242	6, 7, 8, 9, 10
A1248	11, 12, 13, 14, 15
A1254	16, 17, 18, 19, 20
A1260	20, 21, 22, 23, 24
A1262	20, 21, 22, 23, 24
A1268	23, 24, 25, 26, 27

11.2.4. For the initial calibration curve to be considered valid, the percent relative standard deviation of response factors must be less than 20% over the working range if average calibration factor quantitation is used. Note: the % RSD is a useful check for linearity through the origin and is used as a data quality indicator. In general an inverse weighted linear calibration curve with intercept is used for quantitation and is not replaced with the average calibration factor. For linear calibration curve the Correlation Coefficient R must be greater than 0.990.

11.2.5. Once linear calibration has been established it is subjected to an additional check. This check is the comparison of the calculated amount of the low calibration standard for each Aroclor against the expected amount of the standard using the % difference. Re-fitting the calibration data back to the model or calculating the % difference is determined by using the following equation:

% Difference = (Cc-Ce/Ce)x100

Where Cc=Calculated amount of standard, in mass or concentration units.

Ce=Expected amount of standard, in mass or concentration units.

The absolute value of the percent difference between these two amounts for

Every calibration level should be less than or equal to 20%.

11.2.6. Our laboratory uses a computer based chromatography software module (Water Corporation, Empower software) interfaced to the gas chromatograph. The workstation processes the detector signal, performs an analog to digital conversion, and stores the digitized chromatograms on the computer hard disk. Integration of peak areas and production of chromatograms is performed in the Empower software. All data analysis will be carried out in Empower including calculating calibration curves/response factors, report generation, and archival of data.

11.2.7. If a re-calibration is performed, the CCCS must be analyzed again and values calculated using the new relative response factors. If the CCCS fails to meet the percent difference criteria after re-calibration, sample analysis must not proceed until the problem is found and corrected (*i.e.,* GC gas leak, autosampler syringe plugged, broken injector liner).

11.3. Retention Time Windows:

11.3.1. The GC system should be checked by the analyst to make sure it is functioning properly before establishing retention time windows. Select a calibration standard and inject three times over a 72-hour time period.

11.3.2. For each peak calculate the standard deviation resulting from the variation in the three retention times for that peak.

11.3.3. The retention time window is defined as plus or minus three times the standard deviation of the three retention time determinations.

11.3.4. If the standard deviation of the selected peak is zero, then a default standard deviation of 0.01 minutes is used. If it is the last eluting peak that the zero for the standard deviation, then substitute the standard deviation of the peak eluting before the last peak.

12. Procedure

12.1. Sample Extraction and Preparation: The following SOP’s detail sample extraction procedures that are utilized in preparing samples for analysis by this analytical method:

SOP NAME	TITLE	EPA Method
S-NY-O-017	Extraction and Percent Lipids for Fish and Biota	8082A
S-NY-O-088	PCB Extraction Of Wipe	8082A, 3540C
S-NY-O-111	Waste Dilution EPA 3580 for PCB 8082	8082A, 3580A
S-NY-O-140	PCB Screening by GC	3510C, 3520C, 3545A
S-NY-O-141	SW-846 3510C H2O PCB extraction	8082A, 608, 3510C

12.2. Gas Chromatographic Procedures:

12.2.1. Prescreening of sample extracts: See standard operating procedure S-NY-O-140 for details on the PCB screening procedures used prior to final analysis by this method. Prescreening is a fast and effective way to determine if re-extracts are required and dilutions for over ranged samples. The GC will be standardized by using Aroclor 1221, Aroclor 1242, and Aroclor 1260. These three Aroclor formulations incorporate most environmental PCBs found in sample extracts and provide a good estimate of PCB amount for final dilution for this determinative method. A three level calibration curve is utilized (0.50ug/mL, 2.5ug/mL, and 5.0ug/mL standards). The concentration of each Aroclor (grouped as Aroclor 1221, Aroclor 1242, and Aroclor 1260 only) in a sample will be calculated based on the extract volume (not the sample weight or volume) to supply solution concentration values that show if the extract needs to be diluted for final capillary GC analysis. If a dilution is necessary, sample extracts are diluted to a solution concentration near 0.500ug/mL, so ensuring each sample quantifies in the middle of the calibration curve.

12.2.2. Approximately 1.0mL of the final dilution extract is then transferred into a labeled autosampler vial.

12.2.3. The sequence of the analytical queue is set up in the Laboratory Information Management System (LIMS) as a unique batch file. This file contains the exact order in which standards, instrument blanks, and samples will be analyzed. Once the sample set is uploaded into the Empower acquisition/run screen and saved, the sample set is printed and the samples are loaded into the GC autosampler tray in the order specified by the sample set queue.

12.2.4. The following labeling will be used on the autosampler vial and for the sample set file created for the analytical queue.

12.2.4.1. The initial calibration standard will be labeled as 040516A, 040516B, etc. Substitute the actual date of analysis and the Aroclor used in the file name.

12.2.4.2. The instrument blanks will be labeled 070405B01, B02, B03, etc. Substitute the actual date of analysis in the file name.

12.2.4.3. The continuing calibration check standards will be labeled CS160405A CS160405B, etc. Substitute the actual date of analysis and the Aroclor used in the file name.

12.2.4.4. Samples are labeled with the laboratory identification number on the autosampler vial. In the sample set file the laboratory identification number, along with the client identification, sample weight, set volume and dilution are entered.

12.2.5. At this point the chromatography software can be initiated to start data collection. The gas chromatograph is placed into run mode and sample analysis is performed until the analytical queue is complete.

12.2.6. Peak Identification:

12.2.6.1. Target peaks are identified in unknown samples based upon Retention Time (RT). The retention time of an unknown peak must fall within the retention time windows established.

12.2.6.2. Besides using retention time windows to assign peak IDs, the analyst should also rely on their own experience in recognition of multi-response PCB chromatograms. Caution should be exercised when identifying peaks which elute near interferences present in samples and blanks. Comparison of sample chromatograms with method blank and field blank chromatograms is useful in determining chromatographic interferences.

12.2.6.3. This method should be applied with caution when used in determining PCB of interest in unknown sample for which no prior historical information exists. In this case confirmatory column analysis or confirmation by GC/MS analysis may be advised.

12.3. Data Reduction/Reporting:

12.3.1. Final peak assignments and quantitation calculations are performed within the software along with the current instrument calibration. The final concentration results are provided in the reporting section of the software. Final concentration results are reviewed by QA department or other approved manager before release to the client.

12.3.2. Data Qualifiers: Sample Concentration Reports (Certificates of Analysis, Data Package Form 1's and Electronic Data Deliverables (EDDs) are generated using the appropriate data qualifiers as defined in Laboratory Form F-NY-Q-033 "Report Definitions Page" current revision.

13. Quality Control

13.1. This section outlines the necessary quality control samples that need to be generated at the time of sample extraction. The results of the quality control measurement samples document the quality of the data generated.

13.2. Method Blank- With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples and organic-free reagent water blank is processed.

13.2.1. The method blank must exhibit PCB levels less than the matrix defined minimum detection limit (MDL) / reporting limit (RL). If the method blank exhibits PCB contamination above the reportable MDL/RL, the samples associated with the contaminated blank should be re-extracted and analysis repeated. If there is no original sample available for re-extraction then the results should be flagged with a "B" indicating blank contamination. The value measured in the blank is reported for those samples associated with the particular blank out of criteria.

13.2.2. Method blanks can be reported with a "B" flag if the analyte detected is less than 10% of the regulatory limit associated with an analyte or is less than 10% of the sample result for the same analyte, whichever is greater.

13.3. Laboratory Control Sample (LCS)-

13.3.1. A Laboratory Control Spike (LCS), also referred to as a QC reference check standard, is extracted with each batch of samples at a rate of one per 20 samples. For water sample, spike one liter of laboratory organic free water, extract and analyze. For solid and tissue samples spike 10g of sodium sulfate, extract and analyze. For oil samples spike 0.5g of PCB free oil, extract and analyze. An Aroclor is chosen for the LCS analyte, typically based on program requirements or expected sample contamination. Calculate the percent recovery for the PCB spike. If the percent recovery for the LCS is out of criteria, (70-130%) the analysis is out of the control and the problem should be immediately corrected.

13.3.2. The following are default Laboratory Spikes Concentrations:

13.3.2.1. Aqueous Samples: 1.0mL of A1242 at 0.5ug/mL yielding a final sample concentration of 0.500ug/L.

13.3.2.2. Solid Samples: 1.0mL of A1242 at 12.5ug/mL yielding a final sample concentration of 1.25ug/g.

13.3.2.3. Note: Alternate spike concentrations and selection of Aroclors may be applicable based on project specific requirements.

13.4. Duplicate Analysis- Duplicate analysis of the same sample is performed to assess method precision. A duplicate can also be performed as a blind duplicate, so that identification with original sample is withheld. The analysis of a duplicate sample precludes that PCBs are to be found at appreciable levels in samples. If this is not known the analysis of matrix spike/matrix spike duplicates provide more consistent quality control information. The relative percent difference of the two measurements on the sample is calculated on total PCB concentration by the following equation:

RPD = (DUP1-DUP2)/AVG x 100

RPD = Relative Percent Difference
DUP1 = The greater of the measured values
DUP2 = The lesser of the measured values
AVG = Average of the two analyses
relative percent difference must be less than or equal to 30%

13.5. Matrix Spike and Matrix Spike Duplicate (MS/MSD):

13.5.1. A matrix spike is to be analyzed at a rate of one matrix spike per every 20 samples. Also matrix spike duplicate or duplicate sample is to be analyzed at a rate of one per every 20 samples. A matrix spike and an unspiked field sample/sample duplicate s may be appropriate in place of matrix spike/matrix duplicate, for soil and waste samples, where detectable amounts of organics are present.

13.5.2. The following are default Laboratory Matrix Spike Concentrations:

13.5.2.1. Aqueous Samples: 1.0mL of A1242 at 0.5ug/mL yielding a final sample added concentration of 0.500ug/L.

13.5.2.2. Solid Samples: 1.00mL of A1242 at 100ug/mL yielding a final sample added concentration of 10ug/g.

13.5.2.3. Note: Alternate spike concentrations and selection of Aroclors may be applicable based on project specific requirements.

13.5.3. Analyze one unspiked and one spiked sample. Calculate the percent recovery based on PCB concentration of both samples as follows:

P = A-B/T x 100

P = Percent recovery, %
A = concentration of analyte (PCB) in the spike sample aliquot
T = Know true values of the spike concentration
B = Background concentration of analyte (PCB) in the unspiked sample aliquot

13.5.4. Matrix spike recovery information is used to assess the long-term precision and accuracy of the method for each encountered matrix. Matrix spike/matrix spike duplicate results are not used alone to qualify an extraction batch. Generally, percent recovery for MS/MSD samples should be greater than or equal to 70% and less than or equal to 130% based on the total PCB concentration. If the percent recovery is outside the limits, all calculations should be checked and the data should be narrated to describe possible matrix interference.

13.6. Surrogates:

13.6.1. A surrogate compound is added to each sample, matrix spike, matrix spike duplicate, duplicate, method blank, and LCS at time of extraction. The surrogate compounds chosen for this method are Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP). The following are typical surrogate amounts added to normal encountered matrices. These amounts can be adjusted if the PCB background levels are high and the surrogate is being diluted out of analysis range.

13.6.1.1. Soil, sediment, sludge, oil, fuel oil, waste solvent, fish, other aquatic animals, tissue samples: 0.5mL of 0.5ug/mL TCMX/ 5.0ug/mL DCBP set to 25mL final extract volume.

13.6.1.2. Water: 1.0mL of 0.05ug/mL TCMX/ 0.5ug/mL DCBP set to 10mL final extract volume.

13.6.2. Surrogate compound is added to all instrument blanks that are analyzed after CCCS. Surrogate compound must meet lab established limits of TCMX 47-123% and DCBP 35-153%.

13.6.3. The surrogate recoveries must fall within lab established limits of TCMX 47-123% and DCBP 35-153%. If percent surrogate recovery is not within laboratory established limits for either surrogate, the following steps are required.

13.6.2.1. Review calculations that were used to generated surrogate percent recovery values to make certain there are no errors.

13.6.2.2. Check by GC analysis surrogate solutions used during sample extraction steps to ensure that no problems exist with spiking solutions.

13.6.2.3. Review data for chromatographic interferences.

13.6.2.4. Re-extraction and/or re-analysis of samples may be indicated if problems persist with surrogate recoveries. If the surrogate percent recovery is out of limits on the re-extracted samples, low or high surrogate recovery is due to matrix affects and the data can be reported as estimated. If above steps do not lead to satisfactory results then consult with organics manager to resolve the situation.

13.7. Continuing Calibration Check Standard (CCCS):

13.7.1. The initial CCCS is from an alternative source independent of the calibration check standards. It is prepared at a concentration approximately equal to the midlevel calibration standard. This standard is analyzed after the initial calibration standards, every tenth injection, and at the end an analytical sequence. One check standards must be run within a 12 hour analytical shift. The percent recover must be ± 20% of the true value.

13.7.2. If the criterion is exceeded, the analyst should inspect the system to determine the cause and perform maintenance as necessary. The system can then be recalibrated and sample analysis can proceed. Note that all samples which are not bracketed by valid check standards must be re-analyzed when the system is in-control.

13.8. Retention Time:

13.8.1. The retention time (RT) windows are established from the continuing calibration check standard (CCS) peak retention times. The CCS is analyzed three times over a 72-hour period and the standard deviation is calculated from the three retention time measurements. The standard deviation is multiplied by three and this establishes the retention time window for each quantified peak (±3SD). Use the retention time for a peak in the continuing calibration check standard to determine the midpoint of the retention time window for the analysis sequence. If the continuing calibration checks fall outside of these windows update the windows using the previous check standard. If the retention times are still outside the established windows instrument maintenance must be performed and recalibration may be required.

13.8.2. This function is performed in the chromatography software graphically as vertical dropdown retention time markers with retention time window brackets. Besides using the retention time window to assign peaks for quantification, the analyst should also rely on their experience in pattern recognition of multi-response sample analysis.

13.8.3. Retention time studies are available upon request form the QA department.

13.9. Analytical Sequence Queue: The following is an example of the order that initial calibration standards, continuing calibration check standards, method blanks, QC samples, and samples are placed in an analytical sequence. A continuing calibration check standard is run after every nine samples in the analytical sequence. All analytical sequences must end with a continuing calibration check standard regardless of the number of samples. Below is an example of an analytical sequence.

Injections	Material Injected
1-2	Hexane Blank
3-47	Initial Calibration Standards
48	Hexane Blank
49-57	Continuing Calibration Check Standard
58	Hexane Blank w/surrogates
59-68	Samples analyses, including method blanks, matrix spikes, matrix duplicates, matrix spike duplicates, and QC reference check standard. A maximum of nine samples between continuing calibration check standards
69	Continuing calibration check standard
70	Hexane Blank w/surrogates
71	Repeat injections 59-68 sequence

13.10. PCB Aroclor Qualitative Identification and Secondary GC Column Confirmation:

13.10.1. Positive identification of PCB Aroclors is based on comparison of retention time of the five selected quantitation peaks and major non-quantitation peaks for the unknown sample with retention time of reference standards (continuing calibration verification standards). Additionally pattern recognition is used for comparison of unknown samples with reference standards for positive identification. Confirmation of Aroclor presence by secondary GC column analysis may be necessary for highly altered/degraded PCB patterns or for programs including PCB air monitoring, US-EPA CLP protocol and other projects as specified in the site sampling and analysis quality assurance plan.

13.10.2. In cases where multiple Aroclors are present with overlapping chromatographic patterns or interferences are encountered that are not removed with extract cleanup processes one or two quantitation peaks may be dropped and not used for quantitation. A minimum of 3 quantitation peaks must be used for

all unknown samples and standards. When quantitation peaks are dropped for a sample or standard the corresponding peaks are also dropped in the initial calibration sequence for calculation purposes.

13.10.3. Dual Column/Confirmatory Column Analysis by GC- Inject samples under same operating conditions and analytical run QA/QC parameters on a secondary GC column of dissimilar phase (e.g., ZB-1 and ZB-5). Note: If using dual GC column system, samples are injected sequentially through separate injection ports onto both columns. Samples are analyzed and concentration results are reported.

13.10.4. Dual Column/Confirmatory Column Laboratory Default by SW-846:

13.10.3.1. Report lowest concentration of the 2 column results for each individual Aroclor on the merged EDD, Form 1 or Certificate of Analysis (Note: This is appropriate for Aroclor regulated projects).

13.10.3.2. If **RPD percent** exceeds 40% report the lowest concentration result of the two analyses unless observed chromatographic interference or instrumental analysis QA/QC indicates the higher value may be more accurate. P-flag all excursions > 40% and describe interferences or rationale for reporting lower value in Data Narrative.

13.10.3.3. If a concentration is above the PQL on one column and below the PQL on the second column, the qualitative presence is not confirmed and the sample is reported as not detected. **Note: If reporting to the MDL is required do the following:** For reporting to the MDL: a) If one result is greater than the PQL and other result is < PQL (J-flag) Report the **highest** result as confirmed (*unless interference or QC reasons indicate lower value*); b) If one result is above MDL (J-Flag) and second is Not Detected report the concentration as **not detected**. (Presence not confirmed); c) If both results are J-Flag values (< PQL) report the lowest value of the two.

13.10.5. **USEPA-CLP/ASP Program Protocols:**

13.10.4.1. Report **Lowest** Value of the 2 column results for each individual Aroclor on the merged EDD, Form 1 or Certificate of Analysis (Note: This is appropriate for Aroclor regulated projects. E.g. Air Monitoring for EPA TO-10A alternative reporting may be based upon total PCB values for PCB- Total regulated projects).

13.10.4.2. If **Percent Difference** (not RPD%) exceeds 25% then P-flag all excursions > 25%. Note any chromatographic interferences present in Case Narrative.

13.10.4.3. If one result is greater than PQL and other result is < PQL (J-Flag) Report the **lowest** result (J-Flagged) value (*confirmed hit*).

13.10.4.4. If one result is above MDL (J-Flag) and second is Not detected, report the concentration as **not detected** (*presence not confirmed*).

14. Data Analysis and Calculations

14.1. PCB Solution concentration calculation from initial Calibration by Linear Regression:

$$Y_i = aX_i + b$$

X_i = Calibrated Solution Concentration (ng/mL)
Y_i = total area response of 5 PCB quant. peaks (uV-Sec.)
a = slope
b = intercept

Note: In those instances where samples may be quantitated with 3-4 peaks due to interference or overlap, the Empower system automatically quantitates against the calibration using only the area of the selected peaks.

Unknown Solution Conc. X = (Y –b) a

Y = Total area response of PCB Chromatogram (uV-Sec.)
a = slope of ICAL by linear regression
b = intercept of ICAL by linear regression

14.2. Capillary GC: Sample calculations:

14.2.1. The concentration of each identified PCB Aroclor in a sample will be calculated based on the sample weight or volume.

14.2.2. The PCB solution concentration of the extract is calculated as follows:

Solution Conc. = (Y –b)/a

Y = Total area response of PCB Chromatogram (uV-Sec.)
a = slope of ICAL by linear regression
b = intercept of ICAL by linear regression

14.3. Final concentration of samples- Calculations of final PCB concentrations will vary upon matrix, calculations are as follows:

14.3.1. Soil/Sediment/Solids:

Final Conc. = (Sol. Conc.) * (V)*DF/ (M)* (%Total Solids) (1/1000) ug/g

Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

14.3.2. **Water:**

Final Conc.= (Sol. Conc.) * V*DF/[(Vt)](1/1000) ug/L

Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
Vt= Total Volume Extraction (L)

14.3.3. Biota Tissue :

Final Conc. = (Sol. Conc.) * (V)*DF/(M)(1/1000) ug/g

Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

14.3.4. Waste Oil:

Final Conc. = (Sol. Conc.) * (V)*DF/ (M)*(%Total Solids) (1/1000) ug/g

Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

14.4. The calculated concentration for each PCB Aroclor will be compared to its respective sample-specific reporting limit (RL) and method detection limit (MDL). The results with concentrations at or above the MDL but below RL will be reported as detects and flagged as estimated J. The results for peaks with concentrations at or above the RL would be reported as unqualified numeric values.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. The GC analyst is responsible for generating the data and also is the initial individual to review the data. This would include inspection of the chromatographic data, processing the raw data, producing all required data forms, inspection of calibration curves for compliance, surrogate recovery, laboratory control spike recovery, matrix spike/matrix spike duplicate recovery, and continuing calibration compliance.

15.2. Once the initial review of the data is performed by the analyst, decisions are made at that time to accept the data if all criteria are met or to reject sample data if any of the quality control parameters or limits are out of control. Depending on the situation, samples requiring re-extraction will be notified to the appropriate extraction personnel, sample extracts requiring re-injection will be queued for analysis, new calibrations may have to be performed, or samples re-analyzed due to failing continuing check standards.

15.3. The analyst may also consult with the Quality Manager as to the best form of action to take or if the situation warrants corrective action beyond routine practices. If no recourse is available and the data is to be reported out of criteria, a Case Narrative Report is generated and the deviation is documented and reported to the client. The Case Narrative Report is filed with the data and is also useful for production of case narratives that are issued with the final data reports. If a problem exists that requires follow-up to rectify, a LabTrack Ticket (LTT) is issued to document the problem found, steps taken to resolve the problem, and what samples were affected. This LTT is reviewed by the Quality Manager and lab management to verify that appropriate actions have been taken to correct the problem.

15.4. Please see Table 19.1 below for specific Quality Assurance Acceptance Criteria.

16. Corrective Actions for Out-of-Control Data

16.1. The table below outlines the data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	The five point calibration is analyzed initially for all Aroclors and when Continuing Calibration Check standard fail criteria.	- %RSD≤20% for the relative response factors for the calibration standards if using average response factor calibration. Correlation Coefficient R must be >0.990 for Linear Regression.	- Re-analyze the initial calibration standard and/or evaluate/correct instrument malfunction to obtain initial calibration and continuing calibration check standards that meet criteria.
Continuing Calibration Check Standard (CCCS)	- Initially analyze a CCCS immediately following an initial calibration. - After the initial CCCS of the sequence, a CCCS must be analyzed after 10 samples. - Analytical sequence must end with analysis of a CCCS. - CCCS must be analyzed at least once per 12 hour analytical shift. - CCCSs are rotated through all Aroclors in order.	- Calibration factor for the continuing calibration check must ±20% of the true value. - Retention time of all quantitated peaks must be within RT window (reset with each initial CCCS of a sequence). - All samples must be bracketed by a CCCS that meet all criteria stated above.	- If the reason for the failure of the CCCS appears to be a poor injection (or a degraded standard solution), the CCCS will be re-injected (or re-prepared and re-injected) immediately following the failed CCCS. This can only occur if the instrument is being attended by an analyst. If upon re-injection, the CCCS meets all the acceptance criteria and there is no apparent impact on the sample data the analytical sequence will continue and samples will not be reanalyzed. The associated sample data will be reported. - If CCCS failure was not due to a poor injection (or degraded standard solution) or the instrument was unattended at the time of the CCCS failure, correct system, if necessary, and recalibrate. Initial calibration and CCS criteria must be met before sample analysis may begin. Samples that are not bracketed by complaint CCCSs must be reanalyzed. -If acceptable CCCSs are observed later in the sequence, samples bracketed by acceptable CCCSs will be reported. Samples between the failed CCCS and prior/ subsequent complaint CCCS will be re-analyzed.

-Retention Time (RT)	- Use the retention time for peak in the CCSs to determine midpoint of the relative retention time window for the analysis sequence. -Each sample analysis: Rely on RT windows to identify PCB Aroclor to report. Also use pattern recognition and professional judgment for peaks that shift from RT windows, because compound composition may shift RT for GC peaks.	- Each quantitated peak and surrogate peak should be within established windows.	-Inspect chromatographic system for malfunction, correct problem. Perform re-analysis if necessary.
Method Blank	-One per extraction batch of ≤20 samples of the same matrix per day. -Should be analyzed with other associated batch QC samples on the same instrument, but not all samples. -Must undergo all sample preparative procedures.	- Concentration does not exceed the RL/MDL for any PCB Aroclor. - Must meet in house surrogate criteria of TCMX 47-123% and DCBP 35-153%.	- Re-analyze method blank to determine if instrument contamination was the cause. If method blank re-analysis passes, then report samples. -If method blank is found to contain PCB contamination above the RL/MDL for any PCB Aroclor compound , then re-extract and re-analyze all associated samples. -If no sample is available for re-extraction, report data B flagged to indicate method blank contamination.
Laboratory Control Spike (LCS)	- One per extraction batch of ≤20 samples per matrix per day. - Should be analyzed with other associated batch QC samples on the same instrument, but not all samples.	-Percent recovery must be within method limits. - Must meet Aroclor spike criteria of 70-130% recovery - Must meet in house surrogate criteria of TCMX 47-123% and DCBP 35-153%..	-Re-analyze LCS to determine if instrument was the cause. If LCS passes, then report samples. -If LCS recovery is still out of limits, the re-extract and re-analyze all associated samples. -If no sample is available for re-extraction, report data flagged to indicate LCS failed recovery.

Matrix Spike/Matrix Spike Duplicate (MS/MSD)	-One MS/MSD per extraction batch of ≤20 samples per matrix . - A MS and a sample/sample duplicate may be appropriate in place of MS/MSD matrices where detectable amounts of analytes are present.	- Percent recovery for MS must be within method limits - If MS/MSD is analyzed, relative percent difference (RPD) should be within 30%. - Must meet Aroclor spike criteria of 70-130% recovery - Must meet in house surrogate criteria of TCMX 47-123% and DCBP 35-153%. (unless original unspiked sample is also outside of criteria)	-Re-analyze MS and/or MSD to determine if instrument was the cause. If MS and/or MSD pass, then report samples. -Check for errors such as calculations and spike preparation. -Check original unspiked sample results and surrogate recovery for indications of matrix effects. -If no errors are found, and the associated LCS is within limits, then sample matrix effects are likely the cause. Note exceedance in case narrative.
Surrogates	-Calibrated as target compound in the Aroclor A1254 standards. -Surrogates are added to all calibration check standards, blanks (including instrument blanks run after CCCS), samples and QC samples.	- Must meet in house surrogate criteria of TCMX 47-123% and DCBP 35-153%.	-Re-analyze the affected sample or QC sample to determine if instrument was the cause. If surrogate passes, then report samples. -Check for errors in surrogate calculation and surrogate solutions. -If no problem is found, then re-extract and re-analyze the sample. -If re-extraction is within limits and sample extract holding time, then report only the re-analysis. -If the re-extraction is within limits, but out of extraction holding time, then report both sets of data. -If the re-extraction produces surrogate recovery still out of limits, then report both sets of data. -If no sample exists for re-extraction, report data flagged to indicate surrogate failed recovery or have a client re-sample.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Data that is detected to be out-of-control for any reason, when compared to method acceptance criteria, will addressed in the following manner:

17.1.1. If the problem exists with the gas chromatographic instrumentation, appropriate action will be taken to repair and perform maintenance to bring the instrument back to operation condition. Once the instrumentation is determined to be correctly operating analysis can begin again.

17.1.2. If the problem exists with calibration standard solutions, the analyst will prepare new standards and discard the standard solutions that are suspect. Instrument calibration can be performed and analysis can begin once system is control.

17.1.3. If the problem exists with sample extraction and extract preparation, the extraction step that is producing the out of-control situation will be diagnosed and rectified. Once the troubleshooting procedures correct the problem extraction can once again occur and analysis can continue.

17.1.4. In situations where data is reported under out-of-control conditions, the data will be annotated with data qualifiers and/or appropriate descriptive comments defining the nature of the excursion in the sample case narrative. If warranted, a LabTrack Ticket will be issued to define the problem, steps to correct the problem, and final resolution.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. Initial Demonstration of Capability (IDOC) Procedure:

18.2.1. Prepare 4 replicates of a fortified laboratory blank sample (using laboratory reagent water or sodium sulfate) by spiking each sample with 1.0mL of 0.500ug/mL Aroclor solution for water samples and 0.100mL of 100ug/mL Aroclor 1242 solution for solid samples. The Aroclor type used for spiking should be rotated on a yearly basis. Prepare one method blank sample with the batch.

18.2.2. For each replicate the recovery value of the sample must fall in the range of 70-130% (or established lab limits) and the percent RSD must be <20 % for the method performance to be considered acceptable.

18.2.3. This procedure must be repeated using four fresh samples until satisfactory performance has been demonstrated. The initial demonstration of capability is used primarily to preclude the laboratory from analyzing unknown samples via a new, unfamiliar method prior to obtaining some experience with it. It is expected that as laboratory personnel gain experience with this method the quality of data will improve beyond those required here.

18.3. Continuing Demonstration of Performance Procedure:

18.3.1. Annual continuing demonstration of performance may be satisfied by a repeat Initial Demonstration of Performance, the acceptable analysis of an unknown samples (for example PT test sample), or the acceptable analysis of 4 consecutive Laboratory Control Spike samples. Records of continuing demonstration of performance are maintained by the laboratory Quality Assurance Department.

18.3.2. With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples an organic-free reagent water blank is processed.

18.3.3. The method blank should exhibit PCB levels less than the practical quantification limit or reporting limit (PQL or RL). If the method blank exhibits PCB contamination above the reportable quantitation limit, the samples associated with the contaminated blank should be re-extracted and analysis repeated when appropriate. If there is no original sample available for re-extraction or if the associated sample concentrations greatly exceed the blank concentration, then all positive concentration results for the associated samples should be flagged with a "B" indicating blank contamination and a case narrative describing the situation prepared.

18.3.4. A matrix spike/ matrix spike duplicate is to be analyzed at a rate of 1 matrix spike/ matrix spike duplicate per every 20 samples. A duplicate sample may be prepared in lieu of a matrix spike duplicate in place of a matrix spike duplicate when detectable PCB concentrations are known to be present.

18.4. Method Detection Limit:

18.4.1. A method detection limit will be determined for this method whenever major modification to the extraction or analysis procedures are made or at a minimum frequency of every 2 years. A minimum of

seven laboratory organic free water samples or sodium sulfate will be prepared and spiked with chlorinated PCB methyl esters mixture, at a low level and taken through all extraction and analytical procedures.

$$MDL = S * t_{(n-1,\ 1-alpha=0.99)}$$

S = Standard deviation of the replicate analyses
n = Number of replicates
$t_{(n-1,\ 1-alpha=0.99)}$ = Student's t value for the 99% confidence level with n-1
For example: t for 8 replicates = $t_{(7,0.99)}$ = 2.998

18.4.2. The determined MDL must be less than the concentration spiked but greater than one tenth (1/10) the spiked concentration. If not, repeat the MDL determination at an appropriate spike concentration for affected analytes.

19. Method Modifications

19.1. SW-846 EPA Method 8082A specifies to use Aroclors 1016 and 1260 for matrix spike samples unless specified by the client. The laboratory uses Aroclor 1242 as the default spike analyte due to historic contamination in the area coming from this Aroclor.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Safety glasses and disposable gloves must be worn when handling samples and extracts.

22.2. All manipulations of sample extracts should be conducted inside a chemical fume hood. Manipulation of sample extracts outside of a fume hood should be minimized by the analyst.

22.3. Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize themselves with the precautions for handling solvents and chemicals used to process samples. The analyst should refer to the laboratory chemical hygiene plan for further safety information.

22.4. Samples remaining after analysis should either be returned to the customer for disposal or disposed of through the laboratory's disposal plan. Refer to the sample custodian for assistance and also SOP S-NY-O-054, disposal of laboratory waste.

23. Waste Management

23.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste generated during the execution of this method.

23.2. Please refer to SOP S-NY-W-054 regarding how hazardous waste is handled and disposed of by the laboratory.

24. Pollution Prevention

24.1. Pollution prevention is practiced in the laboratory by minimizing usage of solvents and chemicals, so that disposal of waste generated is held to the smallest amount possible. This is directly linked to the types of extraction procedures in place at the laboratory to reduce the volumes of solvents used for semi-volatile extraction procedures. Pace Analytical Services, Inc. employs extraction procedures such as continuous liquid/liquid and solid phase extraction methods to reduce solvent requirements for water extraction protocols and ASE and Soxhlet extractions for solid matrices.

24.2. Pollution prevention also relies on minimizing to the best extent the chemicals and solvents required to perform extraction and analysis procedures. The laboratory personnel strive to purchase chemicals and standards that will be consumed based on anticipated workload. For additional information about laboratory pollution prevention, please refer to laboratory SOP S-NY-S-168.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, “Quality Systems”- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version.

25.4. U.S. EPA SW-846 Method 8082A “Test Methods for Evaluating Solid waste; Volume 1B Laboratory Manual Physical/Chemical Methods”, Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

25.5. U.S. EPA 40 CFR Part 136, “Guidelines Establishing Test Procedures of the Analysis of Pollutants”, July, 1988.

25.6. Standard Methods for the Examination of Water and Waste Water”, 19th Edition 1995, American Public Health Association, American Water Works Association, Water Pollution Control Federation.

25.7. New York State Department of Health, “Environmental Laboratory Approval Program Certification Manual”, Wadsworth Center for laboratories and Research, 1996.

25.8. Guide to Environmental Analytical Methods”, third edition, Genium Publishing Corporation, 1997.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: PCB Stock Standard/Calibration Standard Preparation.

26.2. Attachment II: Continuing Calibration Check Standard Preparation.

26.3. Attachment III: GC Operating Parameters.

26.4. Attachment IV: Chromatograms.

27. Revisions

Document Number	Reason for Change	Date
S-NY-O-314-rev.01	General: converted to new format. General: removed all references to air matrix (PUF). Sections 10.1-10.4: updated standards used and instructions for working standard creation Sections 11.2.5 and 13.6.2: added Section 12.1: revised to match current SOP list. Sections 13.6.3 and 16.1: updated surrogate recovery acceptance limits Section 16.1: specified that surrogate calibration is done with Aroclor 1254 Sections 25.1-25.3: added standard Pace references. Section 26.1: updated attachement	12Feb2015
S-NY-O-314-rev.02	Sections 13.10.2 and 14.1: added documentation regarding quantitation with 3-4 peaks	30March2015
S-NY-O-314-rev.03	Section 5.2: changed to reference 40 CFR MDL procedure Section 12.3.2: removed qualifiers and referred to Report Definitions Page controlled document form Sections 15.3 and 17.1.4: updated to reference LabTrack system Section 19.1: added documentation of modification for use of Aroclor 1242 as default spiking analyte	10June2015

Attachment I: PCB Stock Standard/Calibration Standard Preparation

Table 1: PCB Stock Standard Preparation Table

PCB Stock Standards	**Init Volume (mL)**	**Final volume (mL)**	**Conc. (ppm)**
A1016	5.0	50	10.0
A1221	5.0	50	10.0
A1232	5.0	50	10.0
A1242	5.0	50	10.0
A1248	5.0	50	10.0
A1254	5.0	50	10.0
A1260	5.0	50	10.0
A1262	1.0	100	10.0
A1268	1.0	100	10.0

Unless otherwise noted hexane is the solution used to make all dilutions. *Custom Order

Attachment I: PCB Stock Standard/Calibration Standard Preparation (continued)

Table 2: PCB Calibration Standard Preparation Table (High Level Calibration Curve)

Initial Volume (mL)	Initial Conc. (ug/mL)	Final Volume (mL)	Final Concentration (mg/L)							
			A1016	A1221	A1232	A1242	A1248	A1260	A1262	A1268
5.0	10.0	50.0	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
2.5	10.0	50.0	0.500	0.500	0.500	0.500	0.500	0.500	0.500	0.500
1.25	10.0	50.0	0.250	0.250	0.250	0.250	0.250	0.250	0.250	0.250
0.500	10.0	50.0	0.100	0.100	0.100	0.100	0.100	0.100	0.100	0.100
5.0	1.00	50.0	0.020	0.020	0.020	0.020	0.020	0.020	0.020	0.020

See Table 3 for A1254 Standard Preparation (high level)

Table 2A: PCB Calibration Standard Preparation Table (Low Level Calibration Curve)

Init. Volume (mL)	Initial Conc. (ug/ml)	Final Volume (mL)	Final Concentration (ppm)							
			A1016	A1221	A1232	A1242	A1248	A1260	A1262	A1268
0.5	10.0	50.0	0.100	0.100	0.100	0.100	0.100	0.100	0.100	0.100
2.5	1.0	50.0	0.050	0.050	0.050	0.050	0.050	0.050	0.050	0.050
1.0	1.0	50.0	0.020	0.020	0.020	0.020	0.020	0.020	0.020	0.020
1.0	0.500	50.0	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010
0.50	0.500	50.0	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005

See Table 3A for A1254 Standard Preparation (low level)

Attachment I: PCB Stock Standard/Calibration Standard Preparation (continued)

Table 3: PCB A1254 Calibration Standard Preparation Table (for High Level Curve)

Initial Volume (mL) A1254	Initial Conc. (ug/mL) A1254	Initial Volume (mL) 0.5/5.0 -ppm Surrogate	Final Volume (mL)	Final Concentration (ppm)		
				A1254	TCMX	DCBP
5.0	10.0	0	50	1.000	0	0
2.5	10.0	0	50	0.500	0	0
5.0	10.0	2.00	50	1.000	0.020	0.200
2.5	10.0	1.00	50	0.500	0.010	0.100
1.25	10.0	0.800	50	0.250	0.008	0.080
0.500	10.0	0.500	50	0.100	0.005	0.050
1.00*	1.00	0.200	50	0.020	0.002	0.020

*This initial volume is of the A1254 1.00ug/mL secondary stock solution WITHOUT surrogates

Table 3A: PCB A1254, TCMX and DCBP Calibration Standard Preparation Table (for Low Level Curve)

Initial Volume A1254 (mL)	Initial Conc. A1254 (ug/mL)	Initial Volume (mL) 0.5/5.0 -ppm Surrogate	Final Volume (mL)	Final Concentration (ppm)		
				A1254	TCMX	DCBP
0.5	10.0	0.800	50	0.100	0.00800	0.0800
2.50	1.000	0.500	50	0.050	0.00500	0.0500
1.0	1.000	0.400	50	0.020	0.00400	0.0400
1.0	0.500	0.250	50	0.010	0.00250	0.0250
0.50	0.500	0.100	50	0.005	0.00100	0.0100

Attachment II: Continuing Calibration Check Standard Preparation

Table 1: PCB Continuing Calibration Working Standards prepared from 1000ug/mL Stock Standards

PCB		Initial Volume (mL)	Final Volume (mL)	Concentration (ppm)
A1016		1.0	100	10.0
A1221		1.0	100	10.0
A1232		1.0	100	10.0
A1242		1.0	100	10.0
A1248		1.0	100	10.0
A1254		1.0	100	10.0
A1260		1.0	100	10.0
A1262		1.0	100	10.0
A1268		1.0	100	10.0

Attachment II: Continuing Calibration Check Standard Preparation (continued)

Table 2: PCB Continuing Calibration Standards (High Level) prepared from 10ug/mL CCV Working Standards and all contain surrogates

PCB	Surr. Volume* (mL)	Initial Volume Aroclor (mL)	Final Volume (mL)	Surrogate Concentration TCMX/DCBP (ppm)	Aroclor Concentration (ppm)
A1016	2.0	5.0	100	0.010/0.100	0.500
A1221	2.0	5.0	100	0.010/0.100	0.500
A1232	2.0	5.0	100	0.010/0.100	0.500
A1242	2.0	5.0	100	0.010/0.100	0.500
A1248	2.0	5.0	100	0.010/0.100	0.500
A1254	2.0	5.0	100	0.010/0.100	0.500
A1260	2.0	5.0	100	0.010/0.100	0.500
A1262	2.0	5.0	100	0.010/0.100	0.500
A1268	2.0	5.0	100	0.010/0.100	0.500

*Surrogate stock solution 0.500ug/mL TCMX and 5.0ug/mL DCBP

Attachment II: Continuing Calibration Check Standard Preparation (continued)

Table 3: PCB Continuing Calibration Standards (low Level) prepared from 10.0ug/mL CCV Working Standards and all contain surrogates.

PCB	Surr. Volume* (mL)	Initial Volume Aroclor (mL)	Final Volume (mL)	Surrogate Concentration TCMX/DCBP (ppm)	Aroclor Concentration (ppm)
A1016	1.0	0.500	100	0.005/0.050	0.050
A1221	1.0	0.500	100	0.005/0.050	0.050
A1232	1.0	0.500	100	0.005/0.050	0.050
A1242	1.0	0.500	100	0.005/0.050	0.050
A1248	1.0	0.500	100	0.005/0.050	0.050
A1254	1.0	0.500	100	0.005/0.050	0.050
A1260	1.0	0.500	100	0.005/0.050	0.050
A1262	1.0	0.500	100	0.005/0.050	0.050
A1268	1.0	0.500	100	0.005/0.050	0.050

*Surrogate stock solution 0.500ug/mL TCMX and 5.0ug/mL DCBP

Attachment III: GC Operating Parameters

GC #: GC-21 8082 High Level Method(GEHR inclusive, parameters)
Method: Method 2
Column: ZB-1 Front
ZB-5 Middle
Date: 01/18/2012
Analyst: JKA
File Name: S:\Lab Data\PCB\GC Parameters\[GC21_Parameters.xls]8082 M2

Sample Delivery: SEE LEAP PARAMETERS

Column Oven:

Step	Temp (°C)	Rate (°C/min)	Hold (min)	Total (min)
Initial	140	-------	2.00	2.00
2	200	10	0.00	8.00
3	245	5	13.5	30.5

Stabilization Time (min): 0.20

Injector: Front CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	30

Flow/PSI(Front EFC, Type 1):
Carrier Gas: Helium

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	30.0*	-------	20	20

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 5.0

Injector: Middle CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	30

Flow/PSI(Front EFC, Type 1):

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	30.0*	-------	20	20

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 5.0

Detector: **Front ECD**

ECD Oven Power: ON
Temperature (°C) 300
Electronics: ON
Range: 1

Time	Range	Autozero
Initial	1	YES

Front ECD Adjustment
Time Constant: Fast
Cell Current: CAP
Contact Potential (mV): -650 *
Date of last adjustment 12/15/2011

Front ECD Adjustments
Make-up Flow (mL/min) 35 *
*values may change with use

Middle ECD

ECD Oven Power: ON
Temperature (°C) 300
Electronics: ON
Range: 1

Time	Range	Autozero
Initial	1	YES

Fast
CAP
-380 *
12/15/2011

Middle ECD Adjustments
Make-up Flow (mL/min): 35 *

Analog Output

Detectors: Front: ECD Attenuation 1
Middle: ECD Attenuation 1
Rear: None

Attachment III: GC Operating Parameters (continued)

GC-10 8082 High Level Method - Hydrogen

GC #: 10
Method: Hydrogen 8082
GC Method #: 2
Date: 02/09/2012
Analyst: MTH
File Name:
Column: Front ZB-1 MS 20m 0.18 0.18
Middle ZB-5 20m 0.18 0.18

Sample Delivery: SEE LEAP PARAMETERS

Column Oven:

Step	Temp (°C)	Rate (°C/min)	Hold (min)	Total (min)
Initial	150		1.41	1.41
	290	17.5	0.65	10.04

Stabilization Time (min): 0.5

Injector: Front CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	30

Flow/PSI(Front EFC, Type 1):
Carrier Gas Hydrogen

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	30 *		10.00	10.00

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 5.0

Injector: Middle CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	30

Flow/PSI(Front EFC, Type 1):

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	30 *		10.00	10.00

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 5.0

Detector: Front ECD

ECD Oven Power: ON
Temperature (°C) 300
Electronics: ON
Range: 1

Time	Range	Autozero
Initial	1	YES

Middle ECD

ECD Oven Power: **ON**
Temperature (°C) 300
Electronics: ON
Range:

Time	Range	Autozero
Initial	1	YES

Front ECD Adjustment		
Time Constant:	Fast	Fast
Cell Current:	CAP	CAP
Contact Potential (mV):	250 *	365 *
Date of last adjustment	01/23/2012	01/23/2012
Make-Up Flow (ml/min):	35.0	35.0

*values may change with use

Analog Output

Detectors: Front: ECD Attenuation 1
Middle: ECD Attenuation 1
Rear: None

Attachment III: GC Operating Parameters (continued)

Leap GC Pal Parameters					
Sample injection Methods					
Method	**GC Dual**	**GC Duals**		**Method**	**GC Inj s**
Cylcle	GC Dual	GC Dual		**Cylcle**	GC Inj S
Syringe	10uL	10uL		**Syringe**	10uL
1. Sample Vol	1.0uL	1.0uL		**1. Sample Vol**	1.0uL
1. Air Vol	1.0uL	1.0uL		**Solvent Plug**	200nL
1. Inject to	GC Inj 1	GC Inj 1		**Slv Source**	Standard
Inj Time Diff	0s	0s		**Int Standard**	0nL
2. Sample Offs	1	0		**Std Source**	Standard
2. Sample Vol	1.0uL	1.0uL		**Air Gap (s)**	1.0uL
2. Air Vol	1.0uL	1.0uL		**1. Air Vol Ndl**	1.1uL
2. Inject to	GC Inj 2	GC Inj 2		**Pre Cln Slv 1**	2
Pre Cln Slv 1	2	2		**Pre Cln Slv 2**	2
Pre Cln Slv 2	2	2		**Fill Speed**	5.0uL/s
Pre Cln Sp 1	0	0		**Pull Up Delay**	1.0s
Int Cln Slv 1	2	2		**Inject to**	GC Inj 1
Int Cln Slv 2	2	2		**Inject Speed**	5.0uL/s
Pst Cln Slv 1	2	2		**Pre Inj Del**	0ms
Pst Cln Slv 2	2	2		**Pst Inj Del**	0ms
Fill Volume	10uL	10uL		**Pst Cln Slv 1**	2
Fill Speed	2.5uL/s	2.5uL/s		**Pst Cln Slv 2**	2
Fill Stroke	0	0			
Pull Up Delay	500ms	500ms			
Inject Speed	10uL/s	10uL/s			
Pre Inj Del	0ms	0ms			
Pst Inj Del	0ms	0ms			

Attachment IV: Chromatograms (ZB5 Column/Helium Carrier Gas)

FIGURE 1: A1016 at 0.500ug/mL Plot

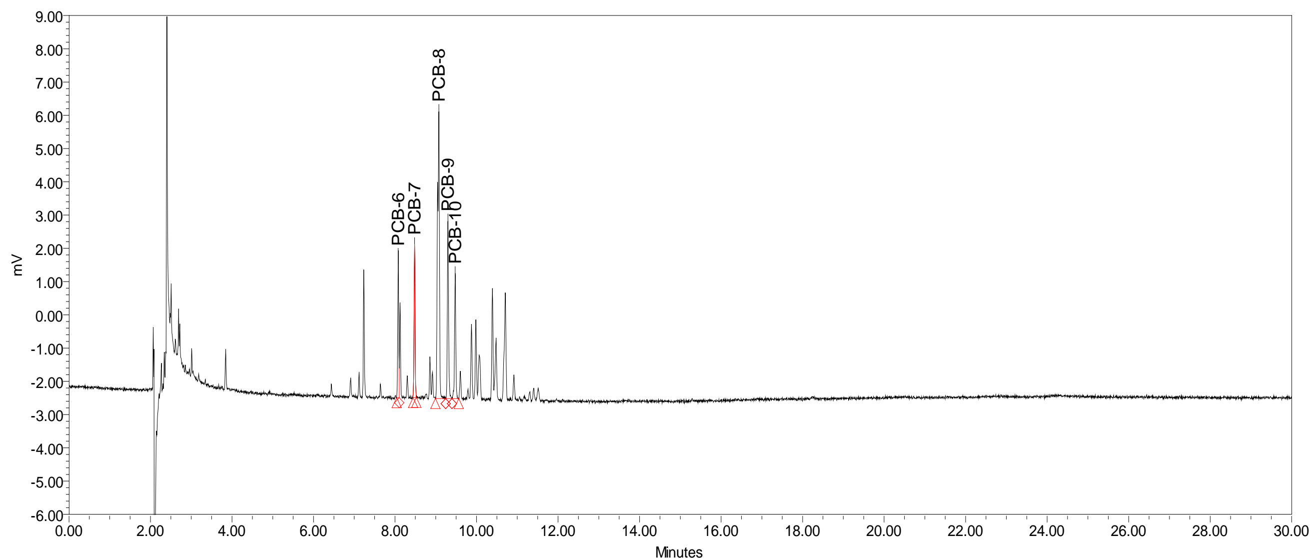
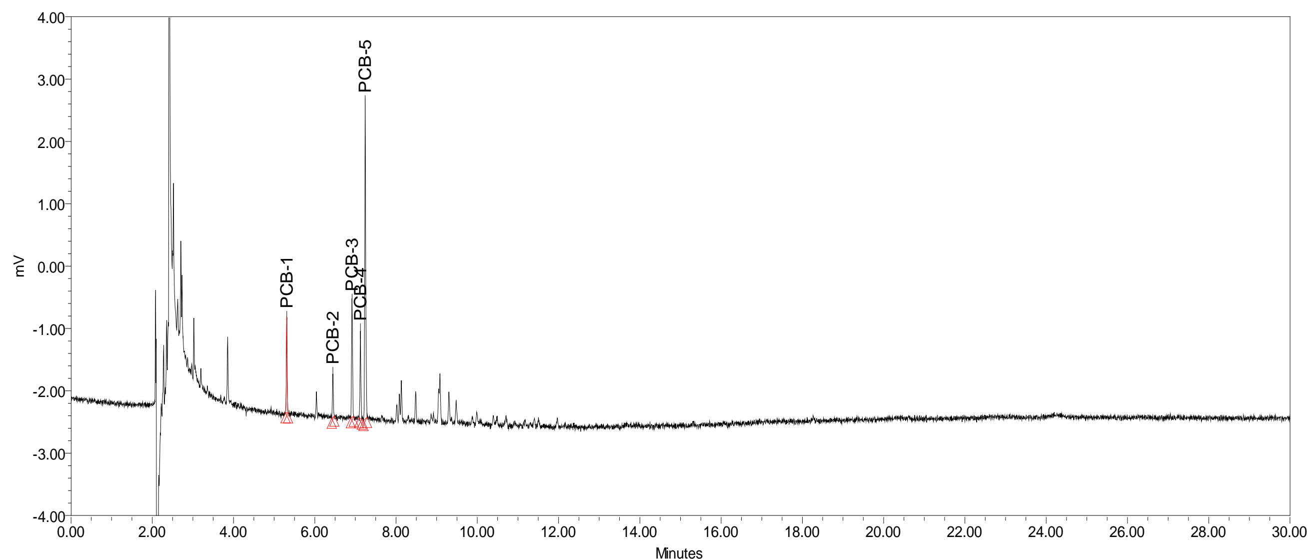


FIGURE 2: A1221 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB5 Column/Helium Carrier Gas) (continued)

FIGURE 3: A1232 at 0.500ug/mL Plot

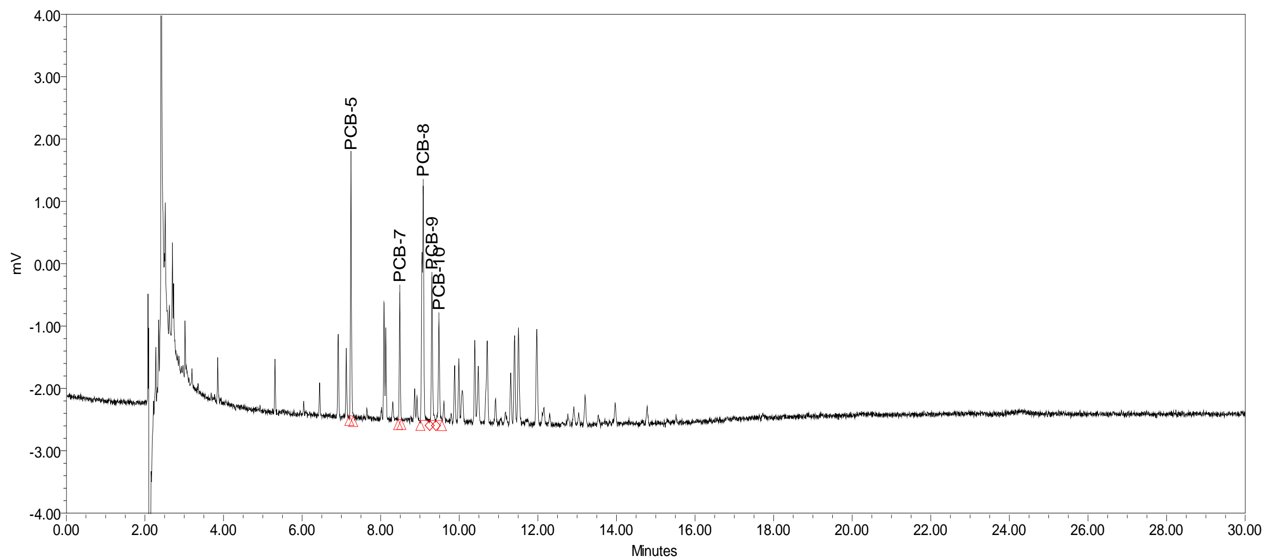
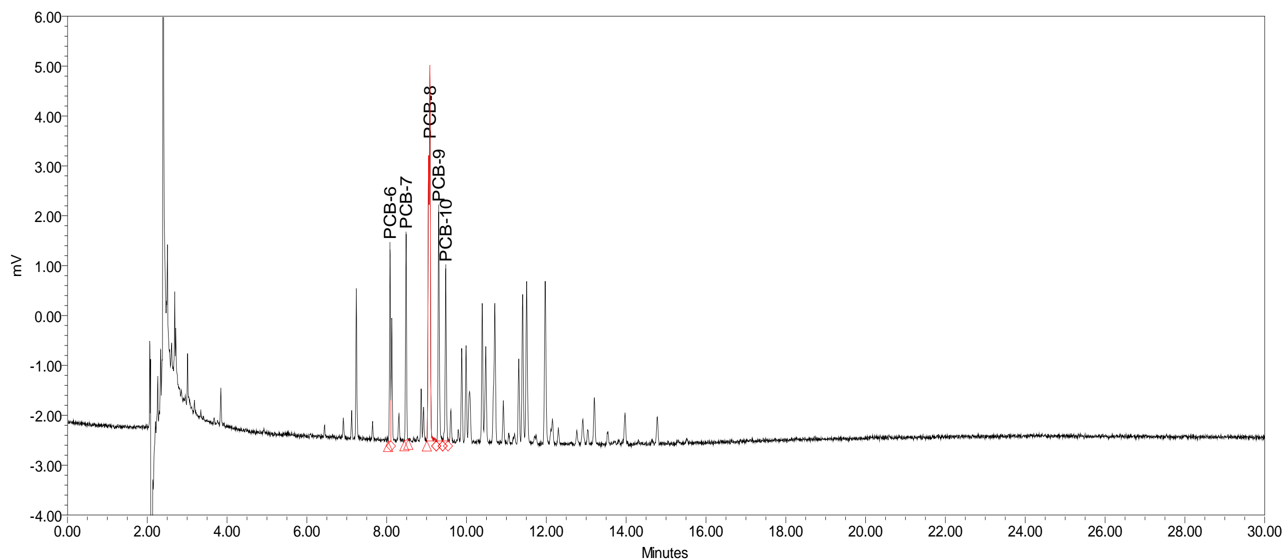


FIGURE 4: A1242 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB5 Column/Helium Carrier Gas) (continued)

FIGURE 5: A1248 at 0.500ug/mL Plot

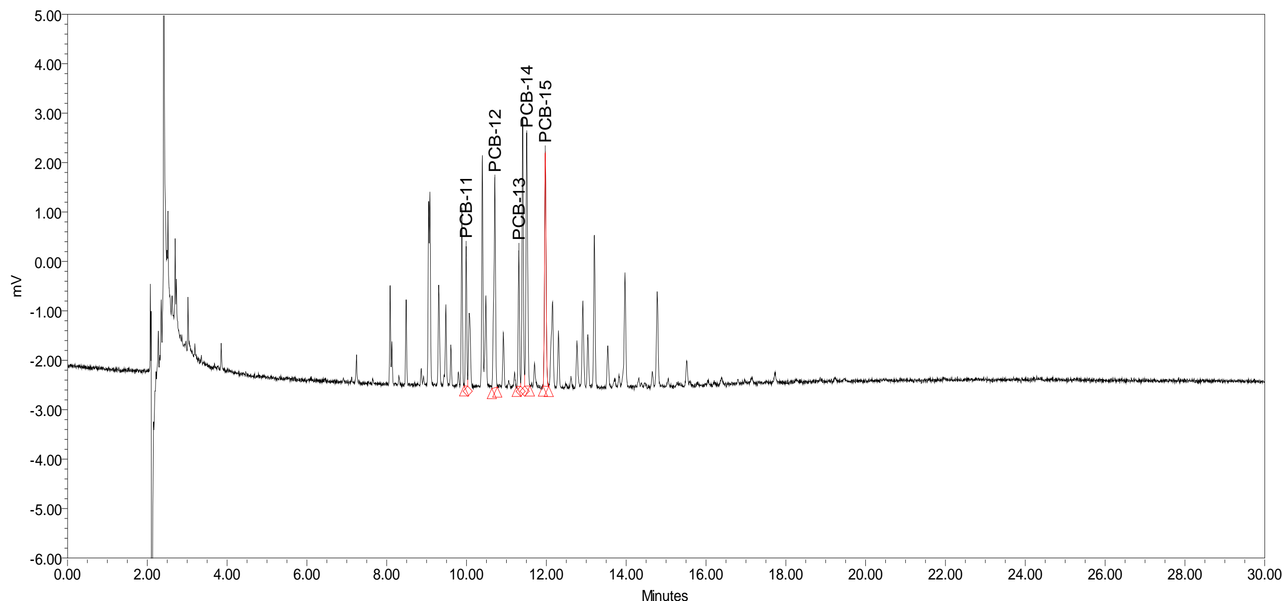
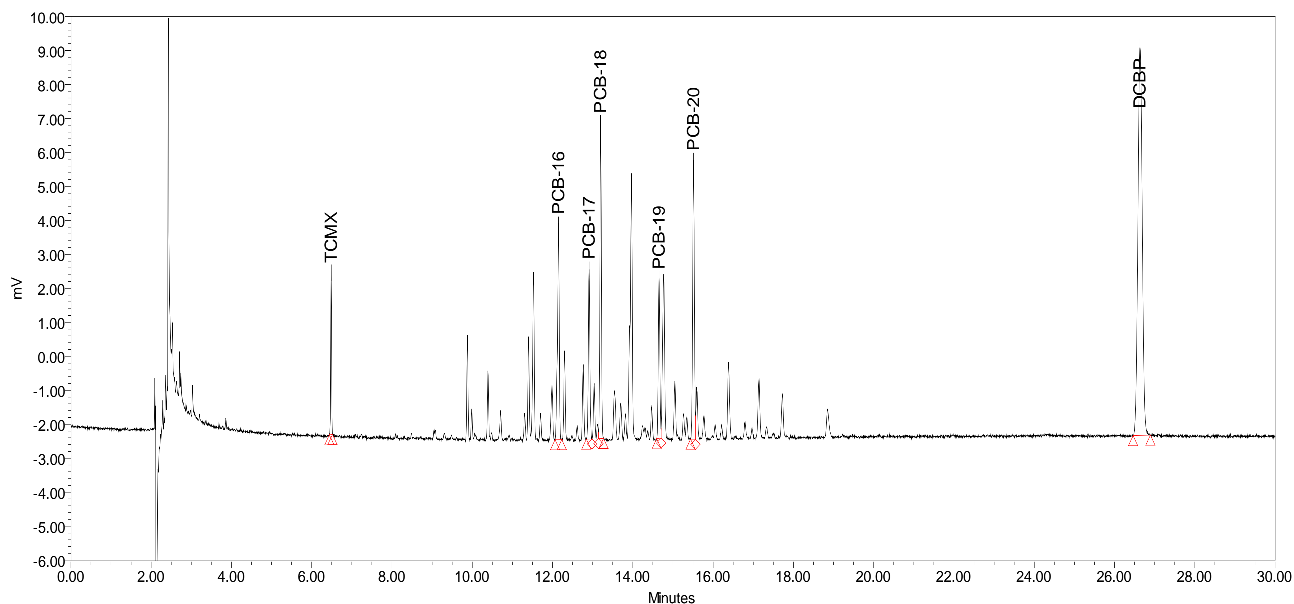


FIGURE 6: A1254 at 0.500ug/mL w/ TCMX & DCBP at 10/100ug/L Plot



Attachment IV: Chromatograms (ZB5 Column/Helium Carrier Gas) (continued)

FIGURE 7: A1260 at 0.500ug/mL Plot

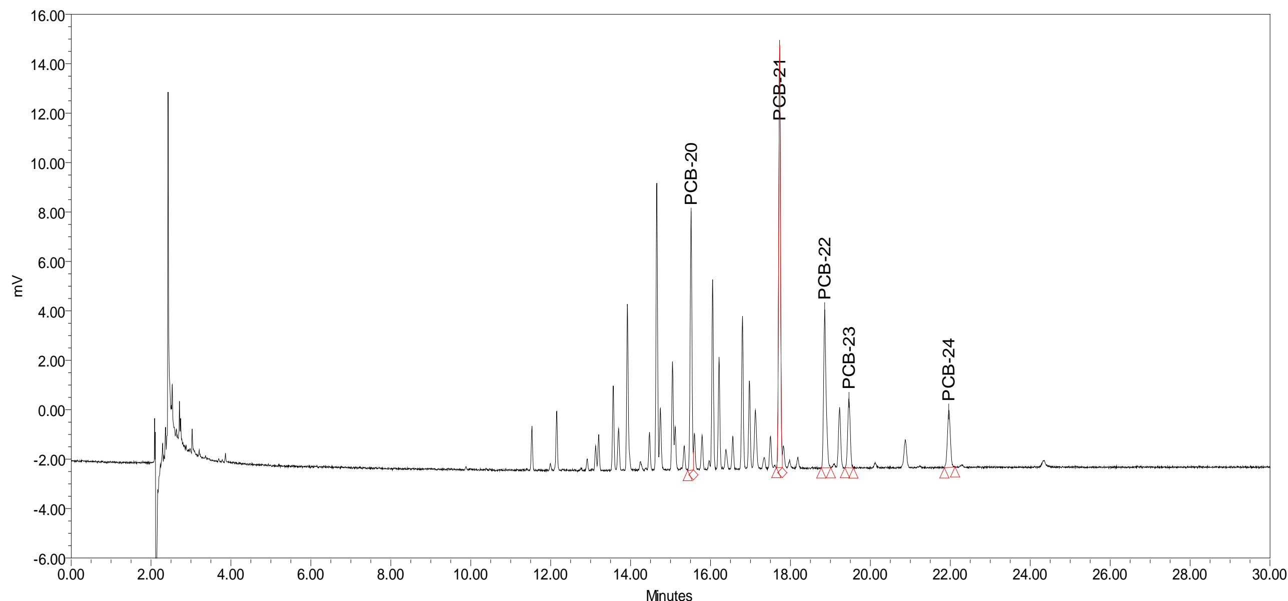
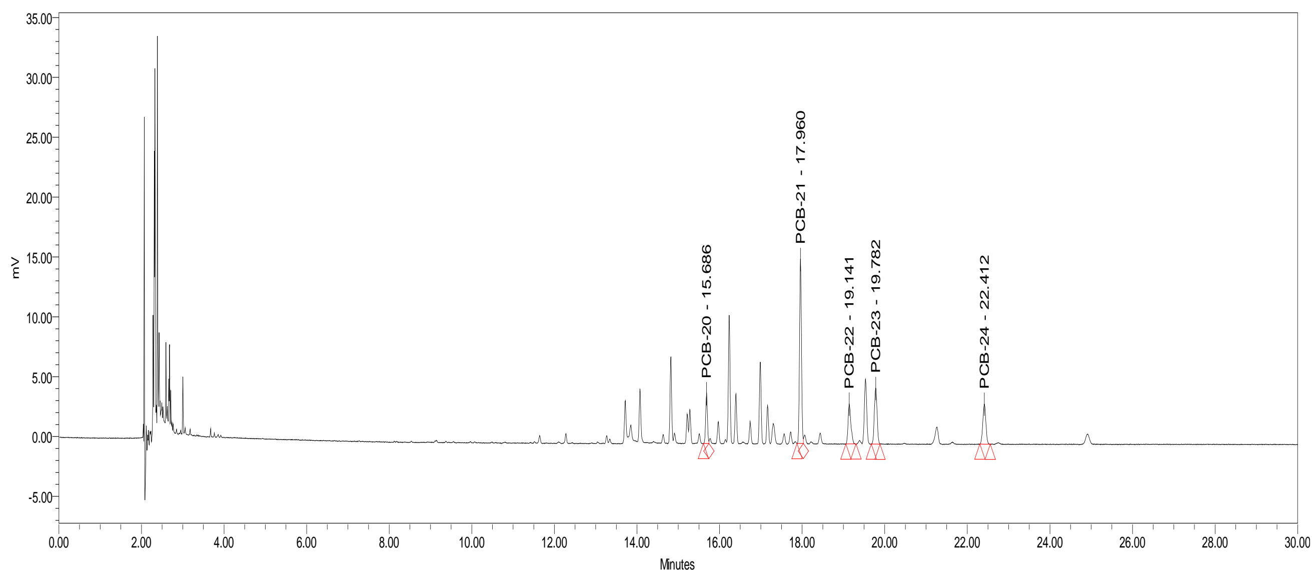
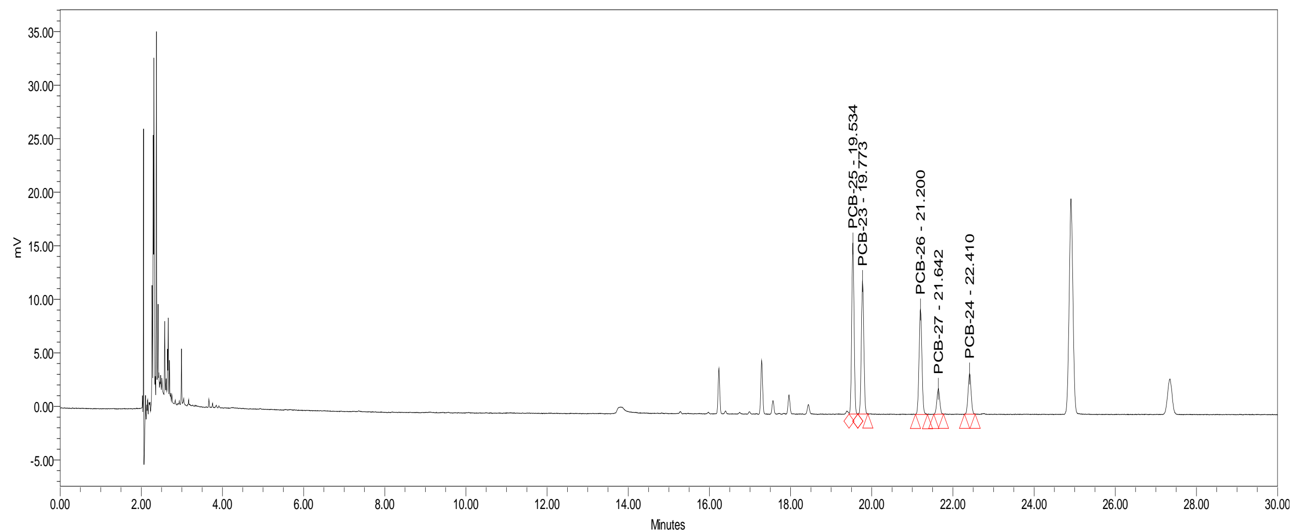


FIGURE 8: A1262 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB5 Column/Helium Carrier Gas) (continued)

FIGURE 9: A1268 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB1 Column/Helium Carrier Gas) (continued)

FIGURE 1: A1016 at 0.500ug/mL Plot

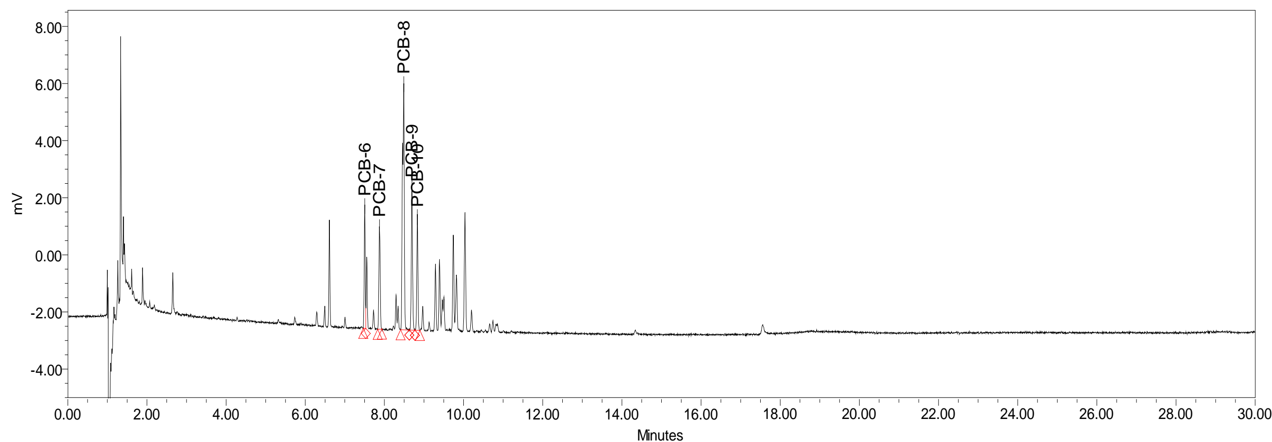
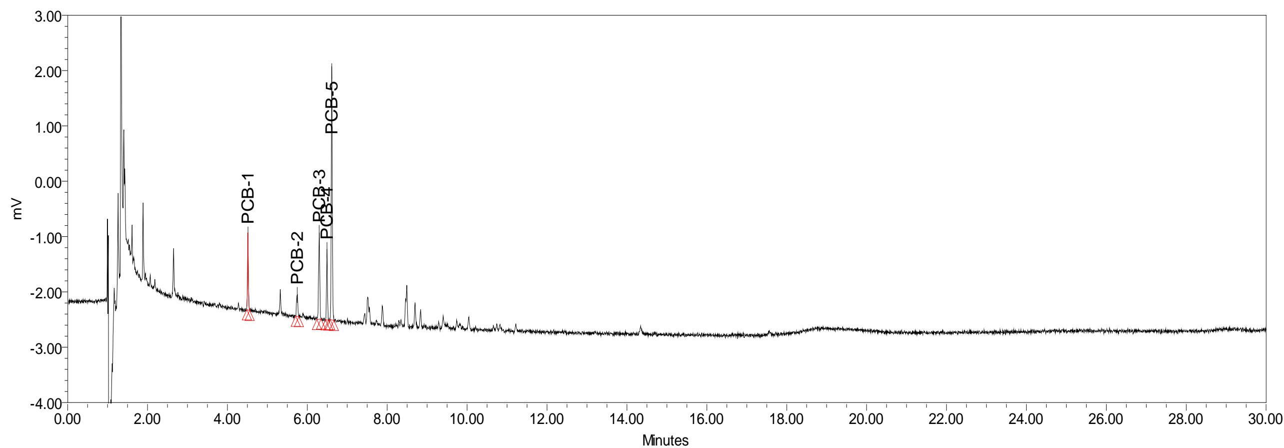


FIGURE 2: A1221 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB1 Column/Helium Carrier Gas) (continued)

FIGURE 3: A1232 at 0.500ug/mL Plot

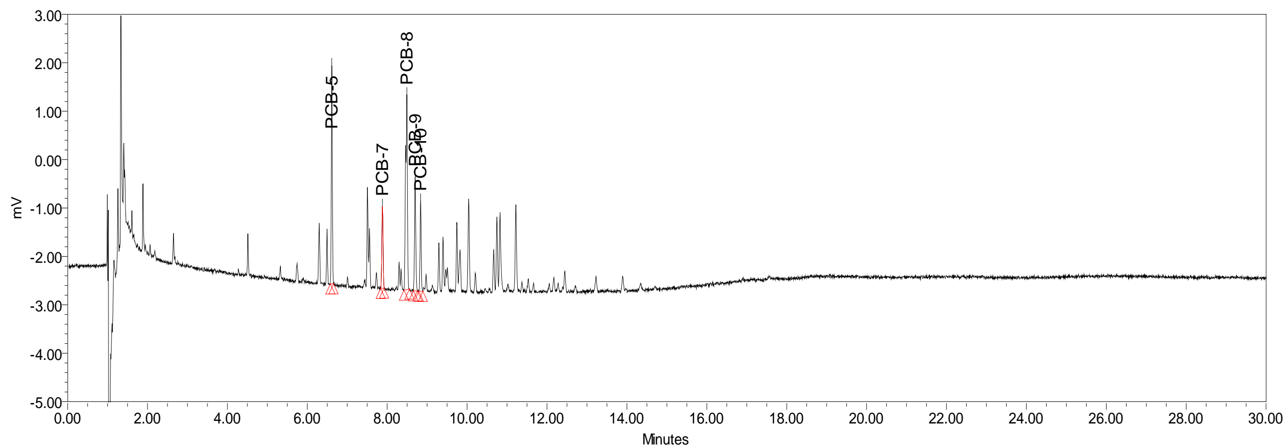
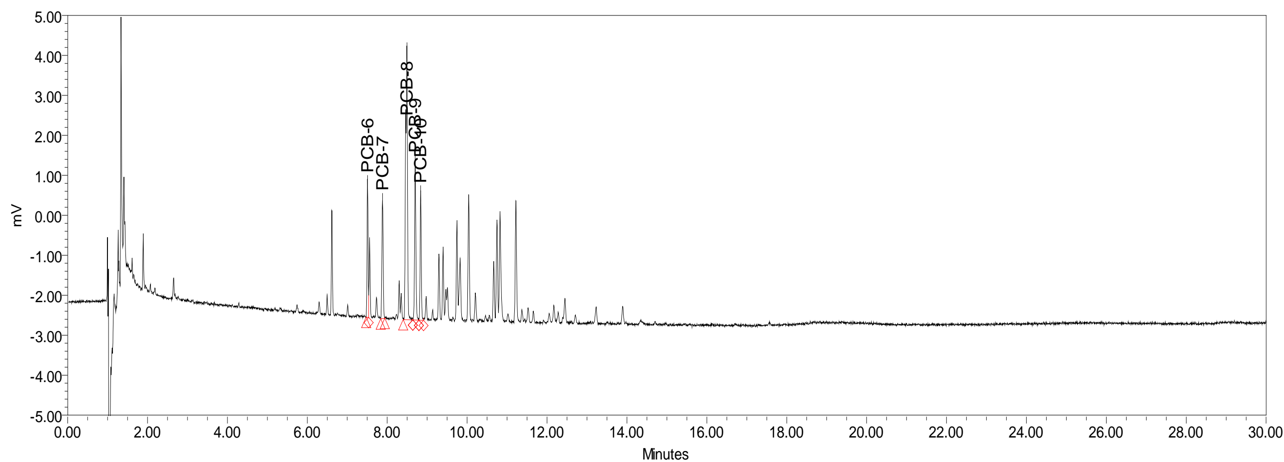


FIGURE 4: A1242 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB1 Column/Helium Carrier Gas) (continued)

FIGURE 5: A1248 at 0.500ug/mL Plot

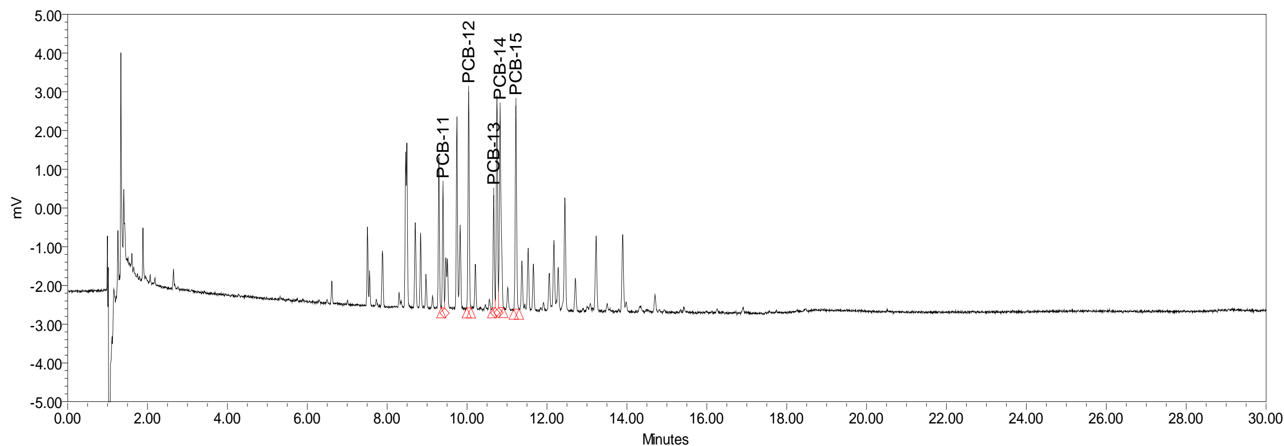
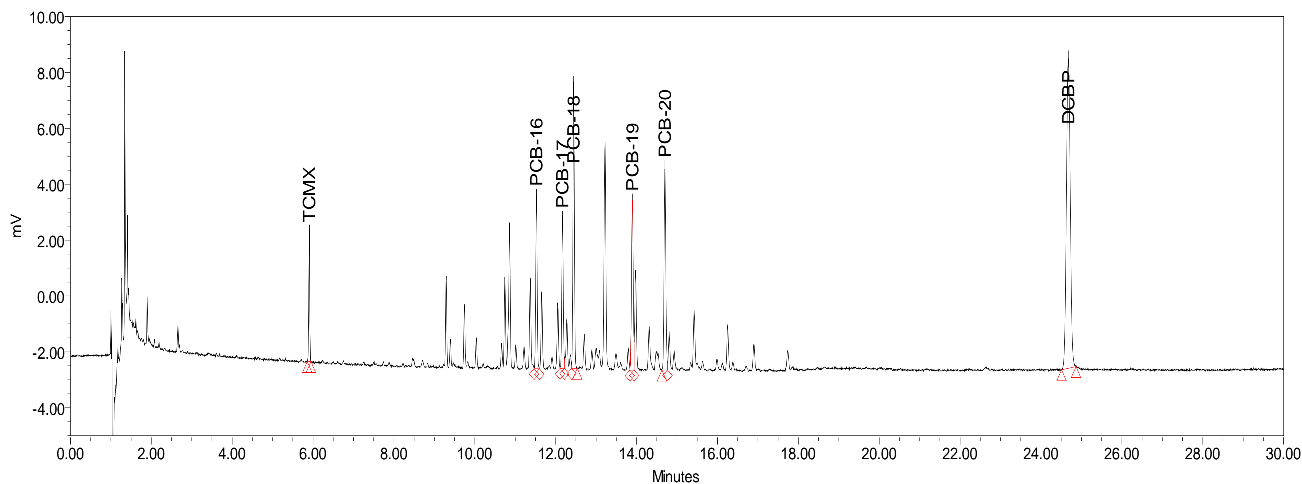


FIGURE 6: A1254 at 0.500ug/mL w/ TCMX & DCBP at 10/100ug/L Plot



Attachment IV: Chromatograms (ZB1 Column/Helium Carrier Gas) (continued)

FIGURE 7: A1260 at 0.500ug/mL Plot

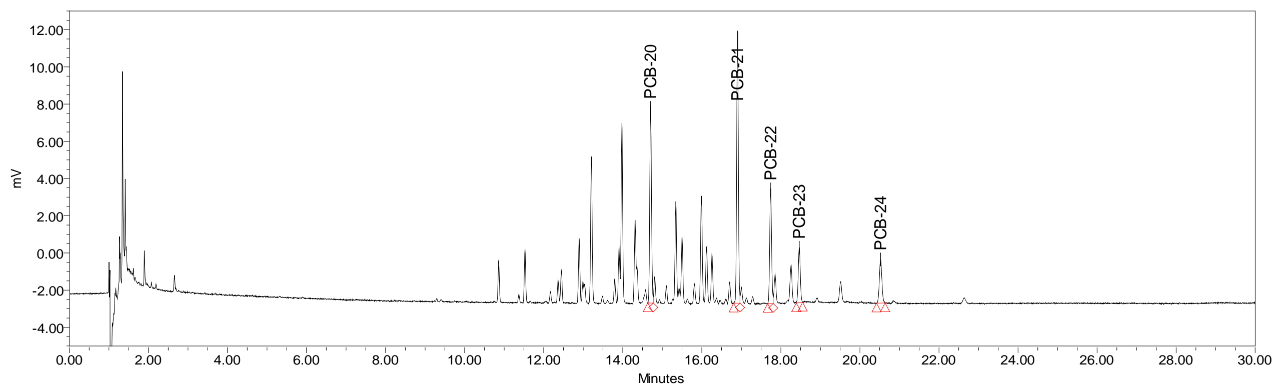
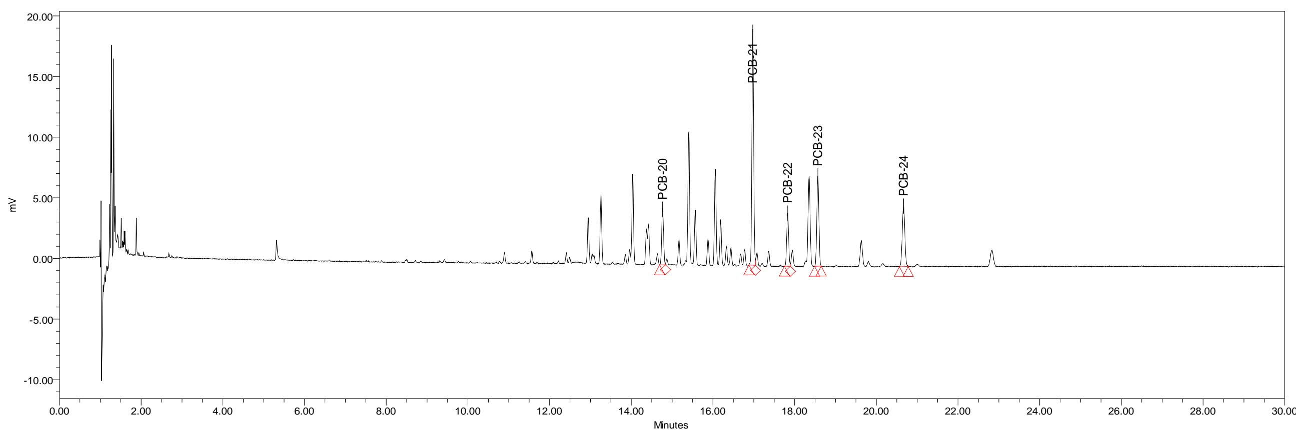
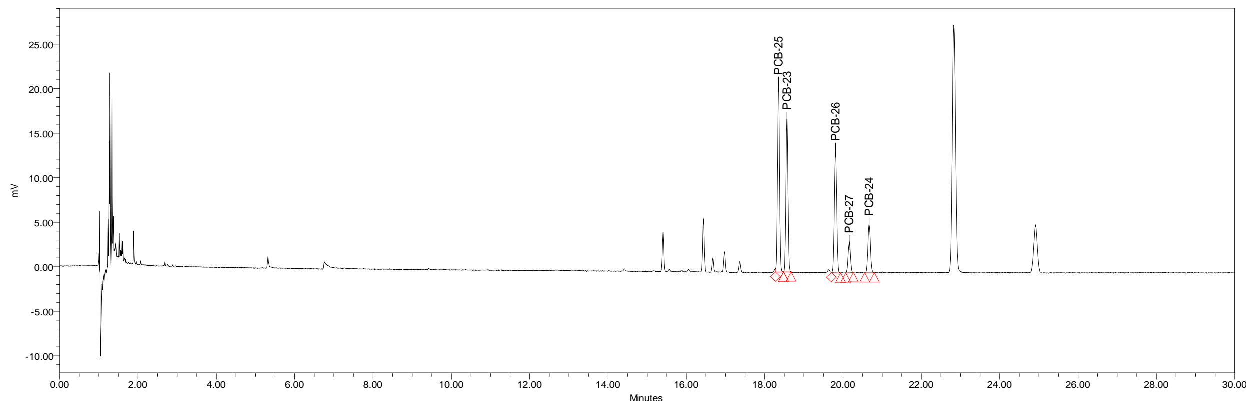


FIGURE 8: A1262 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB1 Column/Helium Carrier Gas) (continued)

FIGURE 9: A1268 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 1: A1016 at 0.500ug/mL Plot

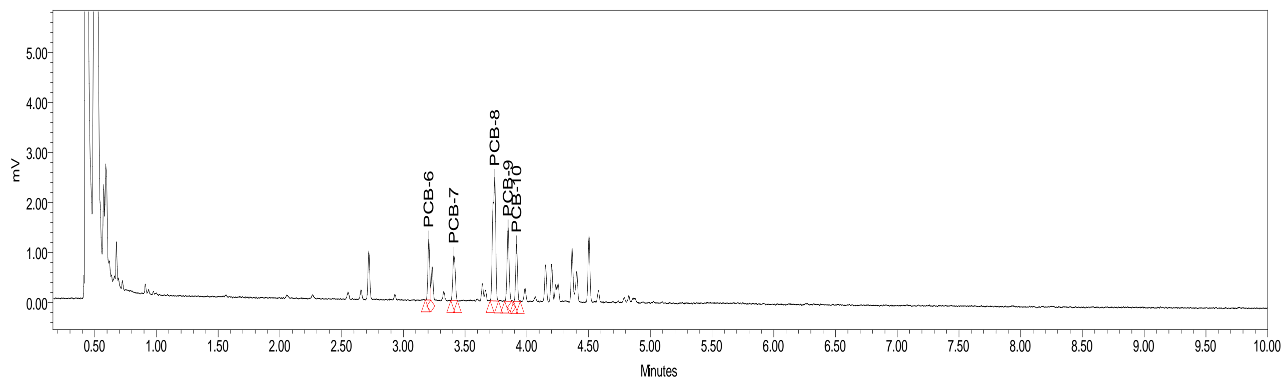
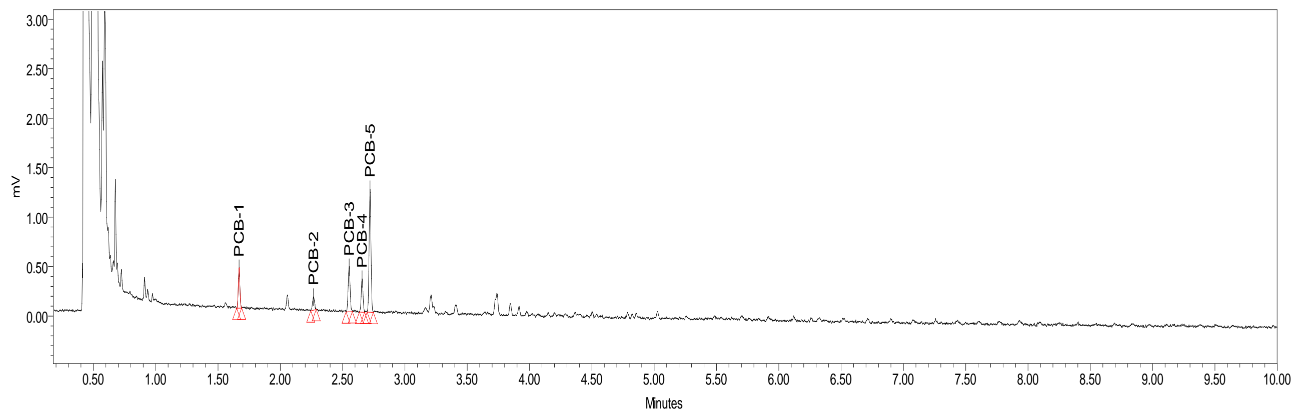


FIGURE 2: A1221 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 3: A1232 at 0.500ug/mL Plot

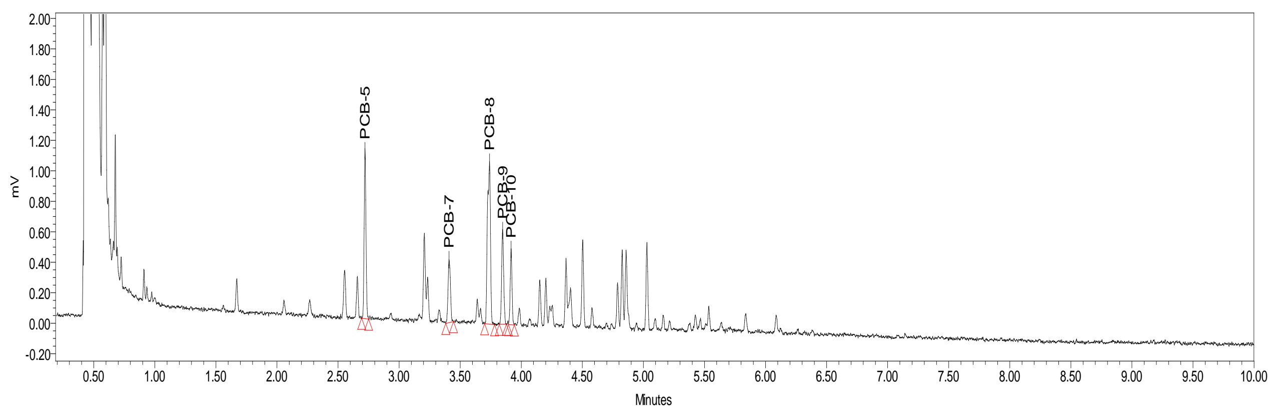
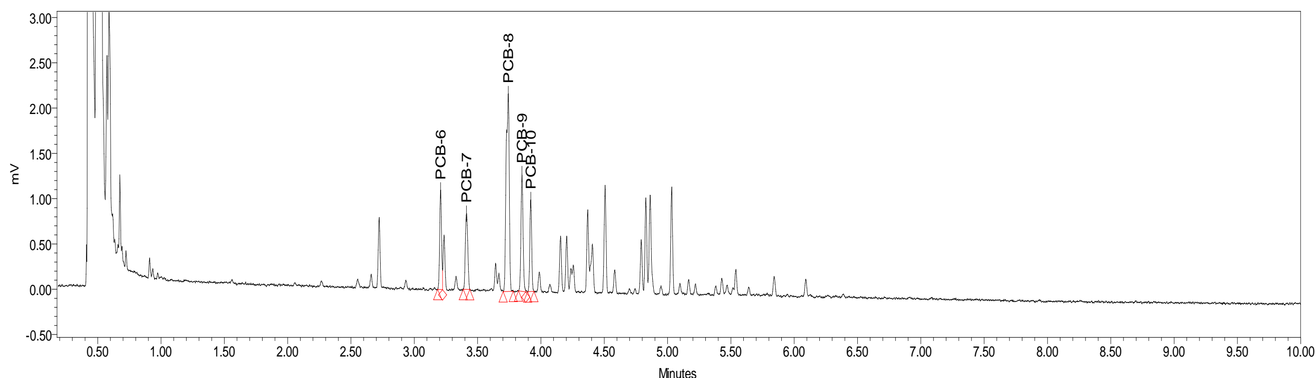


FIGURE 4: A1242 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 5: A1248 at 0.500ug/mL Plot

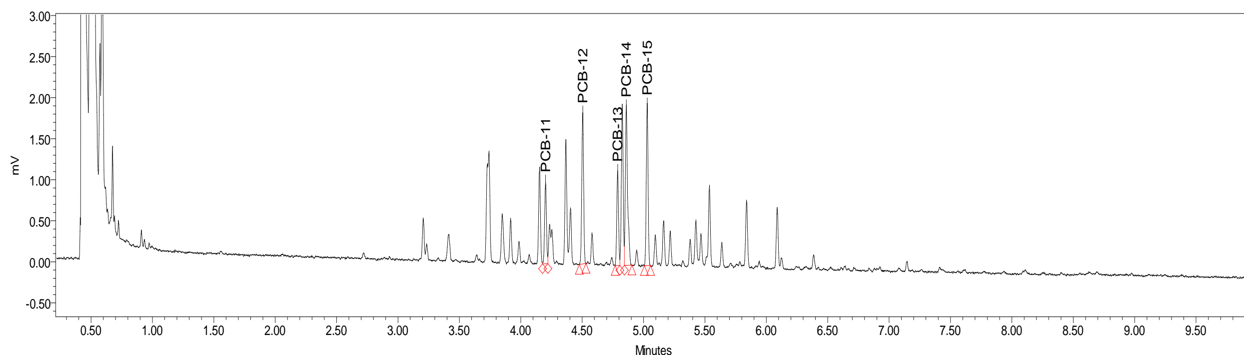
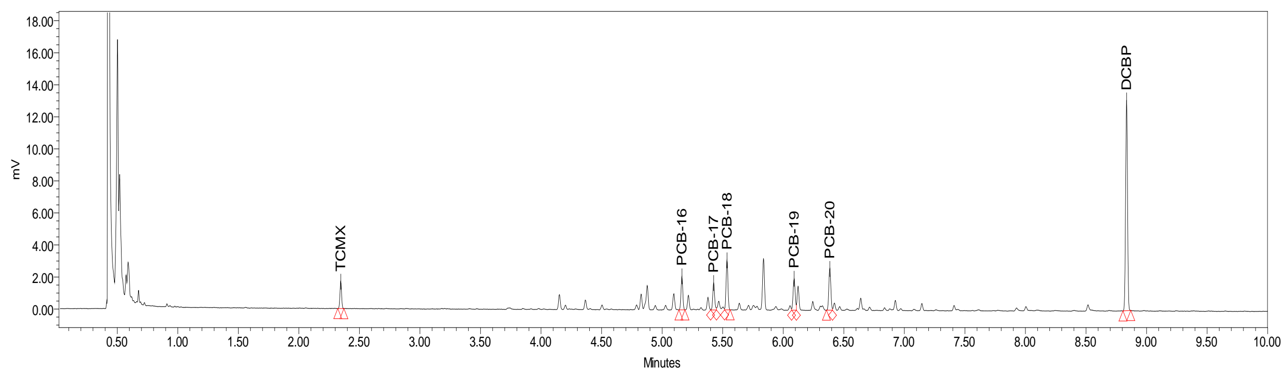


FIGURE 6: A1254 at 0.500ug/mL w/ TCMX & DCBP at 10/100ug/L Plot



Attachment IV: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 7: A1260 at 0.500ug/mL Plot

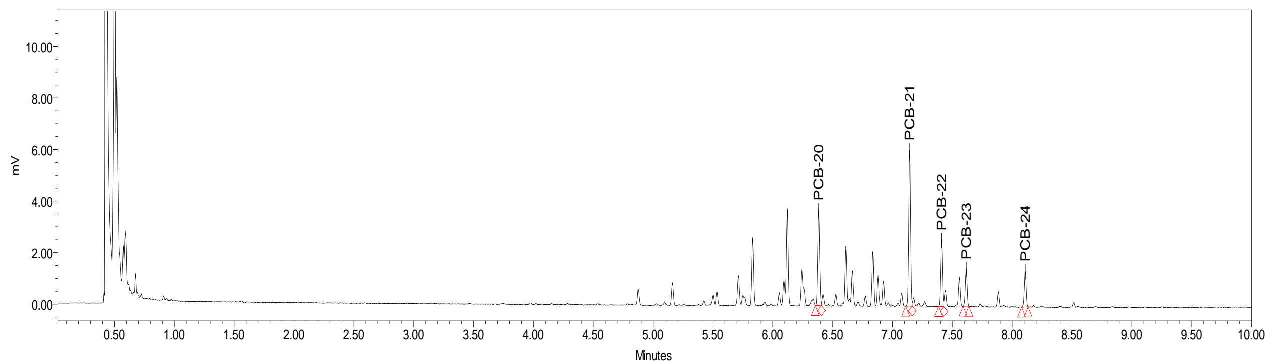
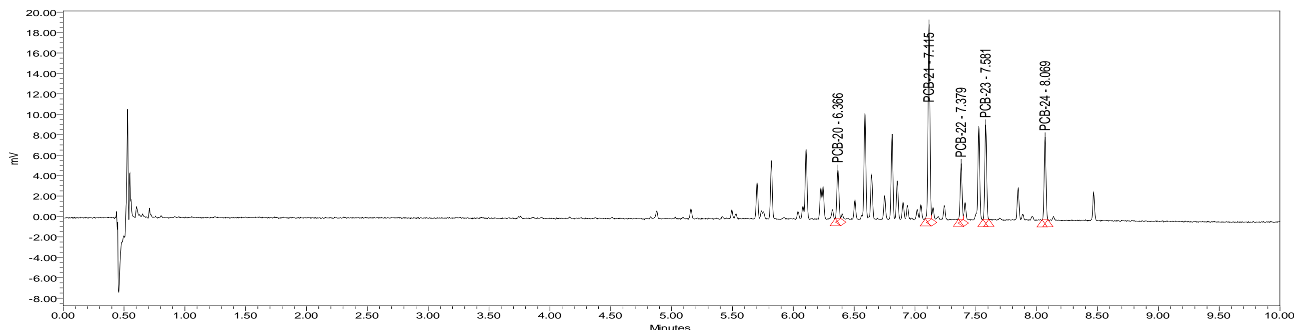
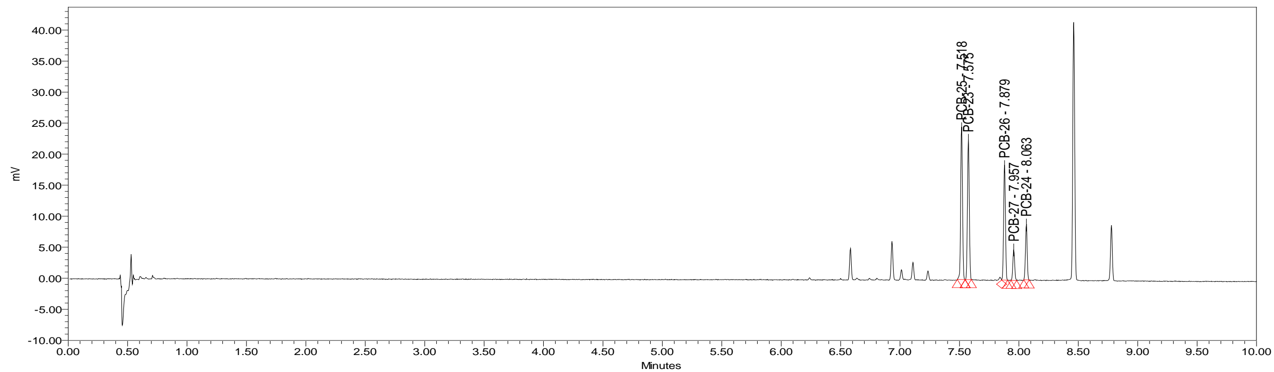


FIGURE 8: A1262 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 9: A1268 at 0.500ug/mL Plot



APPENDIX P3-6
SOP FOR THE PREPARATION AND ANALYSIS OF TRACE METALS BY ICP-AES BY SW-846 METHODS 3005A AND 6010C
(S-LI-M-001-REV.00)

STANDARD OPERATING PROCEDURE

PREPARATION AND ANALYSIS OF TRACE METALS BY ICP-AES

Reference Methods: EPA SW-846 Methods 3005A, 3050B and 6010C

SOP Number:	S-LI-M-001-rev.00
Effective Date:	Date of Final Signature
Supersedes:	6010C_r6

APPROVALS

[signature]	5/15/15
General Manager	Date
[signature]	5/15/15
Quality Manager	Date
[signature]	5/15/15
Department Manager	Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose/Identification of Method

1.1. The purpose of this SOP is to outline the preparation and analysis steps for ICP Metals by EPA SW-846 methods 3005A, 3050B and 6010C.

2. Summary of Method

2.1. This method describes the analysis of trace elements according to the requirements of method 6010C by inductively coupled plasma atomic emission spectroscopy (ICP-AES). The target analyte list (TAL) metals as well as reporting limits are listed in Attachment I.

2.2. Prior to analysis samples must be prepared using appropriate methods. When analyzing ground water samples for dissolved metals, acid digestion is not necessary if the samples were filtered and acid preserved.

2.3. Simultaneous multi-element determinations of trace elements are performed. The basis of Method 6010C is the measurement of atomic emission by an optical spectrophotometric technique. An aliquot of sample is nebulized and the aerosol that is produced is transported to a high energy, high temperature plasma torch where desolvation and excitation occurs. The element specific atomic-line emission spectra that are produced are quantitated by a radio frequency inductively coupled plasma spectrometer. The spectrum is initially dispersed by the fixed grating spectrometer and the intensity at specific analytical wavelengths is monitored by a photomultiplier tube array. Photoelectric currents (i.e., intensities) to the photomultiplier tubes are processed by individual analyte channel cards and collected by the host data system or computer. Background correction and interelement correction techniques are required to compensate for variable background and spectral interferences that may contribute to analytical discrepancies. The possibility of additional interferences may be encountered and appropriate corrections made accordingly.

3. Scope and Application

3.1. In addition to the requirements of this SOP, the guidelines in the Pace Quality Manual must be observed.

3.2. **Personnel**: This SOP is applicable to all personnel involved in the preparation and/or analysis of samples for the ICP metals methods listed.

3.3. **Parameters**: See Attachment I for analytes for this method.

4. Applicable Matrices

4.1. This method is applicable to the determination of dissolved and total metals in all matrices including groundwater, aqueous samples, TCLP, EP extracts, industrial and organic wastes, soils, sludges, sediments, paint chips and other solid wastes requiring digestion prior to analysis. This method may also be used for the analysis of lead in surface wipes.

5. Limits of Detection and Quantitation

5.1. Method detection limits are performed annually by the analysis of seven spiked blanks that have undergone the digestion procedure. The method detection limit is calculated by the procedure defined in 40CFR Part 136 Appendix B.. Specific MDLs are available from the Quality Manager.

5.2. Practical Quantitation Limits (PQL or Reporting Limit) may be found in Attachment I. The PQL may be at or higher than the LOQ.

5.3. Limits of Quantitation (LOQ) are listed in Attachment VI.

5.4. Method detection limits for lead wipe samples are dependent on the area sampled. Results are reported as ug/wipe.

5.5. Instrument detection limits (IDLs) are performed quarterly by the analysis of seven blanks run on three non consecutive days. The instrument detection limit is calculated to be three times the average SD of the standard runs.

6. Interferences

6.1. Spectral interferences can be categorized as follows: overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena, and background contribution from stray light from the line emission of high concentration elements.

6.1.1. The first of these effects can be compensated by utilizing a computer correction of the analytical raw data, which requires the monitoring and measurement of the magnitude of the interference on the analyte to be determined by the interfering element. The inter-element correction (IEC) factor in this case resolves the spectral overlap interference. The second effect may require selection of an alternate wavelength. The third and fourth effects are resolved mostly by the insertion of a background correction placed adjacent to the analyte line.

6.1.2. In addition, users of simultaneous multi-element instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that may be present in a sample but for which there is no channel in the instrument array. For this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed. Analytical systems may exhibit somewhat different levels of interference and the interference effects must be evaluated for each individual system.

6.2. Physical interferences are generally considered to be effects associated with the change in viscosity and surface tension that can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump will lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Also, it has been reported that better control of the argon flow rate may improve instrument performance. This is accomplished with the use of mass flow controllers.

6.3. Memory interferences may result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length.

6.4. Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed can be minimized by careful selection of operating conditions, or use of internal standards. The addition of an element not found in the samples (i.e., lithium) is added at a concentration sufficient for precision, but not so high to alter the salt concentration of the matrix. Yttrium is used as an internal standard.

6.5. Another problem that can occur from high dissolved solids is a salt buildup at the tip of the nebulizer. This affects the aerosol flow rate, which in turn causes instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution have been used to control this problem.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Aqueous samples are collected in at least one plastic 500mL bottles with nitric acid preservative to a pH of < 2. If dissolved metals are required, the sample is filtered immediately on-site by the sampler prior to adding nitric acid preservative.

7.2. For samples requiring dissolved metal analyses, if field filtration of the sample was not performed at the time of collection, the unpreserved sample will be filtered by the laboratory upon receipt and recorded. The filtrates will be acid preserved (~5mL of 1+1 HNO_3/liter) and verified to a pH of < 2. The samples are held for 24 hours prior to digestion. Note: If possible, a deionized water blank will be processed with the filtration apparatus prior to filtering the samples. The blank will be analyzed for sources of possible contamination.

7.3. Soil samples are collected in at least two-ounce PC/PPE plastic, clear or amber glass bottles. The samples will be stored in cabinets or the refrigerator in the department until the time of analysis.

7.4. Wipes for lead analysis are received in glass vials or plastic bags.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section. Other terms are as follows.

8.2. Dissolved Metals: Those elements which will pass through a 0.45µm membrane filter.

8.3. Suspended Metals: Those elements which are retained by a 0.45µm membrane filter.

8.4. Total Metals: The concentration of an analyte determined on an unfiltered sample following treatment by an appropriate digestion procedure.

8.5. Instruments Detection Limit (IDL): Seven replicate blank measurements are determined on three nonconsecutive days. The standard deviation multiplied by three is the calculated IDL. IDLs must be run quarterly.

8.6. Sensitivity: The slope of the analytical curve (i.e., functional relationship between emission intensity and concentration).

8.7. Interference Check Sample: A combination of solutions containing both interfering and analyte elements of known concentrations that can be used to verify background and inter-element correction factors.

8.8. Quality Control Sample: A solution obtained from an outside source having known concentration values to be used to verify the calibration standards. This sample is also known as the initial and/or continuing calibration verification (ICV and CCV respectively).

8.9. Laboratory Control Sample (LCS): Aqueous and solid laboratory control samples must be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the samples.

8.10. Calibration Standards: A series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

8.11. Linear Dynamic Range: The concentration range over which the analytical curve remains linear. This must be analyzed and reported for each element.

8.12. Method Blank: A volume of deionized water containing the same acid matrix as the prepared/digested samples carried through the entire analytical scheme (i.e., matrix blank).

8.13. Matrix Spike (MS), Unspiked Duplicate (Dup) or Matrix Spike Duplicate (MSD): Documents the effect of a matrix, for a given preparation batch. The MS and MSD involve the addition of a known concentration of a standard to the sample prior to digestion. The Dup is a second preparation of a sample. In most cases a MS/Dup are prepared. MS/MSD are prepared if project requested.

8.14. Calibration Blank: A volume of deionized water containing the same acid concentration as the calibration standards.

8.15. Post Digestion Spike (Analytical Spike): Involves the addition of a known amount of concentration of a standard to the digested sample. This spike is needed if the matrix spike percent recovery is not within 75-125%.

8.16. Internal Standard: An analyte, such as yttrium, that is added to the sample to monitor chemical interference.

8.17. Plasma Solution: A solution typically used for optimization of the plasma viewing height with respect to the coil in the R.F. system. A 1000mg/L yttrium standard is often used as the plasma solution.

8.18. Limit of Detection (LOD): An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte-and matrix-specific and may be laboratory-dependent. According to NELAC/TNI, the LOD equates with the MDL.

8.19. Limit of Quantitation: (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The lowest concentration that produces a quantitative result within specified limits of precision and bias.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Equipment and Supplies:

Equipment/Supply	Description	Vendor/ Item # / Description
ICP	ICAP 6000 series	Thermo
RF Generator	2.5kw, crystal controlled at 27.12 MHz, automatically tuned, 6 power level settings are available and can be manipulated by the operator through instrument software	
PA Tube		Thermo / Catalog # 1106-0168
ICP Power Unit	Floor mounted power source provides high Voltage to the RF Generator and all accessories, except the polychromators vacuum pump	Insert Vendor / Catalog #
Alcatel vacuum system	Pump system consisting of a vacuum tank for the spectrometer, direct vacuum pump with digital guage, an automatic high voltage cutoff and a purged optical path (POP)	
Autosampler	CETAC ASX-5200	CETAC
Argon gas	Grade 5 Argon (99.999%) with two stage regulator capable of delivering a flow of 20L/min (50SCFH) at 60 psig	
Peristaltic pump	Variable speed pump required to deliver standard and sample solutions to the nebulizer	

Equipment/Supply	Description	Vendor/ Item # / Description
High flow plasma torch	Kit includes ceramic base and aqueous sample injector	Catalog # 1264-3203
Spray chamber	Cyclonic type	Catalog # 1346-2000
Nebulizer	Meinhard, concentric type TR-30-K2	Catalog # 1343-6500
Graduated cylinders	100mL	
Beakers	250mL	
Filter Funnels		
Filter Paper	Whatman 41 or equivalent and Gelman 0.45um nylon membrane	
Centrifuge		
Analytical balance	Accurate to minimum of 0.01g	
Sample bottles	Polypropylene	
Thermometers	Ranging from 0^oC-200^oC	
Volumetric flasks	Class A	
Adjustable pipettes	Calibrated, range 4-10,000uL	
Hot Block apparatus		Environmental Express
Digestion vessels	Polypropylene	Catalog # SC475
Ribbed watch glasses		Catalog # SC505
FilterMates		Catalog # SC0401
Sterile pads	3”x3” or equivalent; for wipe QC	

10. Reagents and Standards

10.1. Reagents and Standards:

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Deionized (DI) Water	ASTM Type II	
Nitric Acid (HNO_3)	Concentrated	Minimum ACS grade
Nitric Acid (1:1)	Carefully add conc. nitric acid to an equal amount of DI water.	N/A
Hydrogen Peroxide	30%	Minimum ACS grade
Hydrochloric Acid (HCl)	Concentrated	Minimum ACS grade
Hydrochloric Acid (1:1)	Carefully add conc. hydrochloric acid to an equal amount of DI water.	N/A
Element stock solutions	Typically 1000 ug/mL	High purity, NIST traceable
Multi element standard solutions	At mixed concentrations for QC sample requirements.	High purity, NIST traceable

10.2. Standard solutions are prepared by mixing an appropriate volume of the stock standard solution in a volumetric flask and bringing the solution to volume with deionized water. An appropriate acid matrix should be maintained (2% HNO_3 & 5% HCl) for all calibration standards that are prepared. The prepared calibration

solutions can then be transferred to a polyethylene bottle or similar flask for storage. Mixed calibration solutions are prepared as presented and tabulated in Attachment II.

10.3. Pre-certified standard solutions are purchased from high purity standards manufacturers or distributors. These standards are traceable to NIST with certificates of purity and traceability that are kept on file in the metals laboratory. All standards are dated upon receipt. Standards are dated and initialed when opened and tested to determine possible spectral interferences or for the presence of impurities before use.

10.4. Working standards are prepared in class A volumetric flasks. Deionized water is used for the preparation of all reagents and standards and it is analyzed for impurities periodically. All preparation procedures, dates and signatures of preparer are kept logged in the standard preparation log book, along with concentrations and expiration dates. Mixed calibration solutions are prepared fresh daily.

10.5. The calibration blank, (ICB/CCB) is prepared by adding 20mL of HNO_3 and 50mL of HCl to a 1000mL volumetric flask and diluting with deionized distilled water. The calibration solution is analyzed immediately after each calibration check, therefore, a sufficient quantity should be prepared prior to each analytical run.

10.6. The preparation blank, or reagent blank (PBW/PBS) is a solution which contains the same reagents and acid concentrations which were used to process the associated samples. It is carried through the entire sample preparation procedure and diluted to the same volume as the samples.

10.7. The mid and low level calibration verifications (ICV/ICVL and CCV/CCVL) are prepared by adding all elements of interest at concentration levels respective to the midpoints and quantitation limit of the calibration curves. It is prepared in the same acid matrix as the standard solutions and is from a different source than those used to prepare the standardization solutions. The standards are prepared as listed in Attachment VII.

10.8. The low level quantification (LOQ) solution is prepared by adding the appropriate volume of the stock standard solution to match the established reporting limit. The LOQ standard is digested using the same procedures as the samples.

10.9. The ICS working solutions are prepared as needed by diluting the following mixed standards that are obtained from a commercial source:

10.9.1. ICS-A is prepared by adding 20mL of ICS standard A to acidified matrix blank and diluting to 200mL with deionized water. Final concentrations of interfering elements are presented in Attachment IV.

10.9.2. ICS-AB is prepared by adding 20mL of ICS standard A, 2mL of ICS standard B, 0.2mL of 1000mg/L of Se, As, Tl, B, Mo, Sn, Si, Ti, Au and 0.1mL of 1000mg/L of Sr to acidified matrix blank and diluting to 200mL with deionized water. Final concentrations of analyte and interfering elements are presented in Attachment IV.

10.10. The internal standard and ionization solution is prepared by adding 5.0mL of a stock 1000mg/L yttrium standard and 40.0mL of 25.0mg/mL lithium nitrate standard to a volumetric flask containing 900mL of acidified blank and diluting to 1000mL with deionized water.

10.11. The rinse blank, is prepared by adding 20mL of HNO_3 and 50mL of HCl to a 1000mL volumetric flask and diluting with deionized water. The rinse solution is used to flush the system between samples and standards.

11. Calibration and Standardization

11.1. Analytical Sequence: The result of the average replicate exposures must be used for reporting analyte concentrations. The system is rinsed with a matrix blank prior to the start of the analytical run and with a blank between each run. The following is an example of the order of an analytical sequence:

1- {instrument calibration}
2- ICV
3- ICVL
4- ICB
5- ICSAI
6- ICSABI
7- CCV1
8- CCVL1
9- CCB1
10- { 10 samples }
11- CCV2
12- CCVL2
13- CCB2
14- { 10 samples }
15- CCV3
16- CCVL3
17- CCB3
18- { 10 samples }
19- CCVn
20- CCVLn
21- CCBn

11.2. Instrument Calibration: The ICAP6000 is standardized daily, or every time a situation calls for an adjustment to instrument operating parameters or replacement of any components in the nebulization system. Two standards are used to calibrate the system, a blank and an upper range standard as determined by the analytical method employed. See Attachment II Calibration Standards, which lists the concentrations of these solutions.

11.3. Calibration blank: The calibration blank is required during the standardization of the instrument to determine the baseline or background level for the calibration slopes. Blank intensity data is measured at every wavelength for each analyte to be determined.

11.4. Calibration verification (ICV/ICVL and CCV/CCVL): The calibration solutions are instrument check standards that are used to verify the initial performance of the standardization curves. They are subsequently analyzed as continuing reference standards throughout the entire analytical sequence.

11.5. Immediately after instrument calibration, the accuracy of the calibration slopes are verified for all analytes of interest at their respective wavelengths by analyzing the ICV solution. The ICV solutions are of a second source.

11.6. Each CCV analyzed must reflect the conditions of analysis of all associated samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV). The duration of analysis, rinses and other related operations that may affect the CCV measured results may not be applied to the CCV to a greater extent than the extent applied to the associated samples. For instance, the difference in time between a CCV and the CCB following it, as well as the difference in time between the CCV and the sample immediately preceding it, may not exceed the lowest difference in time between any two consecutive analytical samples associated with the CCV.

11.7. Calibration acceptance criteria:

Calibration Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	• Establish initially and annually thereafter. • When continuing calibration check standards fail. • When a change in procedure or instrument.	• Correlation Coefficient ≥ 0.998.	• Evaluate the standards and equipment and reanalyze the initial calibration.

Calibration Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Verification Standard (ICV)	• Immediately after the initial calibration. • Prepared from a source different than the calibration standards. • The daily sample sequence is to be opened with an ICV.	• 90-110% recovery	• Reanalyze and recalibrate if necessary. • Sample analysis cannot begin until a compliant Initial Calibration and ICV meet acceptance criteria. • Samples analyzed after a non-compliant ICV must be reanalyzed.
Limit of Quantification Verification Standard (LOQ/ICVL)/Reporting Limit Verification (RLV)	• Analyzed immediately after the ICAL and ICV. • Prepared at the same concentration as the lowest calibration standard to verify accuracy at the reporting level. • If the reporting level is higher than the lowest calibration standard, the LOQ solution may be prepared at the reporting level.	• 70-130% recovery • Or client data quality objectives, if such exist.	• Evaluate standard preparation. • Reanalyze and recalibrate if necessary.
Continuing Calibration Verification Standard (CCV)	• One per 10 samples (or daily, if fewer than 10 samples) • The analytical sequence must end with a compliant CCV.	• 90-110% recovery	• Samples that are not bracketed with compliant CCVs are to be reanalyzed. • If a re-analysis of a CCV is still non-compliant, the standards and equipment must be evaluated, prepare new standards if necessary and recalibrate.
Limit of Quantification Continuing Calibration Verification (CCVL)	One per 10 samples (or daily, if fewer than 10 samples)	• 70-130% recovery	• Reanalyze and recalibrate if necessary.
Initial and Continuing Calibration Blanks (ICB and CCB)	• ICB after ICV. • CCB per 10 samples (or daily, if fewer than 10 samples) – to be analyzed immediately after each CCV. • The analytical sequence must end	• Should not contain target analytes or interferences at concentrations that impact the analytical results for sample analyses. • The absolute value of	• If the CCBs contain target analytes greater than the CRDL, the blank needs to be re-analyzed. If the re-analysis passes criteria, then reanalyze the associated samples.

Calibration Item	Frequency	Acceptance Criteria	Corrective Action
	with a compliant CCB.	the concentration of target analytes should not be greater than the CRDL.	• If the blank still fails, the system needs to be evaluated for the source of contamination. Re-analysis and recalibrate if necessary.

12. Procedure

12.1. Subsequent to the use of any of the digestion procedures outlined below, all glassware used is cleaned with tap water and a non-phosphate detergent (i.e., Alcojet or similar) and immersed in a 20% HNO_3 bath for two hours, or, if possible, overnight. Finally, the glassware is rinsed with copious amounts of deionized water.

12.2. Water (Total Recoverable or Dissolved Metals) Sample Preparation, Hot Block (Method 3005A Procedure for Acid Digestion of Waters to be analyzed by ICP)

12.2.1. Use 50 mL of well mixed, acid preserved sample and transfer to a SC475 digestion cup.

12.2.2. Prepare aliquots for QC samples (method blank, LCSW, MS, Dup and LOQD).

12.2.3. Add 1 mL of HNO_3 and 2.5mL of HCl and heat in the block at a sample temperature of 95°C. Reduce volume to 10 mL, approximately four hours. Cool sample and adjust volume to 50mL.

12.2.4. Filter with SC0401 FilterMate if necessary to remove insoluble material. Sample is now ready for analysis.

12.3. Soil/Sediment Sample Preparation - Hot Block (Method 3050B Procedure for the Acid Digestion of soils and sediments to be analyzed by ICP):

12.3.1. Mix sample thoroughly to achieve homogeneity and sieve if appropriate. For each digestion procedure, weigh to the nearest 0.01 g and transfer approximately 1g aliquot (or appropriate amount) to the polypropylene digestion vessel.

12.3.2. Prepare aliquots for QC samples (method blank, LCSS, MS, Dup and LOQD).

12.3.3. Add 2.5mL Nitric acid + 2.5ml DI Water and swirl. Cover with a ribbed watchglass or reflux cap and heat the sample in the Hot Block at 95°C for 10 minutes without boiling. Please note- adjust the Hot Block set point temperature so that a 50mL 5% Acid solution (without a watchglass or cover) is heated to 85°C. Allow the sample to cool well and add 2.5mL conc. Nitric Acid and reflux at 95°C for 30 minutes. Repeat this step until no brown fumes are given off by the sample.

12.3.4. Heat sample with the ribbed watch glass for an additional 2 hours. Do not allow the sample to boil or go dry. Cool completely. Very important to have sample well cooled.

12.3.5. Add 1ml of DI water and 1.5 ml of 30% H_2O_2 slowly. Allow an exothermic reaction to occur. Wait 5-10 minutes and place back in the Hot Block with the ribbed watch glass. If effervescence starts to occur lift the samples out of the Hot Block and allow the reaction to continue. Do not let the samples foam out of the vessel. Reducing the Hot Block display temperature by 10°C has shown to reduce the effervescence while still maintaining temperature because of the exothermic reaction occurring with the addition of the H_2O_2.

12.3.6. Continue to add 0.5mL of H_2O_2 (maximum of 5 mls) until the sample remains unchanged in color heating for a total of 2 hours (heat with H_2O_2 for only 30 minutes).

12.3.7. Add 5mL conc. HCl to the sample and cover with a ribbed watchglass and reflux at 95°C for 15 minutes.

12.3.8. After cooling, dilute to 50mL with DI water and use the Filtermate for sample filtration. This step should be performed slowly with little pressure placed on the plunger. If excessive backpressure occurs stop filtration and allow sediments to settle out. Applying pressure to the plunger may cause sample "blow through" and allow sediment to pass through the filter into the digestate. The sample is now ready for analysis.

12.4. Procedure for lead wipe digestion:

12.4.1. Place Wipe into a 250mL conical beaker or equivalent.

12.4.2. Prepare aliquots for QC samples (method blank, LCS, MS/Dup).

12.4.3. Add 10mL of HNO_3, mix gently and cover with a watch glass.

12.4.4. Heat the sample to 95°C and reflux for 10 to 15 minutes without boiling.

12.4.5. Allow sample to cool, add 5mL of concentrated nitric acid and reflux for an additional 30 minutes.

12.4.6. To ensure complete oxidation, repeat step [4]. Evaporate solution to 5mL without boiling.

12.4.7. Allow sample to cool. Add 2mL of deionized water, and 3mL of 30 % hydrogen peroxide (H_2O_2). Cover beaker with a watch glass and warm on hot plate to start the peroxide reaction.

12.4.8. Heat until effervescence subsides. Cool the beaker.

12.4.9. Continue to add 1mL aliquots of 30% H_2O_2 with heat until reaction is minimal or until sample appearance is unchanged.

12.4.10. **NOTE**: Do not exceed a total of 10mL of the Hydrogen peroxide.

12.4.11. Add 5mL of concentrated HCL and 10mL of water, cover and reflux for an additional 15 minutes without boiling.

12.4.12. Cool, dilute to 100mL with deionized water. If necessary, filter digestate through Whatman No. 41 or equivalent filter paper or centrifuge at 2000 – 3000rpm for 10 minutes.

12.4.13. **NOTE**: Final matrix concentration is approximately 5% HNO_3/5% HCL (V/V).

12.5. Prior to analysis, the following QC must be performed: instrument detection limits (IDL), determination of linear range, and calculation of the inter-element correction factors (IECs). See the quality control section for specific requirements.

12.6. Instrument analysis:

12.6.1. Set up the instrument per manufacturer's recommendations. Introduce samples to sampler (manually or by autosampler). Run samples in the same manner as the standards.

12.6.2. Three replicate exposures must be performed for each sample (including QC) in the analytical sequence.

12.6.3. Set up the run/autosampler.

12.6.4. Monitor the QC checks throughout the analytical run. If any of the QC checks fail to meet their specified criteria, follow action as outlined.

12.6.5. Wavelength and background correction locations are listed in Attachment III. Alternate wavelength and background corrections may be substituted if they can provide the needed analytical sensitivity and are corrected for spectral interferences. Because of differences between various models of

ICP spectrometers the analyst should adhere to the manufacturer's recommendations on operating conditions. Operating conditions for the analysis of water and soil matrices are listed as follows:

Plasma Operating Conditions (Typical)

R.F. Power	950 watts
Torch Gas	High Flow
Auxiliary Gas Flow	Low (0.5L/min)
Nebulizer Pressure	24 psi*
Uptake Rate [peristaltic]	1.0 - 2.0mL/min

12.6.6. Turn on the power to the host computer and all other peripherals connected to the computer. Turn power on to instrument and / or other related components, such as vacuum systems, R.F. generator, cooling water recirculator and other electronics if necessary.

12.6.7. Verify liquid argon supply and check regulator for appropriate incoming pressure (80 psig). If the argon level is low or dewar is empty, connect a new dewar and order a replacement.

12.6.8. All startup procedures including plasma ignition are initiated through the host computer. Allow the plasma as well as the instrument (i.e., R.F. generator) to thermally stabilize prior to profiling the spectrometer. This typically requires a 30 to 45 min warm up period. The ICAP 6000 is a self profiling instrument.

12.6.9. Select the analytical method to be used from the Operations - Analysis menu. Standardize the instrument with the prepared calibration standard solutions. Flush the system with the rinse blank between analysis of each calibration standard.

12.6.10. Begin the analytical sequence by introducing the calibration verification (ICV) followed by the calibration blank (ICB).

12.6.11. **NOTE**: Three replicate exposures are performed for all standard, QC, and sample analyses and the average of these exposure results is reported.

13. Quality Control

13.1. A preparation blank (reagent blank) is used as a means of monitoring and correcting for contamination levels which may have resulted during the sample preparation procedure.

13.2. At least one aqueous and/or solid laboratory control sample (LCSW/LCSS) must be analyzed for all analytes of interest per matrix for every group of aqueous or solid samples in a sample delivery group (SDG) or for each batch of samples undergoing the digestion procedure, whichever is more frequent. The aqueous LCS consists of a prepared standard solution at concentration levels equivalent to the calibration verification. After preparation, the aqueous LCS is analytically verified by an ICP scan to be within specified quality control limits before the solution is used for any digestion procedures. If any of the analytes in the LCS are not within QC limits the solution is re-made and/or re-tested to determine the cause of the inaccuracies. The solid LCS is a certified QC standard purchased from an outside reputable source. The aqueous LCS is prepared as in Attachment VII.

13.3. Interelement Corrections: The ICP interelement correction factors are determined semi-annually. Correction factors for spectral interference due to Al, Ca, Fe, and Mg must be determined for all wavelengths

used for each analyte reported. Correction factors for spectral interference due to analytes other than Al, Ca, Fe, and Mg must be reported if they were applied.

13.4. The interference check sample is a solution prepared to contain known high level concentrations of interfering elements and known low level concentrations of other analytes. The solution will confirm the magnitude of interferences and monitor the performance of the interelement correction factors (IEC). The ICS is analyzed as verification that interference levels are corrected by the analytical system within specified quality control limits.

13.4.1. The interference check sample consists of two solutions; ICS-A, containing only interfering elements (Al. Ca, Fe, Mg) and ICS-AB, containing low level analytes combined with the interfering elements.

13.4.2. As a verification of background correction points (BCP) and interelement correction factors (IEC's), the ICS solutions must be analyzed for each analyte of interest at the beginning of every analytical run.

13.4.3. Analysis of the ICS solutions is initiated by sequentially analyzing the ICS-A followed by the ICS-AB solution.

13.4.4. Interference check sample (ICS) analysis: Concentration results for the ICS solutions (ICS-A/ICS-AB) must be within ± 20% of the true analyte values otherwise the analysis is terminated, corrective measures taken and the instrument re-standardized.

13.4.5. Concentration results for all analytes other than the interferents (Al, Ca, Fe, Mg) in the ICS-A should be =2× the CRDL. No specific QC criterion is given or action required if this is not the case, however analyst should assess the performance of the instrument, the method interelement correction (IEC) factors, or ICS solution, and decide whether to terminate the analysis.

13.5. A spiked sample analysis is intended as a means for assessing the effect of sample matrix on the sample preparation procedure and the overall performance of the analytical method employed. At least one spiked sample analysis is performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration level (i.e. low, medium) or for each SDG, whichever is more frequent. If no sample is assigned for spiked sample analysis, the laboratory will select one. The analyte spike must be added before any digestion procedures. Concentrations levels for the matrix spike are listed in Attachment V.

13.6. At least one duplicate sample analysis is performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration level (i.e., low, medium) or for each SDG, whichever is more frequent. If no sample is assigned for duplicate sample analysis, the laboratory will select one. Concentration results for duplicate sample analyses of aqueous samples are in units of µg/L and for solid samples units are expressed in mg/kg, dry weight basis.

13.7. Internal standard and ionization solution. Yttrium at a concentration of 5mg/L is used as an internal standard for ICP analyses on the trace analyzer. 1000mg/L of lithium is also added to control ionization effects that may contribute to analytical difficulties in quantifying concentrations for sodium and potassium. All element concentrations are based on the ratio of analyte intensity to yttrium intensity over the entire integration period. The yttrium and lithium solution is introduced through a tee which combines the sample and the internal standard and ionization solution and mixes the solution by way of a capillary coil directly connected to the instruments nebulizer.

13.8. Linear Range Analysis (LRA): A linear range verification check standard is reported on a semiannual basis for all elements. The concentration of the elements in the standard must be within ±10% of the true value if it is to be maintained as an upper limit concentration, beyond which results cannot be reported under this protocol without dilution of the analytical sample

13.9. If an analyte is above linear range of the instrument, a dilution will be prepared to bring it within the analytical range of the ICP.

13.10. Batch Quality Control items:

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	• One per batch, • Or 1 per 20 samples, whichever is more frequent. • Processed with and under the same conditions as samples and goes through all the steps of the analytical procedure.	• Should not contain target analytes or interferences at concentrations that impact the analytical results for sample analyses. • Absolute value of concentration of target analytes should not be greater than CRDL. If blank is >CRDL, associated samples must be <10X the prep blank results	• If the blank contains target analytes greater than the PQL, the blank needs to be re-analyzed. If the re-analysis passes criteria, then reanalyze the associated samples. • If the blank still fails, the system needs to be evaluated for the source of contamination and affected samples reanalyzed. • If reanalysis of samples is not possible, report data flagged to indicate method blank contamination.
Laboratory Control Sample (LCS)	• One per batch, or 1 per 20 samples, whichever is more frequent. • Prepared from a source different than the calibration standards – same as the ICV)	• When evaluated as an ICV, the criteria of 90-110% recovery must be met. • When evaluated as an LCS, the method requirement of 80-120% recovery must be met unless the LCS is a solid CRM, and then the manufacturer's acceptance limits can be used.	• Redigest and reanalyze all samples associated with the LCS. • Reanalyze all samples associated with the ICV. • If reanalysis of samples is not possible, report data flagged to indicate LCS failed recovery.
Limit of Quantitation Check Sample (LOQ) (Digested)	• One per batch, or 1 per 20 samples, whichever is more frequent. • Prepared from a source different than the calibration standards – same as the ICV)	• When evaluated as an ICV, the criteria of 90-110% recovery must be met. • When evaluated as an LOQ, the method requirement of 70-130% recovery must be met.	• Redigest and reanalyze all samples associated with the LOQ. (If sample is non-detect and LOQ recovers high, redigestion is not required and samples can be reported. • Reanalyze all samples associated with the ICVL. • If reanalysis of samples is not possible, report data flagged to indicate LOQ failed recovery.
Matrix Spike Sample (MS)	• One per 20 samples or daily, if fewer than 20	• 75-125% recovery; unless analyte	• If the recovery is outside the limits,

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
	samples.	concentration is >4x the spike level.	repeat the sample, duplicate and matrix spike analysis once. If it passes, then report samples. • Check for errors in calculation and spike preparation. • If the matrix spike still exceeds the limits, but the LCS/ICV has acceptable recovery, then the method is in control and sample matrix effects are likely the cause. The data should be qualified in the case narrative or using QC notes in the LIMS for non-package work.
Sample Duplicate (DUP)	• One per 20 samples or daily, if fewer than 20 samples.	• ± 20% RPD • For result values less than five times the PQL, a control limit of ± the PQL will be used.	• Check sample label, calculation, dilution factors. • If results are grossly different (i.e., very high result and non-detect) re-analyze to confirm.
ICP Serial (L) Dilution Analysis (SD)	• At least one ICP serial dilution sample analysis is performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration level (i.e., low, medium) or for each SDG, whichever is more frequent.	• If the element concentrations exceed the instrumental detection limit by a factor of 50 or greater, a 5× dilution of the original sample must be within 10%.	• If the recovery is outside the limits the sample is flagged with an “E” qualifier.

14. Data Analysis and Calculations

14.1. Reagent blanks (prep blanks) will be analyzed and reported in appropriate units, such as micrograms per liter (µg/L) for aqueous samples and milligrams per kilogram (mg/kg) for solid samples. Concentrations below the determined instrument detection limit (IDL) should be reported as undetectable.

14.2. Analytical samples will be analyzed and reported in appropriate units, such as micrograms per liter (µg/L) for aqueous samples and milligrams per kilogram (mg/kg) for solid samples. If dilutions were performed, the sample concentrations must be multiplied by the dilution factor. Concentrations below the determined instrument detection limit (IDL) should be reported as undetectable.

14.3. Quantitative results for solid samples are calculated on a dry weight basis and a separate determination of total percent solids must be performed. The final concentrations are calculated as follows:

$$\text{Concentration (mg/kg)} = \frac{\mathbf{C \times V}}{\mathbf{W \times TS}}$$

Where: C = digestate analyte concentration (mg/L)*

V = final preparation volume (mL)

W = wet weight of sample (g)

TS = dryness factor; total solids as a decimal fraction (i.e., 95% total solids equals a 0.95 TS)

14.4. Certain parameters are calculated from the elemental ICP analysis. The molar factor will be used to calculate the result. For example:

Calculation of Silica: mg/L Silica = mg/L Silicon x 2.14

14.5. Percent recoveries of individual analytes are calculated and reported in units appropriate to the sample matrix. The following equation is used:

$$R = \frac{SSC - SC}{SA} \times 100\%$$

Where: R = percent recovery

SSC = spiked sample concentration

SC = sample concentration

SA = spike added

14.6. Relative percent difference (RPD) of individual analytes are calculated as follows:

$$RPD = \frac{S - D}{\frac{(S + D)}{2}} \times 100\%$$

Where: RPD = relative percent difference

S = sample concentration

D = duplicate sample concentration

14.7. Percent difference (%D) of individual analytes are calculated as follows:

$$\%D = \frac{I - S}{I} \times 100\%$$

Where: I = initial sample concentration

S = serial dilution concentration (= sample result × 5)

14.8. Calculated quality control data associated with sample analyses is a good indication of the characteristic sample data and should be provided with the sample data and its package.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Reported data for all analytical samples and quality control checks as outlined in Method 6010C must adhere to the criteria and guidelines as specified by the protocol. Instrument detection limits, linear range concentrations, inter-element check standards and all related data generated and collected by an analytical procedure must be documented for quality control purposes and maintained as an assessment to the usability, validity and overall integrity of the data.

15.2. Raw analytical data is maintained in both hard copy format and disk format which is transferred to the QA/QC department for an initial verification of data integrity and completeness.

15.2.1. For data submissions requiring disk deliverables with the raw analytical data, the systems file format used for storage of the analytical data generated during the sample sequence and subsequently stored to the acquisition system or host computer is converted from its original file format to an ASCII file format.

15.3. Data packages are generated by LIMS software system which enables all quality control data and samples result data to be presented in an appropriate format. All forms and raw data pertaining to a sample analysis are bound as a data package and mailed to the client. An additional copy of the analytical data package is kept in house for future reference and perusal in the event there is a result or data discrepancy.

15.4. Inorganic data reporting forms are generated by the LIMS software package which initially verifies the integrity of the raw analytical data.

16. Corrective Actions for Out-of-Control Data

16.1. Corrective action begins with the analysts who are responsible for knowing when the analytical process is out of control.

16.2. Corrective actions should be initiated for out of control events such as when QC checks exceed acceptance limits or exhibit trending, holding time failures, loss of sample, equipment malfunctions or evidence of sample contamination.

16.3. See Sections 11.7 and 13.7 for data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

16.4. Non-conformances are noted by the preparation of a report containing the following:

16.4.1. Parameter/ SDG;

16.4.2. Client ID. / Dates collected and received;

16.4.3. Non-conformance;

16.4.4. Root cause;

16.4.5. Corrective action taken;

16.4.6. Action to prevent re-occurrence / follow-up;

16.4.7. Non-conformance forms are initiated by the analyst and forwarded to the department supervisor, project manager and quality assurance officer;

16.4.8. Client notification is handled by the project manager if applicable.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Samples that have been analyzed during a non- compliant run will be rejected. The samples will be reanalyzed after the problem is resolved and the system recalibrated.

17.2. If the sample matrix is such that interferences prevent the system from meeting calibration and quality control requirements, the client will be notified with a course of action determined.

17.3. All problems associated with the analysis of a sample group will be outlined in the case narrative of the data package or with the use of QC Notes in the LIMS, if required.

17.4. See Sections 11.7 and 13.7 for procedures in handling out-of-control data.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

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. Method Detection Limit (MDL) Study

18.4. Instrument Detection Limit (IDL) Analysis: Prior to analyzing any types of samples under the 6010C Protocol, instrument detection limit analyses are performed, and the results for each analyte tabulated in micrograms per liter for all instruments used for metals analysis. Instrument detection limits are updated quarterly and must meet the minimum concentration levels (PQLs) specified in Attachment I.

18.4.1. Instrument detection limits are calculated by analyzing a blank standard obtaining the average standard deviation of three days of analyses of a minimum of seven replicates.

18.4.2. Instrument detection limits are recorded on FORM X-IN for each instrument used and submitted with each analytical data package.

18.5. Linear Range Analysis (LRA): A linear range verification check standard is reported on a semiannual basis for all elements. The concentration of the elements in the standard must be within ±10% of the true value if it is to be maintained as an upper limit concentration, beyond which results cannot be reported under this protocol without dilution of the analytical sample. The linear range is recorded on FORM XII-IN for each instrument used and submitted with each analytical data package.

18.6. Interelement Corrections for ICP: The ICP interelement correction factors must be determined annually. Correction factors for spectral interference due to Al, Ca, Fe, and Mg must be determined for all wavelengths used for each analyte reported. Correction factors for spectral interference due to analytes other than Al, Ca, Fe, and Mg must be reported if they were applied.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. For information and guidance related to instrument/equipment maintenance, refer to the instrument manuals and other documents listed in the Reference section.

21. Troubleshooting

21.1. Troubleshooting procedures come from experience and training. Problems that the analyst encounters may be elevated to the department supervisor. Other means of problem resolution may come from review of prior related corrective action reports, instrument maintenance records, internal and external audits and lab notes.

22. Safety

22.1. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposed to these chemicals must be reduced to the lowest possible level by personal protection and engineering measures. MSDS are available for all chemicals used in the lab and are available for review.

22.2. Safety glasses, a lab coat, and disposable gloves must be worn when handling chemicals and samples.

22.3. Sample handling should be conducted in fume hoods.

23. Waste Management

23.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste.

23.2. Procedures for handling waste generated during this analysis are addressed in the current waste management SOP.

23.3. All wastes deemed toxic are collected in the laboratory and are disposed by professional companies licensed in waste disposal.

24. Pollution Prevention

24.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Use pollution prevention techniques in laboratory operation whenever feasible.

24.2. Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

24.3. Control releases from hood and bench operations and comply with discharge permits and regulations, hazardous waste identification rules, and land disposal restrictions.

24.4. The generated waste has to be disposed in a manner not to cause pollution.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update IV, February 2007 EPA SW846 methods 6010C, 3005A and 3050B.

25.5. ADMIN02: Computers and Programs.

25.6. ME017: Cetac Operator's Manual and Supplies Catalog Instrument Asx 520 Cd 670034.

25.7. ME018: Thermo Scientific Neslab Thermoflex Recirculating Chiller.

25.8. ME019: ICAP 6000 Series ICP-AES Spectrometer Operator Manuals 849940090001.

25.9. ME019A: ICAP Software Issue 08.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Recommended Wavelengths and PQLs.

26.2. Attachment II: Mixed Standards Preparation.

26.3. Attachment III: Element Data.

26.4. Attachment IV: Interferent and Analyte Concentrations for ICS.

26.5. Attachment V: Concentration Levels for Spiked Sample Analysis.

26.6. Attachment VI: Limits of Quantitation.

26.7. Attachment VII: Standard Preparation of ICV and LCS.

27. Revisions

Document Number	Reason for Change	Date
S-LI-M-001-rev.01	Converted to Pace SOP format. IECs run semi-annually. Volumes of acid /h2o and standard prep tables revised to match practice.	5/14/15

Attachment I: Recommended Wavelengths and PQLs

Element	Wavelength [a] (nm)	Estimated Limit[b](µg/L)	PQL (µg/L)	PQL (µg/kg)
Aluminum (Al)	308.215	45	200	20,000
Antimony (Sb)	206.833	32	60	6,000
Arsenic (As)	193.696	53	10	1,000
Barium (Ba)	455.403	2	200	20,000
Beryllium (Be)	313.042	0.3	5	500
Boron (B)	249.773	5	500	50,000
Cadmium (Cd)	226.502	4	2.5	500
Calcium (Ca)	317.933	10	5000	500,000
Chromium (Cr)	267.716	7	10	1,000
Cobalt (Co)	228.616	7	50	5,000
Copper (Cu)	324.754	6	25	2500
Gold (Au)	267.500		200	
Iron (Fe)	259.940	7	100	10000
Lead (Pb)	220.353	42	5	500
Lithium (Li)	670.784	5	---	---
Magnesium (Mg)	279.079	30	5000	500,000
Manganese (Mn)	257.610	2	15	1500
Mercury (Hg)				
Molybdenum (Mo)	202.030	8	20	1,000
Nickel (Ni)	231.604	15	40	4,000
Phosphorus (P)	213.618	51	---	---
Potassium (K)	766.491	see note c	5000	500,000
Selenium (Se)	196.026	75	10	10000
Silver (Ag)	328.068	7	10	1,000
Sodium (Na)	588.995	29	5000	500,000
Strontium (Sr)	407.771	0.3	100	---
Thallium(Tl)	190.864	40	10	1,000
Tin (Sn)	189.990	---	250	25,000
Vanadium (V)	292.402	8	50	5,000
Zinc (Zn)	213.856	2	20	2,000

[a]The wavelengths listed are recommended because of their sensitivity and overall acceptance, Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. In time, other elements may be added as more information becomes available and as required.

[b]The estimated instrumental detection limits shown are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[c]Highly dependent on operating conditions, plasma parameters, and nebulizer pressure.

Attachment II: Mixed Standards Preparation- Standardized Solutions for 6010C

Standard	ID	Volume of Stock Standard Added	Final Concentration	Volume of Solution
#1	BLANK	10mL HNO_3 & 25mL HCl	matrix	500mL
#2	AGSBH	0.40mL of 1000mg/L Ag 0.40mL of 1000mL/l Sb 1.0mL of 1000mg/L P	2.0mg/L 2.0mg/L 5.0mg/L	200mL
#3	ICSH	2.0mL of 10,000mg/L Al 2.0mL of 10,000mg/L Ca 2.0mL of 10,000mg/L Mg 2.0mL of 10,000mg/L Fe	500mg/L 500mg/L 500mg/L 200mg/L	200mL
#4	FURNH	0.20mL of 1000mg/L As 0.20mL of 1000mg/L Pb 0.20mL of 1000mg/L Se 0.20mL of 1000mg/L Tl	1.0mg/L 1.0mg/L 1.0mg/L 1.0mg/L	200mL
#5	MULTI	1.0mL of 1000mg/L MultiCAL STD A 1.0mL of 1000mg/L MultiCAL STD B 1.0mL of 1000mg/L Au 1.0mL of 1000mg/L B, Mo, Ti, Sr, Sn, Si (SM-849-010)	* * * * * * *	200mL
#6	FENAL	0.10mL of 10,000mg/L Fe 0.10mL of 10,000mg/L Na	5.0mg/L 5.0mg/L	200mL
#7	NAK100	4.0mL of 10,000mg/L Na 4.0mL of 10,000mg/L K	100mg/L 100mg/L	200mL

* Standard #5 :Final Concentrations of Au, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Si, Sn, Sr, Ti, V, Zn = 5.0mg/L.

NOTE: All standard solutions contain a 2% HNO_3 & 5% HCl acid matrix.

Attachment III: Element Data for Method 6010C

ELEMENT	WAVELENGTH [nm1]	Background Offset	# of IEC factors used	Standard Name
Ag	328.068	-18	2	AGSBH
Al	308.215	+28	1	ICSH
As	189.042 ×2	-28	3	FURNH
Au	267.500		0	MULTIH
B	249.678 ×2	-10	2	MULTIH
Ba	493.409	-28	0	MULTIH
Be	313.042	-28	0	MULTIH
Ca	317.933	-28	2	ICSH
Cd	226.502 ×2	+28	2	MULTIH
Co	228.616	-28	1	MULTIH
Cr	267.716	-28	2	MULTIH
Cu	324.754	-18	1	MULTIH
Fe 3	271.441	+10	1	ICSH
Fe 3	259.940	+28	0	FENAL
K	766.491	-28	0	NaK100
Mg	279.079	-28	2	ICSH
Mn	257.610	-28	3	MULTIH
Mo	202.030 ×2	-28	0	MULTIH
Na 3	330.232	-28	0	NAK100
Na 3	588.995	+28	0	FENAL
Ni	231.604 ×2	-28	0	MULTIH
P	213.618	-28	0	AGSBH
Pb 2	220.351	-10	4	FURNH
Pb 2	220.352 ×2	+10	5	FURNH
Sb 2	206.831	+10	2	AGSBH
Sb 2	206.832 ×2	-10	3	AGSBH
Se 2	196.021	-10	4	FURNH
Se 2	196.022 ×2	+10	5	FURNH
Sn	189.989	-28	0	MULTIH
Sr	421.552	-28	0	MULTIH
Ti	334.941	+28	1	MULTIH
Tl	190.864 ×2	+28	4	FURNH
V	292.402	+10	1	MULTIH
Zn	213.856 ×2	-28	3	MULTIH

[1] Wavelength ×2 indicates a second order analytical line.
[2] Analytical data is determined by a summing channel (S) which quantitates the results of the individual wavelengths.
[3] Instrumental wavelength switching is utilized for the quantitation of iron and sodium at low versus high concentration levels. Iron at 259.940 nm and sodium at 588.995 nm are the more sensitive analytical wavelengths used for sample concentrations up to 10mg/L iron and 20mg/L sodium.

Attachment IV: Interferent and Analyte Concentrations for Interference Check Sample (ICS)

Interferents ICS-A	Concentration [mg/L]	Analytes ICS-AB[1]	Concentration [mg/L]
Al	500	Ag	1.0
		Au	1.0
Ca	500	B	1.0
Fe	200	Ba	0.5
Mg	500	Be	0.5
		Cd	1.0
		Co	0.5
ICS-AB[1]	[mg/L]	Cr	0.5
Se	1	Cu	0.5
As	1	Mn	0.5
Tl	1	Mo	1.0
Si	1	Ni	1.0
Ti	1	Pb	1.0
P	1	Sn	1.0
Sr	1	Ti	1.0
		V	0.5
		Zn	1.0
		Sr	0.5
		P	0.5

[1] The same analyte concentrations present in the ICS-A are added to the ICS-B solution.

Attachment V: Concentration Levels for Spiked Sample Analysis

Analyte	Water/ [µg/L]	Soil Matrix[mg/kg]
Aluminum	2,000	*
Antimony	500	100
Arsenic	40	8
Barium	2,000	400
Beryllium	50	10
Boron	1000	200
Cadmium	50	10
Calcium	*	*
Chromium	200	40
Cobalt	500	100
Copper	250	50
Gold	500	---
Iron	1,000	*
Lead	20	4
Magnesium	*	*
Manganese	500	100
Molybdenum	1000	200
Nickel	500	100
Potassium	*	*
Selenium	10	2
Silver	50	10
Sodium	*	*
Thallium	50	10
Tin	2000	400
Titanium	1000	200
Vanadium	500	100
Strontium	500	500
Phosphorous	500	500

* = no spike required

Attachment VI: Limits of Quantitation (LOQ) Concentrations

Element:	Units	Concentration
Aluminum	ppm	0.20
Antimony	ppm	0.06
Arsenic	ppm	0.01
Gold	ppm	0.2
Barium	ppm	0.2
Calcium	ppm	1.0
Chromium	ppm	0.01
Cobalt	ppm	0.05
Copper	ppm	0.025
Iron	ppm	0.1
Lead	ppm	0.005
Magnesium	ppm	1.0
Manganese	ppm	0.015
Nickel	ppm	0.04
Potassium	ppm	5.0
Selenium	ppm	0.01
Sodium	ppm	5.0
Thallium	ppm	0.01
Vanadium	ppm	0.05
Zinc	ppm	0.02
Beryllium	ppm	0.005
Cadmium	ppm	0.0025
Silver	ppm	0.01
Boron	ppm	0.05
Strontium	ppm	0.05
Molybdenum	ppm	0.02
Silica	ppm	0.025
Titanium	ppm	0.05
Tin	ppm	0.05

Attachment VII: Standard Preparation of ICV and LCS Solutions

Stock Standard	Volume of Standard / 500mL	Element	Concentration [µg/mL]2
Ag [1000µg/mL]	0.5mL	Ag	1.0
Sb [1000µg/mL]	0.5mL	Sb	1.0
		Al	50
ICVT-2 [10,000µg/mL]	2.5mL	Ca	50
		Mg	50
Fe [10,000µg/mL]	2.5mL	Fe	50
		As	0.5
H2M#1 [1000ug/L]	0.25mL	Pb	0.5
		Se	0.5
		Tl	0.5
		B	2.5
		Ba	2.5
		Be	2.5
		Cd	2.5
		Co	2.5
T-ICV [1000µg/mL]	1.25mL	Cr	2.5
		Cu	2.5
		Mn	2.5
Au (1000ug/mL)	1.25mL	Au	2.5
		Ni	2.5
		Ti	2.5
		V	2.5
		Zn	2.5
K [10,000µg/mL]	4.0mL	K	80
Na [10,000µg/mL]	4.0mL	Na	80
Si [1000µg/mL]	1.25mL	Si	2.5
Sn [1000µg/mL]	1.25mL	Sn	2.5
Sr [1000µg/mL]	1.25mL	Sr	2.5
P [1000ug/mL]	1.25mL	P	2.5

[1] The ICVL is prepared by adding 0.125mL each of the Fe & Na 10,000µg/mL standards to 500mL of deionized water. The final analyte concentrations are 2.5µg/mL.
[2] The final ICV/LCS solution contains a 2% HNO_3 & 5% HCl acid matrix.

Attachment VII: Standard Preparation of LOQ/ICVL Solutions

Stock Standard	Volume of Standard / 1000 mL	Element	Concentration [µg/L]
Pb [10 µg/mL]	200 µL	Pb	2 [1]
Fe [1000 µg/mL]	80 µL	Fe	80 [1]
Se [10 µg/mL]	500 µL	Se	5 [1]
LOQ Stock [various]	1000 µL		
Al [200 µg/mL]		Al	200
Be [5 µg/mL]		Be	5
Cr [10 µg/mL]		Cr	10
Pb [3 µg/mL]		Pb	3 [1]
Ni [40 µg/mL]		Ni	40
Si [20 µg/mL]		Si	20
Tl [10 µg/mL]		Tl	10
Zn [20 µg/mL]		Zn	20
Sb [60 µg/mL]		Sb	60
B [50 µg/mL]		B	50
Co [50 µg/mL]		Co	50
Mg [200 µg/mL]		Mg	200 [1]
P [50 µg/mL]		P	50
Ag [10 µg/mL]		Ag	10
Sn [50 µg/mL]		Sn	50
As [10 µg/mL]		As	10
Cd [2.5 µg/mL]		Cd	2.5
Cu [25 µg/mL]		Cu	25
Mn[15 µg/mL]		Mn	15
K [200 µg/mL]		K	200
Na [200 µg/mL]		Na	200
Ti [50 µg/mL]		Ti	50
Ba [200 µg/mL]		Ba	200
Ca [200 µg/mL]		Ca	200 [1]
Fe [20 µg/mL]		Fe	20 [1]
Mo [20 µg/mL]		Mo	20
Se [5 µg/mL]		Se	5 [1]
Sr [50 µg/mL]		Sr	50
V [50 µg/mL]		V	50
K [10,000µg/mL]	480 µL	K	4800 [1]
Na [10,000µg/mL]	480 µL	Na	4800 [1]
Mg [10,000µg/mL]	80 µL	Mg	800 [1]
Ca [10,000µg/mL]	80 µL	Ca	800 [1]
Au [1000 µg/mL]	200 µL	Au	200

[2] The final LOQ/ICVL solution contains a 2% HNO_3 & 5% HCl acid matrix.

[1] The following is a list of the final concentration of analytes from multiple stock standards:

Element	Concentration [µg/L]	Element	Concentration [µg/L]
Calcium	1000	Potassium	5000
Iron	100	Selenium	10
Lead	5	Sodium	5000
Magnesium	1000		

APPENDIX P3-7
SOP FOR THE PREPARATION AND ANALYSIS OF MERCURY BY MANUAL COLD VAPOR TECHNIQUE BY SW-846 METHOD 7470A
(S-LI-M-002-REV.01)

STANDARD OPERATING PROCEDURE

PREPARATION AND ANALYSIS OF MERCURY BY MANUAL COLD VAPOR TECHNIQUE

Reference Methods: EPA Method 245.1 and SW-846 Methods 7470A and 7471B

SOP Number:	S-LI-M-002-rev.01
Effective Date:	Date of Final Signature
Supersedes:	S-LI-M-002-rev.00

APPROVALS

General Manager	6/1/15 Date
Quality Manager	6/1/15 Date
Department Manager	6/1/15 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose/Identification of Method

1.1. The purpose of this SOP is to outline the preparation and analysis steps for Mercury by Manual Cold Vapor Technique for aqueous samples per EPA Method 245.1 and SW-846 Method 7470A, and for solid samples per SW-846 Method 7471B.

2. Summary of Method

2.1. Methods 7470A and 245.1: A prepared liquid sample is reduced with SnCl2 to form elemental mercury vapor. The mercury vapor flows into a liquid gas separator where argon is introduced to carry the vapor through a drying tube to remove water. The dry mercury vapor passes through an optical cell, positioned in the light path of a mercury lamp. The mercury lamp emits radiation that is filtered allowing a wavelength of 253.7 nm to pass. The absorbance of light at this wavelength, by the mercury vapor is measured as a function of mercury concentration.

2.2. Method 7471B: A weighed portion of a sample is digested with acid, followed by oxidation with potassium permanganate, potassium persulfate and reduced with hydroxylamine. Stannous chloride is added to form elemental mercury vapor. The mercury vapor flows into a liquid gas separator where argon is introduced to carry the vapor through a drying tube to remove water. The dry mercury vapor passes through an optical cell, positioned in the light path of a mercury lamp. The mercury lamp emits radiation that is filtered allowing a wavelength of 253.7 nm to pass. The absorbance of light at this wavelength, by mercury vapor is measured as a function of mercury concentration.

3. Scope and Application

3.1. In addition to the requirements of this SOP, the guidelines in the Pace Quality Manual must be observed.

3.2. **Personnel**: This SOP is applicable to all personnel involved in the preparation and/or analysis of samples for the Mercury methods listed.

3.3. **Parameters**: Mercury.

4. Applicable Matrices

4.1. The aqueous methods (7470A or 245.1) are applicable to surface waters, groundwater, wastewaters and saline waters. The solid method (7471B) is applicable to the determination of mercury in soils, sediments, sludge and other solid matrices.

5. Limits of Detection and Quantitation

5.1. MDL studies are performed annually by the analysis of seven low level standards at three to five times the expected MDL and calculated by the procedure defined in 40CFR Part 136 Appendix B.

5.2. Current Method Detection Limits (MDLs) are on file and available by request from the Quality Manager..

5.3. Methods 7470A and 245.1: The standard reporting limit for mercury is 0.2 ug/L. The approximate achievable detection limit is 0.1 ug/L. Method 7471B: The approximate achievable detection limit is 0.05 mg/kg.

6. Interferences

6.1. Sulfide interference is eliminated by the addition of potassium permanganate. Concentrations as high as 20mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

6.2. High concentrations of copper cause interference. Copper concentrations as high as 10mg/L had no effect on recovery of mercury from spiked samples.

6.3. Samples that are high in chlorides, such as waters, brines and some industrial effluents, are known to cause interference. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation of 253 nm. This free chlorine must be removed before analysis. These samples may require additional permanganate. An excess of hydroxalmine sulfate reagent should be added to remove the free chlorine.

6.4. Interference from certain volatile organic materials which absorb at this wavelength is also possible. A preliminary run without reagents can determine if this type of interference is present.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Aqueous samples: The samples are in new plastic bottles. The samples do not need to be shipped iced however care should be taken from temperature extremes.

7.2. Solid samples: The samples are collected in new glass or plastic wide mouth containers. The samples are shipped and stored at 4°C (± 2°C).

7.3. Aqueous samples must be preserved with nitric acid to a pH of 2 or lower upon collection of the sample. For method 245.1 samples, if the lab determines that the sample has not been preserved to a pH <2, the lab must preserve the sample to a pH <2 and then must wait 24 hours after preservation to start prepping the sample for analysis.

7.4. All samples: The holding time is 28 days from sample collection.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. Analysis Batch: A sequence of samples, which are analyzed within a 24 hour period and include no more than 20 field samples. An Analysis Batch must also include all required QC samples, which do not contribute to the maximum field sample total of 20.

8.3. Laboratory Control Sample (LCS): Aqueous and solid laboratory control samples must be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the samples.

8.4. Calibration Standard (CAL): A solution prepared from the primary dilution standard solution(s) or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

8.5. Initial Calibration Standards: A series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

8.6. Initial Calibration Verification Standard (ICV)/ Quality Control Sample (QCS): A solution of a second source different than that of the calibration standards, which is analyzed initially, prior to any field sample analyses, which verifies the previously established calibration curve.

8.7. Instrument Performace Check (IPC): For samples being analyzed by method 245.1, a solution of the same source as the calibration standards, and near the midpoint, will be analyzed.

8.8. Continuing Calibration Verification Standard (CCV): A CAL solution which is analyzed after every tenth field sample analyses, not including QC samples, which verifies the previously established calibration curve and confirms accurate analyte quantitation for the previous ten field samples analyzed. The concentration for the continuing calibration check standards should be either at a middle calibration level or at the highest calibration level.

8.9. Field Duplicates (FD): Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

8.10. Laboratory Duplicate (LD) -- Two sample aliquots (LD1 and LD2), taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicate precision associated specifically with the laboratory procedures by removing variation contributed from sample collection, preservation and storage procedures.

8.11. Laboratory Fortified Sample Matrix (LFM)/Spike – An aliquot of an environmental field sample to which a known quantity of mercury is added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical result (when compared to the result for the LFB). The background concentrations of mercury, in the sample matrix, must be initially determined in a separate aliquot and the measured value in the LFM corrected for this background concentration. One per batch will be performed. If the batch is for method 245.1, an LFM will be performed at a frequency of 10% of the samples.

8.12. Linear Calibration Range (LCR) – The concentration range over which the instrument response is linear.

8.13. Laboratory Reagent Blank (LRB) – An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, filtration and reagents that are used with other samples. The LRB is used to determine if mercury or other interferences are present in the laboratory environment, the reagents, or the apparatus.

8.14. Limit of Detection (LOD): An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte-and matrix-specific and may be laboratory-dependent. According to NELAC/TNI, the LOD equates with the MDL.

8.15. Limit of Quantitation: (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The lowest concentration that produces a quantitative result within specified limits of precision and bias.

8.16. Linear Dynamic Range (LDR): The concentration range over which the instrument response is linear. Will be determined annually.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Equipment and Supplies:

Equipment/Supply	Description	Vendor/ Item # / Description
Automated Mercury analyzer	Leeman Hydra or Hydra II with WinHg software	

Equipment/Supply	Description	Vendor/ Item # / Description
Mercury lamp		Catalog # 217-00003
Drying Tube		
Liquid/gas separator		
Pump tubing		
Autosampler tubes	14mL disposable polypropylene	
Hot Block digestor	Capable of maintaining temperature of 95°C for two hours	
Digital thermometer (mercury free)	Standardized against a thermometer certified at 95°C	
Digestion vessels	Polypropylene	Catalog # SC475
Ribbed watch glasses		Catalog # SC505
FilterMates		Catalog # SC0401
Analytical balance		
Spatula		
Weighing vessel or paper		

10. Reagents and Standards

10.1. Reagents and Standards:

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
De-ionized (DI) Water	ASTM Type II	
Nitric Acid	Concentrated; low mercury	
Sulfuric Acid	Concentrated; low mercury	
Potassium Permanganate		
Potassium Permanganate solution (5% w/v)	Dissolve 100g of potassium permanganate to a final volume of 2000mL of distilled water	N/A
Potassium Persulfate		
Potassium Persulfate solution (5% w/v)	Dissolve 100g of potassium persulfate to a final volume of 2000mL of distilled water	N/A
Sodium Chloride		
Hydroxylamine Sulfate		
Sodium chloride/ hydroxylamine sulfate solution	Dissolve 240g of sodium chloride and 240g of hydroxylamine sulfate in distilled water and dilute to a final volume of 2000mL	N/A
Stannous Chloride		
Stannous Chloride solution	Add 75g of stannous chloride into 100mL concentrated hydrochloric acid. Heat gently to dissolve. Dilute to 500mL with distilled water.	N/A
Mercury stock solution	1 mg/L	High Purity or ERA
Mercury check standard	1 mg/L	Ultra Scientific

10.2. Calibration Standards

10.2.1. For aqueous solutions make a 5x dilution of Cal standard and 2nd source standard(2mL→10mL)

	Aqueous		
Std	Vol of Int. (mL)	Final Vol (mL)	Conc. ppb
0.0	0.00	20.0	0.00
0.2	0.02	20.0	0.20
1.0	0.10	20.0	1.00
2.0	0.20	20.0	2.00
5.0	0.50	20.0	5.00
10	1.00	20.0	10.00
*ICV	0.10	20.0	1.00
CCV	0.50	20.0	5.00
CRI	0.02	20.0	0.20

	Soil		
Std	Vol of Int. (mL)	Final Vol (mL)	Conc. ppb
0.0	0.00	50.0	0.00
0.2	0.01	50.0	0.20
1.0	0.05	50.0	1.00
2.0	0.10	50.0	2.00
5.0	0.25	50.0	5.00
10	0.50	50.0	10.00
*ICV	0.05	50.0	1.00
CCV	0.25	50.0	5.00
CRI	0.01	50.0	0.20

* 2nd Source

.

10.3. Preparation Blank (PBW):

10.3.1. For aqueous samples: Add 20 mL of distilled water to a 50 mL digestion vessel.

10.3.2. For solid samples: Add 2 glass beads to a 50 mL digestion vessel. Results will be calculated as if 0.30 g aliquot was weighed.

10.4. For solid samples: Laboratory Control Sample (LCS): Weigh a 0.2-0.3 g portion of a certified solid reference material (with a known true value) and digest and analyze according to sample procedures.

10.5. For aqueous samples: Laboratory Control Sample (LCS) is the same solution as the ICV.

10.6. For aqueous samples - 1.0 ug/L matrix spike: Add 100 uL of the 200 ug/L stock standard to 20mL of sample.

10.7. For soil samples - 1.0 ug/L matrix spike: 50 uL of the 1 mg/L stock standard to the soil sample.

10.8. 10% HCl Rinse: Dilute 100mL of conc. hydrochloric acid to 1 liter of distilled water.

10.9. Aqua Regia: Prepare immediately before use by adding 3 parts concentrated HCl to 1 part of HNO_3.

10.10. Sulfuric acid, 0.5N: Dilute 14mL concentrated sulfuric to 1 liter of deionized water.

11. Calibration and Standardization

11.1. Analytical Sequence:

11.1.1. Following calibration, the analytical run should begin with an Initial Calibration Verification Standard (ICV). If there are method 245.1 samples in the run, an IPC will be analyzed as well.

11.1.2. Immediately following the ICV(IPC), the Initial Calibration Blank (ICB) shall be analyzed.

11.1.3. Following the ICB, a continuing calibration verification standard (CCV) is analyzed.

11.1.4. A continuing calibration blank (CCB) follows the CCV.

11.1.5. A standard at the reporting limit of 0.20ug/L (CRA/LOQ standard) is run and counted as a regular sample.

11.1.6. A preparation blank (PBW) is then analyzed. Note: CCVs and CCBs must then be analyzed at a frequency of not less than 10%. Also, the run sequence must end with a CCV and CCB.

11.1.7. Up to 18 samples can be analyzed (use the 6 digit client ID to designate a sample).

11.1.8. A duplicate analysis is performed.

11.1.9. A matrix spike analysis is performed.

11.1.10. A CCV and CCB must follow.

11.1.11. The analytical sequence is to be recorded in the mercury run logbook

11.2. Initial Calibration: A blank and 5 standards are analyzed.

11.3. Pour digested calibration standards from digestion vessels into standard auto sampler cups.

11.4. Calibration acceptance criteria:

Calibration Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	• Run daily. • Establish initially and annually thereafter. • When continuing calibration check standards fail. • When a change in procedure or instrument.	• Correlation Coefficient ≥ 0.995.	• Evaluate the standards and equipment and reanalyze the initial calibration.
Initial Calibration Verification Standard (ICV)	• Immediately after the initial calibration. • Prepared from a source different than the calibration standards. • The daily sample sequence is to be opened with an ICV.	• 90-110% recovery	• Reanalyze and recalibrate if necessary. • Sample analysis cannot begin until a compliant Initial Calibration and ICV meet acceptance criteria. • Samples analyzed after a non-compliant ICV must be reanalyzed.
Instrument Performance Check (IPC)	• Immediately after the initial calibration. • Prepared from a source same than the calibration standards. • Only required if samples on the run are analyzed by method 245.1. • Will follow the ICV.	• 95-105% recovery	• Reanalyze and recalibrate if necessary. • Sample analysis cannot begin until a compliant Initial Calibration and ICV meet acceptance criteria. • Samples analyzed after a non-compliant ICV must be reanalyzed.
Limit of Quantification Verification Standard	• Analyzed immediately after the ICAL and	• 70-130% recovery • Or client data quality	• Evaluate standard preparation.

Calibration Item	Frequency	Acceptance Criteria	Corrective Action
(LOQ)/Reporting Limit Verification (RLV)	ICV. • Prepared at the same concentration as the lowest calibration standard to verify accuracy at the reporting level. • Processed under the same conditions as samples and goes through all the steps of the analytical procedure. • If the reporting level is higher than the lowest calibration standard, the LOQ solution may be prepared at the reporting level.	objectives, if such exist.	• Reanalyze and recalibrate if necessary.
Continuing Calibration Verification Standard (CCV)	• One per 10 samples (or daily, if fewer than 10 samples) • The analytical sequence must end with a compliant CCV.	• 90-110% recovery	• Samples that are not bracketed with compliant CCVs are to be reanalyzed. • If a re-analysis of a CCV is still non-compliant, the standards and equipment must be evaluated, prepare new standards if necessary and recalibrate.
Initial and Continuing Calibration Blanks (ICB and CCB)	• ICB after ICV. • CCB per 10 samples (or daily, if fewer than 10 samples) – to be analyzed immediately after each CCV. • The analytical sequence must end with a compliant CCB.	• Should not contain target analytes or interferences at concentrations that impact the analytical results for sample analyses. • The absolute value of the concentration of target analytes should not be greater than the CRDL.	• If the CCBs contain target analytes greater than the CRDL, the blank needs to be re-analyzed. If the re-analysis passes criteria, then reanalyze the associated samples. • If the blank still fails, the system needs to be evaluated for the source of contamination. Re-analysis and recalibrate if necessary.

12. Procedure

12.1. Aqueous Digestion Procedure:

12.1.1. A record of all mercury digestions including initial volumes, final, volumes, dilution factors, sample IDs and spikes are to be entered in the LIMS system.

12.1.2. Transfer 20mL of sample, or an aliquot diluted to 20mL, containing no more than 1.0ug of mercury, to a 50mL digestion vessel.

12.1.3. Add 1.0 mL of conc. sulfuric acid.

12.1.4. Add 0.5 mL of conc. nitric acid.

12.1.5. Add 3 mL of potassium permanganate solution and let stand for 15 minutes. Note: Add additional permanganate if the purple color doesn't persist during this time.

12.1.6. Add 1.6 mL of potassium persulfate and cover with watch glass or reflux cap.

12.1.7. Heat for 2 hours in the Hot Block at 95±5°C. Cool samples.

12.1.8. Add 1.2 mL of sodium chloride-hydroxlamine sulfate solution to reduce excess permanganate.

12.2. Solid Digestion Procedure:

12.2.1. A record of all mercury digestions including sample weights, final volumes, dilution factors, sample IDs and spikes are to be recorded entered in the LIMS system.

12.2.2. Weigh a 0.25-0.30 g portion of a well homogenized sample and place in the bottom of a digestion vessel.

12.2.3. Add 2.5 mL deionized water and 2.5 mL aqua regia. Heat for 2 minutes at 95 +/- 3°C.

12.2.4. Allow samples to cool and add 25 mL deionized water.

12.2.5. Add 7.5 mL of potassium permanganate solution. Let stand 15 minutes and add additional portions (if needed). Purple color must persist for 15 minutes. Remember to add the same amount to standards and blanks. Mix.

12.2.6. Heat samples at 95 +/- 3°C for 30 minutes.

12.2.7. Cool and add 3.0 mL sodium chloride/hydroxylamine sulfate solution.

12.2.8. Cool samples.

12.2.9. Under a hood, bring to 50mL final volume.

12.3. Sample Analysis:

12.3.1. Pour digested samples from digestion vessels to autosampler tubes in the order of the analysis run sequence.

12.3.2. When analysis is finished, place reductant tube on the 10% HCL rinse bottle and flush system for 5 minutes. Then replace the HCL with distilled water and flush for an additional 5 minutes.

12.3.3. Return to instrument operation screen and turn off gas and peristaltic pump. Release the cassettes holding the peristaltic pump tubing to prolong tubing life.

12.3.4. Absorbance (peak height) of mercury is measured as a function of mercury concentration. Results are reported in ug/L.

12.3.5. Results must fall within the range of the calibration graph or standards used. If a result is greater than 10ug/L, a dilution must be performed.

12.3.6. If a dilution is performed, the dilution factor must be applied to the sample value to get a final result.

12.3.7. All quality control requirements must be met before analysis results can be reported.

13. Quality Control

13.1. Quality Control items:

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	• One per batch, • Or 1 per 20 samples, whichever is more frequent. • Processed with and under the same conditions as samples and goes through all the steps of the analytical procedure.	• Should not contain target analytes or interferences at concentrations that impact the analytical results for sample analyses. • Absolute value of concentration of target analytes should not be greater than CRDL. • For 245.1 verify if the blank is greater than 10% of the sample or 2.2 times the MDL	• If the blank contains target analytes greater than the CRDL, the blank needs to be re-analyzed. If the re-analysis passes criteria, then reanalyze the associated samples. • If the blank still fails, the system needs to be evaluated for the source of contamination and affected samples reanalyzed. • If reanalysis of samples is not possible, report data flagged to indicate method blank contamination.
Laboratory Control Sample (LCS) (same as ICV)	• One per batch, or 1 per 20 samples, whichever is more frequent. • Prepared from a source different than the calibration standards – same as the ICV)	• When evaluated as an ICV, the criteria of 90-110% recovery must be met. • When evaluated as an LCS, method 7470A and 7471B: 80-120% recovery must be met unless the LCS is a solid CRM, and then the manufacturer's acceptance limits can be used. • Method 245.1: 85-115%	• Reanalyze and samples associated with the LCS/ICV must be reanalyzed. • If reanalysis of samples is not possible, report data flagged to indicate LCS failed recovery.
Matrix Spike Sample (MS)	• Methods 7470A and 7471B: MS/MSD per 20 samples or daily, if fewer than 20 samples. • Method 245.1: MS per 10 samples (10%)	• Method 7470A: 75-125% recovery; unless analyte concentration is >4x the spike level. • Method 245.1: 70-130% • Method 7471B: 80-120% recovery	• If the recovery is outside the limits, repeat the sample, duplicate and matrix spike analysis once. If it passes, then report samples. • Check for errors in calculation and spike preparation. • If the matrix spike still exceeds the limits, but the LCS/ICV has acceptable recovery, then the method is in control and sample

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
			matrix effects are likely the cause. The data should be qualified in the case narrative or using QC notes in the LIMS for non-package work.
Sample Duplicate (DUP)	• One per 20 samples or daily, if fewer than 20 samples.	• ± 20% RPD • For result values less than five times the PQL, a control limit of ± the PQL will be used.	• Check sample label, calculation, dilution factors. • If results are grossly different (i.e., very high result and non-detect) re-analyze to confirm.

14. Data Analysis and Calculations

14.1. Absorbance (peak height) of mercury is measured as a function of mercury concentration. Results are reported in ug/L.

14.2. Results must fall within the range of the calibration graph or standards used. If a result is greater than 10ug/L, a dilution must be performed.

14.3. If a dilution is performed, the dilution factor must be applied to the sample value to get a final result.

14.4. All quality control requirements must be met before analysis results can be reported.

14.5. A second review is performed by the department supervisor, or designee. The run is signed and dated by the supervisor, or designee, upon completion of the review.

14.6. Calculation: Percent recovery for standard (ICV, CCV):

$$\%\ \text{Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100\%$$

14.7. Calculation: Percent Recovery for matrix spike:

$$\%\ \text{Recovery} = \frac{\text{Spiked Sample Result} - \text{Sample Result}}{\text{Spike Added}} \times 100\%$$

14.8. Calculation: Relative Percent Difference (RPD):

$$\text{RPD} = \frac{\text{Sample Result} - \text{Duplicate Result}}{\text{Average Result}} \times 100\%$$

14.9. Mercury mg/kg (dry weight):

$$\text{Hg (mg/kg) Conc.} = \frac{\text{Result (ug/L)} \times \text{Final Volume(L)}}{\text{Weight of Sample (g)} \times \frac{\%\text{TS}}{100\%}}$$

Where %TS = percent total solids

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Reported data for all analytical samples and quality control checks as outlined in Methods 7470A, 245.1 and 7471B must adhere to the criteria and guidelines as specified by the protocol. Reporting limits, check standards and all related data generated and collected by an analytical procedure must be documented for quality control purposes and maintained as an assessment to the usability, validity and overall integrity of the data.

15.2. See Sections 11.4 and 13.1.

15.3. The analyst is responsible for generating the data and also is the initial individual to review the data. The review must include at least the following procedures:

15.3.1. Inspection of records in run log for completeness;

- Standard and reagent lot numbers, support equipment, spike amounts, calculations, dilution factors, container/bottle used for analysis, reporting limits.

15.3.2. Determination of whether the results meet the laboratory-specific quality control criteria;

15.3.3. Checks to determine consistency with client/project-specific measurement quality objectives (MQOs) if such exists;

15.3.4. Checks to ensure that the appropriate sample preparatory and analytical SOPs and methods were followed, and that chain-of-custody and holding time requirements were met;

15.3.5. Checks to ensure that all calibration and quality control requirements were met;

15.3.6. Checks for complete and accurate explanations of anomalous results, corrective action, and the use of data qualifiers in the case narrative or LIMS QC notes.

15.3.7. Record of any non-standard condition of the test, test environment, sample or any deviation from standard operating procedure.

15.4. If analysis is deemed acceptable, data will be imported into the LIMS.

15.4.1. Another review is performed for correctness of results, including prep factors, dilution factors, spike amounts and recoveries, sample and QC references and appropriate qualifiers.

15.4.2. If additional information is to be communicated to the data user about a particular sample, a “QC Note” is entered by the analyst.

15.4.3. Once data has been reviewed in the LIMS, the analyst or supervisor will “QA” the sequence which indicates the data has been reviewed and is ready for reporting.

16. Corrective Actions for Out-of-Control Data

16.1. Corrective action begins with the analysts who are responsible for knowing when the analytical process is out of control.

16.2. Corrective actions should be initiated for out of control events such as when QC checks exceed acceptance limits or exhibit trending, holding time failures, loss of sample, equipment malfunctions or evidence of sample contamination.

16.3. See Sections 11.4 and 13.1

16.4. Non-conformances are noted by the preparation of a report containing the following:

16.4.1. Parameter/ SDG;

16.4.2. Client ID. / Dates collected and received;

16.4.3. Non-conformance;

16.4.4. Root cause;

16.4.5. Corrective action taken;

16.4.6. Action to prevent re-occurrence / follow-up;

16.4.7. Non-conformance forms are initiated by the analyst and forwarded to the department supervisor, project manager and quality assurance officer;

16.4.8. Client notification is handled by the project manager if applicable.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Samples that have been analyzed during a non- compliant run will be rejected. The samples will be reanalyzed after the problem is resolved and the system recalibrated.

17.2. If the sample matrix is such that interferences prevent the system from meeting calibration and quality control requirements, the client will be notified with a course of action determined.

17.3. All problems associated with the analysis of a sample group will be outlined in the case narrative of the data package or with the use of QC Notes in the LIMS, if required.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. Internal method performance is established and monitored with use of the following:

18.2.1. Method Detection Limit studies

18.2.2. Demonstration of Capability

18.2.3. Precision and accuracy

18.2.4. Positive and negative controls

18.2.5. Measurement of sample matrix effects

18.2.6. Control charts.

18.3. Instrument Detection Limits (IDLs) will be performed every 6 months. IDLs are 3 times the average standard deviations, obtained on 3 non-consecutive days, from the analysis of a standard at a concentration of 3-5 times the estimated IDL, run consecutively seven times.

19. Method Modifications

19.1. Volumes have been reduced to allow the use of a digestion block and to minimize waste. The chemistry/ratios of reagents have not been modified.

20. Instrument/Equipment Maintenance

20.1. For information and guidance related to instrument/equipment maintenance, refer to the instrument manuals and other documents listed in the Reference section.

21. Troubleshooting

21.1. Troubleshooting procedures come from experience and training. Problems that the analyst encounters may be elevated to the department supervisor. Other means of problem resolution may come from review of prior related corrective action reports, instrument maintenance records, internal and external audits and lab notes.

22. Safety

22.1. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposed to these chemicals must be reduced to the lowest possible level by personal protection and engineering measures. MSDS are available for all chemicals used in the lab and are available for review.

22.2. Safety glasses, a lab coat, and disposable gloves must be worn when handling chemicals and samples.

22.3. Sample handling should be conducted in fume hoods.

23. Waste Management

23.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste.

23.2. Procedures for handling waste generated during this analysis are addressed in the current waste management SOP.

23.3. All wastes deemed toxic are collected in the laboratory and are disposed by professional companies licensed in waste disposal.

24. Pollution Prevention

24.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Use pollution prevention techniques in laboratory operation whenever feasible.

24.2. Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

24.3. Control releases from hood and bench operations and comply with discharge permits and regulations, hazardous waste identification rules, and land disposal restrictions.

24.4. The generated waste has to be disposed in a manner not to cause pollution.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. "Test Methods for Evaluating Solid Waste," EPA/SW-846, USEPA, Water Characterization Branch, Update IV, EPA SW846 methods 7470A and 7471B.

25.5. EPA Method 245.1.

25.6. ADMIN02: Computers and Programs.

25.7. ME001: Hydra Automated Mercury Analyzer Manual 150-00210 Rev C.

25.8. ME003: Maintenance Manual for PS Autosampler.

25.9. ME007H: Leeman Labs PS200 Automated Mercury Analyzer Master Diskette, Rev 3.001; Part # 810-00018.

25.10. ME015: Leeman Labs Win Hg Version 1.5; Part # 810-00039.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-LI-M-002-rev.00	Converted to Pace SOP format. Removed all DoD references. Added Section 8.7 IPC for 245.1	5/15/15
S-LI-M-002-rev.01	Added Calibration table and updated for standard preparation.	6/1/15

APPENDIX P3-8
SOP FOR TOTAL RECOVERABLE OIL AND GREASE ANALYSIS IN WATERS N-HEXANE EXTRACTABLE MATERIAL (SGT-HEM) BY EXTRACTION AND GRAVIMETERY BY EPA 1664A (S-LI-I-016-REV.02)

STANDARD OPERATING PROCEDURE

TOTAL RECOVERABLE OIL AND GREASE AND PETROLEUM HYDROCARBON ANALYSIS IN WATERS N-HEXANE EXTRACTABLE MATERIAL (SGT-HEM) BY EXTRACTION AND GRAVIMETERY

Reference Method: EPA 1664A

Local SOP Number:	S-LI-I-016-rev.02
Effective Date:	Date of Final Signature
Supersedes:	S-LI-I-016-rev.01

APPROVALS

[signature]	7/23/15
General Manager	Date
[signature]	7/23/15
Quality Manager	Date
[signature]	7/23/15
Department Manager	Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Identification of Test Method

1.1. This Standard Operating Procedure (SOP) describes the procedure for the analysis of Total Recoverable Oil and Grease and Petroleum Hydrocarbon Analysis in Waters N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetery by EPA 1664A.

2. Summary of Method

2.1. A 1-L sample is acidified to a low pH (<2) and serially extracted with n-hexane in a solid phase extractor. The extract is dried in a solvent evaporation system.

2.2. The solvent is evaporated from the extract and the residue (HEM) is weighed. If the HEM is to be used for the determination of petroleum hydrocarbons (SGT-HEM), the HEM is re-dissolved in n-hexane.

2.3. For SGT-HEM determination, an amount of silica gel proportionate to the amount of HEM is added to the solution containing the re-dissolved HEM to remove absorbable materials. The solution is filtered, then evaporated, and the SGT-HEM is weighed.

2.4. The parameters of Oil and Grease (HEM) and Petroleum Hydrocarbon (SGT-HEM) are defined by the method. The measurement may be subject to interferences and the results should be evaluated accordingly.

3. Scope and Application

3.1. This method is applicable to the analysis of hexane extractable materials (HEM) (oil and grease) from waters and wastes, as well as SGT-HEM, silica gel treated hexane extractable materials (Petroleum Hydrocarbons).

3.2. The method is not applicable to measurement materials that volatilize at temperatures below ~ 40°C. Petroleum fuels from gasoline through #2 fuel oil may be partially lost in the solvent removal operation.

3.3. Some crude oils and heavy fuel oils contain materials that are not soluble in hexane. The reference method (EPA 1664) is capable of measuring HEM in the range of 5 to 1000 mg/l, and may be extended to higher levels by analysis of smaller sample volumes collected separately. Reference a) lists a method detection limit (MDL) of 1.4 mg/l and a minimum level of quantitation (ML) as 5.0 mg/l.

4. Applicable Matrices

4.1. This method is applicable to the analysis of hexane extractable materials (HEM) (oil and grease) from waters and wastes, as well as SGT-HEM, silica get treated hexane extractable materials (Petroleum Hydrocarbons).

5. Limits of Detection and Quantitation

5.1. MDL studies are performed annually by the analysis of seven low level standards at three to five times the expected MDL and calculated by the procedure defined in 40CFR Part 136 Appendix B. The MDLs define the lowest levels, where positives will be found with 99 percent confidence with the particular analytical method in clean media.

5.2. For HEM and SGT-HEM in this method, the method detection limit (MDL) is 1.4 mg/L and the minimum level of quantitation (ML) is 5.0 mg/L.

5.3. The lab must be able to achieve an MDL that is 1.4 mg/L or less. Current Method Detection Limits (MDLs) are on file and available by request from the Quality Manager

5.4. The routine lab reporting limit is referred to as the PQL (Practical Quantitation Limit). The PQL is 5 mg/L.

6. Interferences

6.1. Solvents, reagents, glassware, and other sample processing supplies may yield artifacts that affect results.

6.2. Sodium sulfate and silica gel particulates may inflate results for HEM and SGT-HEM, remove by passing the extract through the filter paper.

6.3. The method is not applicable to measurement materials that volatilize at temperatures below ~ 45°C. Petroleum fuels from gasoline through #2 fuel oil may be partially lost in the solvent removal operation.

6.4. Some crude oils and heavy fuel oils contain materials that are not soluble in hexane.

6.5. Interferences extracted from samples are source dependent and many contain substances which interfere with the extraction procedure, a smaller sample size may be required to be utilized for analysis.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Collect 1L of representative sample in a glass bottle.

7.2. Preservation by reducing the pH to <2 with H_2SO_4 or HCL and refrigeration or icing to 4°C.

7.3. The NYSDEC ASP holding time is 26 days from VTSR

7.4. The EPA holding time is 28 days from collection.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual.

9. Equipment and Supplies

Equipment/Supply	Description
Analytical Balance	Capable of weighing 0.1mg
Volumetric flask	100mL glass Class A
Volumetric pipette	Class A
Filter paper	Whatman No. 40 or equivalent
Vacuum	
Pump	
Magnetic stirrer	Teflon coated stirring bars
Horizon SPE-DEX 3000XL Solid Phase Extractor	Collection vessel holder Disk holder Bottle Cap adapters Sample inlet valves Solvent reservoirs Water waste collection vessel Solvent waste collection vessel Automatic controller
Horizon SpeedVap III	Solvent Evaporation system
Vials	40mL
Separatory funnels	125 mL
Erlenmeyer flasks	125mL
Aluminum weighing pans	
Plastic transfer pipets	
pH paper	Full range
Extraction disks	47mm (Horizon P/N 1664-47-HT), 90mm (Horizon P/N 1664-90-HT)
Phase separator paper	
Glass rods	Fisher Capillary tubes Cat.# 22-260-973
Food Coloring	

10. Reagents and Standards

10.1. Hydrochloric acid, 1:1: Mix equal volumes of concentrated HCl and distilled water.

10.2. n - Hexane, ACS grade low residue

10.3. Methanol, ACS grade

10.4. Sodium sulfate, anhydrous crystal.

10.5. Silica gel, 60-200 mesh, Davidson Grade 950 or equivalent. Should contain 1-2% water as defined by residue test at 130°C. Adjust by overnight equilibration if needed.

10.6. Hexadecane: 98% minimum purity

10.7. Stearic acid: 98% minimum purity

10.8. Oil and Grease standard solution (TV=40 mg/l) currently purchased from 2 outside sources.

10.9. Hexadecane/stearic acid (1:1) spiking solution. Currently purchased commercially prepared. To make: Prepare in acetone at a concentration of 4 mg/ml each.

10.9.1. Place 400 ± 4 mg stearic acid and 400 ± 4 mg hexadecane in a 100 ml volumetric flask and fill to the mark with acetone. The solution may require warming for dissolution.

10.9.2. Transfer the solution to a vial and mark solution level on the vial and store in the dark at room temperature.

10.9.3. Immediately prior to use, verify the level on the vial and bring to volume with acetone, if necessary. The solution may require warming for dissolution.

10.10. Sulfuric acid- H_2SO_4 - mix 1 part H_2SO_4 and 3 parts reagent water.

10.11. Reagent water, ASTM Type I- deionized distilled water free of trace elements

11. Calibration and Standardization

11.1. Calibrate the analytical balance at 2 mg and 1000 mg using class "S" weights, and note weights in logbook.

11.2. Calibration shall be within ± 10% (i.e. ±0.2 mg) at 2 mg and ± 0.5% (i.e. ±5 mg) at 1000 mg. If values are not within these limits, recalibrate the balance.

12. Procedure

12.1. Preparation of the sample batch

12.1.1. Bring samples to room temperature, including MS/MSD if sample volume allows. (If not enough sample volume for MS/MSD, perform a LFB/LFBD).

12.1.2. Measure approximately 1L (+/- 50mL) of reagent water to a clean sample bottle for the method blank, LCS (and LFB/LFBD if applicable).

12.1.3. Fortify LCS/LFB/MS with Hexadecane/stearic acid spiking solution.

12.1.4. For samples, verify that the pH is < 2 using the following procedure:

- Dip glass rod into the well mixed sample.
- Withdraw the stirring rod and allow a drop of the sample to fall on or touch the pH paper.
  - ú NOTE: Do not dip the pH paper into the bottle or touch it to the sample on the lid.
- If the sample is at neutral pH, add 5-6 mL of HCL or H_2SO_4 solutions (see Section 10) to the sample. If the initial pH is higher, use a proportionately larger amount of HCL or H_2SO_4 .
- Replace the cap and shake the bottle to mix thoroughly.

- Re-check the pH, as above.
- To ensure that no extractable material is lost on the stirring rod, add the glass rod to the sample bottle. The rods are small enough not to interfere with the sample extraction.
- Add the appropriate amount of HCL or H_2SO_4 solution to the MB, LCS, LFB/LFBD and MS/MSD to adjust to pH < 2.

12.2. Instrument Preparation

12.2.1. Turn on automatic controller on the SPE, make sure nitrogen gas is delivering at lease 120 psi to the system, and turn on vacuum pump.

12.2.2. The system must be purged before samples are extracted.

12.2.3. Insert an empty disk holder assembly into the assembly holder.

12.2.4. Twist an empty 40 ml vial onto the collection vessel holder and put onto instrument.

12.2.5. Onto an empty 1 liter bottle put a sample inlet valve and invert onto the sample platform of the SPE. (Do this for all 3 stations of the SPE).

12.2.6. Set all 3 stations to Method 30 (Purge method) using the automatic controller. Run all stations and repeat.

12.2.7. Remove the empty bottle, remove the empty disk holder, remove the collection vessel and dispose of the solvent in the proper waste receptacle.

12.2.8. Put a clean collection vessel (40 ml vial or separatory funnel) onto holder and connect to SPE.

12.2.9. Put a clean 47 mm extraction disk in the disk holder and connect to SPE. (If the sample is dirty, use a 90 mm extraction disk).

- The extraction disk used must be noted in the logbook. QC must be performed for each disk type.

12.2.10. Mark the water level on the bottle for volume determination. A standard bottle filled to the neck contains 1L and need not be measured.

12.2.11. Connect a sample inlet valve onto each sample bottle. If the sample bottle has a wide mouth, first connect a cap adapter to the bottle and then connect the inlet valve to the adapter.

12.2.12. Invert the sample bottle onto the SPE sample platform.

12.2.13. Note the extraction station for each sample in the logbook. QC Samples must be rotated throughout out the extraction stations.

12.3. Extraction: Method 26 is used for all extractor units. Use Method 28 in place of 26 for all stations using 90 mm disks.

12.3.1. Method 26 prewets the disk, processes the sample and rinses with hexane.

Method 26			
Prewet Hexane		**Prewet Methanol**	
Dispense	**6 seconds**	**Dispense**	**6 seconds**
Saturate	**1 second**	**Saturate**	**1 second**
Soak	**30 seconds**	**Soak**	**30 seconds**
Drain	**1 minute**	**Drain**	**3 seconds**
	Air Dry	**3:00 Minutes**	

Rinse	**Dispense**	**Soak**	**Elute**
1.Hexane	**4 seconds**	**45 seconds**	**0 seconds**
2.Hexane	**4 seconds**	**45 seconds**	**45 seconds**
3.Hexane	**4 seconds**	**45 seconds**	**0 seconds**
4.Hexane	**4 seconds**	**45 seconds**	**45 seconds**

12.3.2. Set the automatic controller to Method and run the samples.

12.3.3. When samples are completed the controller will read IDLE for each station.

12.3.4. Method 26 conditions the disk with methanol during the prewet step to remove residual water from the disk and then rinses with hexane to remove the remaining oil and grease.

Method 26			
Prewet Hexane	**0 seconds**	**Prewet Methanol**	
Dispense	**0 seconds**	**Dispense**	**4 seconds**
Saturate	**0 seconds**	**Saturate**	**1 second**
Soak	**0 seconds**	**Soak**	**20 seconds**
Drain	**0 seconds**	**Drain**	**1 minute**
	Air Dry	**0 seconds**	
Rinse	**Dispense**	**Soak**	**Elute**
1. Hexane	**4 seconds**	**45 seconds**	**0 seconds**
2. Hexane	**4 seconds**	**45 seconds**	**30 seconds**
3. Hexane	**4 seconds**	**45 seconds**	**1 minute**

12.3.5. Run Method 26.

12.3.6. When samples are completed the controller will read IDLE for each station.

12.3.7. If it is difficult to see the hexane/water layer add into the collection vessel, 1-2 drops of food coloring to distinguish between the hexane layer and any water that may have collected.

12.3.8. Rinse the disk holder and collection vessel holder and sample inlet valves with warm water in-between samples and repeat.

12.3.9. When all samples are completed follow the manufacturer instructions for shutting down the SPE.

12.3.10. Weigh an aluminum-weighing pan for each sample and record in log book under tare weight.

- Note: Weighing pans should be stored in a desiccator prior to obtaining tare weight to ensure accuracy.

12.3.11. Insert aluminum pans into a space on the Speed-Vap. Transfer the hexane layer of the sample extract (top layer) into the pan, using phase separator papers to remove any water as needed.

12.3.12. At a temperature set to 40°C (per Horizon), vacuum set to the on position and air pressure at ~25 psi. Adjust the air pressure to give a vigorous rotation without spilling the hexane. Evaporate the sample on the Speed-Vap. Remove dry pans as soon as possible to avoid losing HEM.

12.3.13. When solvent has dried, weigh the pan and record in the log book under dried weight.

12.3.14. Determine the weight of HEM by subtracting the tare weight from the dried weight.

12.4. SGT-HEM Determination

12.4.1. HEM should be determined initially to insure that the silica gel capacity is not exceeded. If it has been previously determined that the silica gel capacity will not be exceeded, proceed to 14.2.3. If the HEM is less than the SGT-HEM reporting limit, report the SGT-HEM as a less than, no further analysis is required.

12.4.2. Extractable materials in silica gel - Because the capacity of silica gel is not known for all substances, it is presumed that 3 g will adsorb 100 mg of all absorbable materials. The amount of silica gel that can be used for adsorption in the SGT-HEM procedure below has been limited to 30 g because of concerns about possible extractable impurities in the silica gel. Therefore, if the extract contains more than 1000 mg of HEM, split the extract per the following procedure

- Using 50 ml of hexane, rinse contents of pan into 100 ml fluted volumetric flask.
- Dilute to mark with hexane.
- Adsorption with silica gel
  - Add 3.0 ± 0.3 g of anhydrous silica gel to the boiling flask for every 100 mg of HEM, or fraction thereof, to a maximum of 30 g of silica gel. For example, if the weight of HEM is 735 mg, add 3 x 8 = 24 g of silica gel.
  - Add a PTFE coated stirring bar to the flask and stir the solution on a magnetic stirrer for a minimum of 5 minutes.
  - Filter the solution through n-hexane moistened filter paper into a pre-dried, weighed aluminum pan. Rinse the silica gel and filter paper with several small amounts of n-hexane to complete the transfer.
  - Place the pan in the Speed Vap. (Adjust as in section 14.2.12) to evaporate the solution. Determine the weight of SGT-HEM.

13. Quality Control

13.1. Demonstration of Capability (DOC); Initial (IDOC) and Continuing (CDOC): Each analyst must have documentation of successfully performing an IDOC before his or her results for any method and analyte can be reported. Thereafter each analyst must demonstrate continued proficiency annually or whenever there is a change in instrument type, personnel or test method. Procedures for performing IDOCs and CDOCs can be found in the current PASI- Long Island Quality Manual.

13.2. See Attachment I.

13.3. Internal Standards and Surrogates are not used for this method as per SM 22nd and NELAC general wet chemistry requirements.

14. Data Analysis and Calculations

14.1. Calculate the concentration of HEM (“oil and grease”) by the following equation:

$$HEM(mg/L) = \frac{W_{HEM}(mg)}{W_S\,(L)}$$

W_{HEM} = Weight of hexane extractable material
W_S = Sample Volume (L)

14.2. Calculate the concentration of SGT-HEM ("petroleum hydrocarbons") by the following equation:

$$SGT\text{-}HEM\ (mg/L) = \frac{W_{SGT}\,(mg)}{W_S\,(L)}$$

W_{SGT} = Weight of silica gel treated hexane extractable material
W_S = Sample volume (L)

14.3. Percent recoveries are calculated and reported in units appropriate to the sample matrix. The following equation is used:

$$R = \frac{SSC - SC}{SA} \times 100\%$$

Where: R = percent recovery
SSC = spiked sample concentration
SC = sample concentration
SA = spike added

14.4. Relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{S - D}{\frac{(S + D)}{2}} \times 100\%$$

Where: RPD = relative percent difference
S = sample concentration
D = duplicate sample concentration

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. The analyst is responsible for generating the data and also is the initial individual to review the data. The review must include at least the following procedures:

15.1.1. Inspection of records in run log for completeness;

- Standard and reagent lot numbers, support equipment, spike amounts, calculations, dilution factors, container/bottle used for analysis, reporting limits.

15.1.2. Determination of whether the results meet the laboratory-specific quality control criteria;

15.1.3. Checks to determine consistency with client/project-specific measurement quality objectives (MQOs) if such exists;

15.1.4. Checks to ensure that the appropriate sample preparatory and analytical SOPs and methods were followed, and that chain-of-custody and holding time requirements were met;

15.1.5. Checks to ensure that quality control requirements were met;

15.1.6. Checks for complete and accurate explanations of anomalous results, corrective action, and the use of data qualifiers in the case narrative or LIMS QC notes.

15.1.7. Record of any non-standard condition of the test, test environment, sample or any deviation from standard operating procedure.

15.2. If analysis is deemed acceptable, data will be imported into the LIMS.

15.2.1. Another review is performed for correctness of results, including prep factors, dilution factors, spike amounts and recoveries, sample and QC references and appropriate qualifiers.

15.2.2. If additional information is to be communicated to the data user about a particular sample, a “QC Note” is entered by the analyst.

15.2.3. Once data has been reviewed in the LIMS, the analyst or supervisor will “QA” the sequence which indicates the data has been reviewed and is ready for reporting.

16. Corrective Actions for Out-of-Control Data

16.1. Corrective action begins with the analysts who are responsible for knowing when the analytical process is out of control.

16.2. Corrective actions should be initiated for out of control events such as when QC checks exceed acceptance limits or exhibit trending, holding time failures, loss of sample, equipment malfunctions or evidence of sample contamination.

16.3. If the blank fails, the source of the failure should be determined and rectified.

16.4. If the LCS fails, the standard should be verified and re-prepared if necessary.

16.5. Check data for calculation or transcription error.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Samples that have been analyzed during a non-compliant run will be rejected. The samples will be reanalyzed after the problem is resolved.

17.1.1. If reanalysis is not an option due to limited sample volume, a re-sample may be requested or data will be qualified.

17.1.2. If a re-sample is not possible, data will be qualified.

17.2. If the sample matrix is such that interferences prevent the system from meeting quality control requirements, the client will be notified with a course of action determined.

17.3. All problems associated with the analysis of a sample group will be outlined in the case narrative of the data package or with the use of QC Notes in the LIMS, if required.

18. Method Performance

18.1. Internal method performance is established and monitored with use of the following:

18.1.1. Demonstration of Capability

18.1.2. Positive and negative controls

18.1.3. Precision and accuracy

18.1.4. Control Charts

18.1.5. Quality Control Samples (PTs) performed semi-annually.

19. Instrument/Equipment Maintenance/Troubleshooting

19.1. For information and guidance related to instrument/equipment maintenance, troubleshooting and computer hardware and software refer to the following documents:

Document # (latest revision)	Document Title
QAM	*Quality Assurance Quality Control Manual*
ADMIN002	*Computers and Programs*
WC035B	*SPE -DEX 3000 XL SERIES Users GUIDE*
WC034	*Operating Instruction Mettler Toledo AX And MX/UMX Balances*
WC007	*Speed -Vap Ii 9000 Solvent Evaporation System User Guide*

19.2. Troubleshooting procedures also come from experience and training. Problems that the analyst encounters may be elevated to the department supervisor. Other means of problem resolution may come from review of prior related corrective action reports, instrument maintenance records, internal and external audits and lab notes.

20. Safety

20.1. Safety glasses, a lab coat, and disposable gloves must be worn when handling chemicals and samples.

20.2. The toxicity or carcinogenicity of each reagent used in this SOW has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposed to these chemicals must be reduced to the lowest possible level by personal protection and engineering measures. MSDS's are available for all chemicals used in the lab and are available for review.

21. Waste Management

21.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste.

21.2. Refer to the lab's *Sample and Waste Management* SOP.

22. Pollution Prevention

22.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Use pollution prevention techniques in laboratory operation whenever feasible.

22.2. Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

22.3. Control releases from hood and bench operations and comply with discharge permits and regulations, hazardous waste identification rules, and land disposal restrictions.

22.4. The generated waste has to be disposed in a manner not to cause pollution.

22.5. All wastes deemed toxic are collected in the laboratory and are disposed by professional companies licensed in waste disposal.

23. References

23.1. EPA method 1664A and EPA SW 846 9070.

23.2. National Environmental Laboratory Accreditation Conference (NELAC) Constitution, Bylaws and Standard, approved June 5, 2003, (EPA/600/R-04/003)

23.3. 2009 TNI Laboratory Accreditation Standards, EL-V1-2009.

23.4. 40 CFR Parts 136 Vol. 77, No. 97 / Friday, May 18, 2012 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; Final Rule

24. Tables and Figures

24.1. ATTACHMENT I: QUALITY CONTROL ACCEPTANCE CRITERIA AND CORRECTIVE ACTION PLAN

25. Revisions

Document Number	Reason for Change	Date
S-LI-I-016-rev.00	Transition to PACE format.	5-14-2015

S-LI-I-016-rev.01	Section 7.2 added or HCL for preservative. Section 12.1.8 - specify use of adapter to close sample bottle.	5/21/15
S-LI-I-016-rev.02	Added Section 12.1 – Preparation of sample batch, revised Section 12.1.4-pH verification, added 12.12.2.9 document disk size and QC, 12.2.10 – store weighing pans in desiccators, , 12.2.13 document rotation of QC through extraction stations.	7/22/15

ATTACHMENT I: QUALITY CONTROL ACCEPTANCE CRITERIA AND CORRECTIVE ACTION PLAN

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Demonstration of Capability (IDC)	• Per analyst; Before any samples are	• O&G 83-101% recovery	• If requirements are not met evaluate

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
	analyzed Analyze four replicates of a LFB	• TPH 83-116% recovery	standard preparation. • Reanalyze and recalibrate if necessary.
Method Blank (MB)	• One per batch, • Or 1 per 20 samples, whichever is more frequent. • Processed with and under the same conditions as samples and goes through all the steps of the analytical procedure.	• Should not contain target analytes or interferences at concentrations that impact the analytical results for sample analyses. • Target analytes should not be greater than PQL.	• If any analyte concentration in the blank is above the PQL, the lowest concentration of that analyte in the associated samples must be 10 times the blank concentration. Otherwise, all samples associated with the blank, with concentrations less than ten times the blank concentration and above the PQL, must be reanalyzed. • If concentration of the blank is below the negative PQL, then all samples reported below ten times PQL associated with the blank must be reanalyzed.
LCS/LFB **If not enough sample is provided by a client a LFB and LFBD can be run to check matrix accuracy*	• One per batch, • Or 1 per 20 samples, whichever is more frequent.	• O&G 78-114% recovery • TPH 64-132% recovery	• Evaluate the standards and equipment and reanalyze the initial calibration.
Matrix Spike Sample (MS)	• One per 20 samples or daily, if fewer than 20 samples.	• O&G 78-114% recovery • TPH	• If the recovery is outside the limits, repeat the sample, duplicate and matrix spike analysis once.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
		64-132% recovery	If it passes, then report samples. • Check for errors in calculation and spike preparation. • If the matrix spike still exceeds the limits, but the LFB has acceptable recovery, then the method is in control and sample matrix effects are likely the cause. The data should be qualified in the case narrative or using QC notes in the LIMS for non-package work.
MS Duplicate ***OR*** ***(alternative)*** Sample Duplicate	• One per 20 samples or daily, if fewer than 20 samples.	• O&G RPD ±18% • TPH RPD±34%	• Check sample label, calculation, dilution factors. • If results are grossly different (i.e., very high result and non-detect) re-analyze to confirm.

Appendix B

Facility O&M Plan, Table 5-1,
Structures and Equipment
Decontamination Procedures

Table 5-1 Structures and Equipment Decontamination Procedures

1	2	3	4	5	6
Material	**Typical Equipment and Structures**	**Decontamination Method**	**Sampling***	**PCB Decontamination Levels**	**Disposition Options**
Surfaces unexposed to PCB-containing sediments	Pre-Cast Box Culverts, rails, pre-cast ties, structural exteriors (roofs, exterior walls), Seal Water System, Rail Support Bldg (floor and steel)	N/A	None	N/A	Unrestricted use; distribution in commerce
Unpainted metal surfaces	Galvanized metal, corrugated steel pipe, steel members (filter cake staging enclosures, water treatment bldg, dewatering bldg)	Power Wash (or equivalent method of surface cleaning)	Yes (wipe)	< 10 µg/100 cm^2	Unrestricted use; distribution in commerce
Painted metal surfaces	Steel members - (rail support bldg, water treatment bldg, dewatering bldg).	Power Wash (or equivalent method of surface cleaning)	Yes (wipe and chip)	< 10 µg/100 cm^2 and < 1 ppm (in coating)	Unrestricted use; distribution in commerce
	Steel members - filter cake staging enclosures, container handling mechanisms.			< 100 µg/100 cm2 and < 25 ppm (in coating)	Low-occupancy use
	Gravity Thickener, Filter Press System			< 50 ppm (in coating)	Disposal in scrap metal recovery oven or; Recycle at smelter operating in accordance with 40 C.F.R. § 761.72
Other porous materials and surfaces	Pre-cast materials, dewatering and water treatment building floors, Unloading Wharf	Any method of surface cleaning or scarification	Yes (wipe and chip)	< 10 µg/100 cm^2 and < 1 ppm	Unrestricted use
	Fine Staging Area floor slabs, loading platforms			< 100 µg/100 cm^2 and < 25 ppm	Low-occupancy use
			Yes (chip)	< 50 ppm	Disposal in non-TSCA landfill
Plastic	HDPE corrugated pipe, Process Equipment Components (*e.g.* , Clarifier System)	Power Wash (or equivalent method of surface cleaning)	Yes (wipe)	< 10 µg/100 cm^2	Unrestricted use; distribution in commerce
	Process Equipment Components		Yes (chip)	< 50 ppm	Disposal in non-TSCA landfill
Movable equipment (excluding vessels)	Front End Loaders, Skid Steers, Excavators, Container Handling Systems	Power Wash (or equivalent method of surface cleaning)	None	N/A	Unrestricted use; distribution in commerce
Vessels	Barges, Scows	Power Wash (or equivalent method of surface cleaning)	Yes (wipe)	generally < 100 µg/100 cm^2 and <10 µg/100 cm^2 at high contact areas (e.g., hand rails)	Restricted Use (e.g., no food use)
Vehicles - entering and leaving site (Exclusion Zone)	Front End Loaders, Tractor Trailers, Fuel Trucks	Power Wash (or equivalent method of surface cleaning)	None	N/A	Unrestricted use; distribution in commerce
Containers	Sediment slurry tank, Granular activated carbon vessels, above grade storage tanks	Rinse (including with cleaners that do not contain organic solvents)	None	N/A	Unrestricted use; distribution in commerce

Notes:

1. Sampling methods, including the number and location of sampling points, will depend on a number of factors, including the extent and nature of the subject material's contact with > 50 ppm PCBs. The sample point selection methods chosen in any particular situation may be different from and alternatives to those set forth in 40 CFR Part 761, Subparts N – R.

2. Rail cars before being released from the project must be inspected pursuant to the "Empty Rail Car Inspection and Release Procedure" (Transportation and Disposal Plan, Attachment C).

3. These decontamination procedures do not apply to structures, equipment and vessels being cleaned for continued service on the GE Hudson River Project for handling of non-TSCA sediments. These cleaning procedures are presented in the *Transportation and Disposal Plan.*