NAVAL SECURITY GROUP ACTIVITY FACILITY
Building #85 Complex and Wullenweber Circulatory Disposed Antenna Array
Route 195/Corea Road
Village of Corea in Gouldsboro, Maine 04624

SITE SPECIFIC QUALITY ASSURANCE PROJECT PLAN ADENDUM

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
BROWNFIELD ASSESSMENT PROGRAM

JULY 2015

Prepared For:
HANCOCK COUNTY PLANNING COMMISSION

Prepared by:
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CAMPBELL
ENVIRONMENTAL GROUP

July 21, 2015

Mr. Thomas Martin
Hancock County Planning Commission
395 State Street
Ellsworth, Maine 04605

Re: Phase II Environmental Site Assessment Work Plan and Cost Estimate
Building #85 Complex and Wullenweber Circulatory Disposed Antenna Array
Route 195/Corea Road, Village of Corea in Gouldsboro, Maine 04624

Dear Thomas:

Campbell Environmental Group, Inc. (CEG) is pleased to submit this work plan and cost estimate to the Hancock County Planning Commission, to conduct a Phase II Environmental Site Assessment (ESA) at the Building #85 Complex and Wullenweber Circulatory Disposed Antenna Array (Site), located off Route 195/Corea Road, in the Village of Corea in Gouldsboro, Maine **(Figure 1)**. This work plan was developed to further investigate specific areas of concern (AOCs) as outlined in CEGs Draft *Phase I Environmental Site Assessment, Building #85 Complex and Wullenweber Circulatory Disposed Antenna Array* located Route 195/Corea Road, Village of Corea in Gouldsboro, Maine 04624 report, dated June 23, 2015. Recognized environmental conditions (REC) at the Site include the following;

REC#1: Lead paint was verified on the exterior of Building #85 during a 1996 inspection and assumed to be in the interior of many of the buildings as well. The paint is significantly chipping with large sections completely peeled from the walls, doors, and other surfaces.

REC#2: There are large areas within the Wullenweber CDAA that contain no plant life. The cause of this could be related to geotextile located directly below the surface, excessive amounts of herbicides, or another unrelated reason.

REC#3: Potential fuel spills related to the abandoned #2 fuel oil underground storage tank (UST) southwest of Building #85 may have impacted soil and groundwater. In addition, CEG is not certain that the adjacent 8,000-gallon diesel fuel UST has been removed.

REC#4: The presence of broken fluorescent light bulbs, particularly in Building #85, may have resulted in the release of mercury to the floors and walls.

REC#5: The pesticide mixing area, west of Building #103, has not been tested for herbicides or pesticides.

173 Gray Road, Falmouth, Maine 04105. (207) 253-1990. FAX (207) 253-1988. EMAIL: ceg@cegenvironmental.com

REC#6: There is a floor drain located in Building #156 that has an unknown discharge point. A significant amount of solvents were used in this building and if releases occurred, it is likely that they were discharged to the floor drain and its discharge point.

REC#7: Asbestos was detected in Building #85 and should be reevaluated to determine if the condition of the floor has deteriorated enough to be considered friable.

REC#8: Polychlorinated biphenyls may be present in the building materials of Buildings #85 and #103.

If you have any questions or comments, please call our office at (207) 253-1990. CEG appreciates the opportunity to work with you on this project.

Sincerely,

Danica Wallace
Senior Geologist

Richard B. Campbell
Maine Certified Geologist
President

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1.0 INITIAL SITE CONCEPTUAL MODEL

This section presents the initial conceptual model for the Building #85 Complex and Wullenweber Circulatory Disposed Antenna Array (CDAA), Route 195/Corea Road, Village of Corea Site. The model is based on information presented in CEGs Draft Phase I ESA dated June 23, 2015. This conceptual model may be revised in the Phase II ESA report, based on the data gathered during the Phase II investigation.

1.1 Site Description and History

The Subject Property which has been called the Gouldsboro Corea Business Park consists of four lots designated by the Town of Gouldsboro tax assessor's office as Map 42, Lots 29, 30, 31, and 32. The Subject Property is a small subset of what was originally part of the Naval Security Group Activity, Winter Harbor (NSGAWH). This Naval base consisted of three distinct sites with the main base (100.1 acres) located in Acadia National Park on Big Moose Island, the operations site which comprises this site was 451.5 acres and was located in Corea, and the housing area (23 acres) was located in Winter Harbor. The Subject Property lots are also designated as Lots 1 through 3 with the center lot (which houses all of the buildings) having no defined lot name on the Boundary Survey for Division of Land of Acadia Capital Corporation, State Route 195, Gouldsboro, by Edward J. Wainwright (PLS #1080) and dated May 10, 2010. According to the Gouldsboro tax assessor, there are currently no designated street addresses for these parcels. The size of each lot is listed below:

Table 1
Subject Property Lot Designations and Acreage

Tax Map/Lot	2010 Survey Lot Designation	Acreage
42/29	Lot 1	12.61
42/30	Lot 2	3.98
42/31	Lot 3	4.10
42/32	Not Named	8.8

The Subject Property is accessed by an unnamed driveway off Route 195 (aka Corea Road) and based on its location, is within Shoreland zoning. The Subject Property contains a right-of-way to the Atlantic Ocean, located directly west of the Subject Property. The Subject Property contains four lots (1, 2, 3 and the unnamed center lot). These lots form a circle that is encompassed by the former Wullenweber CDAA which was a radio direction-finder. The main building, which served as the operations center for this portion of the facility, is Building #85. Building #85 is surrounded by additional smaller buildings labeled as a septic tank building (#229), maintenance building (#217), maintenance building (#156), generator building (#103), and an electronics storage building (#226). All buildings associated with NSGAWH, were sequentially labeled by number in order of construction date.

The Subject Property and general vicinity are relatively flat except for the area surrounding Building #85 that is raised. CEG suspects the raised area surrounding Building #85 is fill which was used during building construction. The vicinity that surrounds the Subject Property is composed of heath and forested land. The Atlantic Ocean is located directly west of the property line. The area that formerly comprised the antenna arrays is lightly vegetated and contains predominately fine sand to coarse gravel. Some wetlands are also found throughout the area. A drainage area surrounds the northern, eastern, and southern portions of the former antenna arrays.

In 1960, the NSGAWH received appropriations for construction of a new radio direction-finder facility at the 451.5 acre Corea operations center. According to the *Environmental Baseline Study for Transfer* (EBST) *Naval Security Group Activity Winter Harbor, Maine Corea Operations Site* report prepared by Malcolm Pirnie and dated November 2000, the Wullenweber CDAA was constructed between 1960 and 1962. Buildings erected near the center of the array were subsequently built with the purpose of the station's participation in the Classic Wizard Advancement Tactical Ocean Surveillance Systems and as the center for training personnel involved, worldwide, in the system's maintenance and operation. Several additions to Building #85 were constructed in 1971 and 1988. In 1963, all operations were transferred from the main base in Winter Harbor to the Corea Site. Other buildings supporting operations conducted in Building #85 were constructed throughout the next thirty plus years. Building 229 was installed to provide potable water treatment and storage capabilities to the Building #85 complex. Radome 0 also known as Building #215 was also constructed in 1988 and located in between the northerly Building #153 complex and the Building #85 complex.

According to the *Potable Water System Study*, prepared by Stearns & Wheeler, LLC, and dated March 1996, the NSGA Corea Site was not served by a water supply facility sanctioned by the drinking Water Program of the Maine Department of Human Services. The Building #85 complex was supplied with potable water hauled from the alternate NSGA water supply facility located in Winter Harbor. The water was transferred to Building 229 which contained two 8,000 gallon underground storage tanks to store, treat and circulate potable water. Building #85 also had a non-potable water supply from the Radome 0 well located approximately half way between the Building #153 and Building #85 Complexes and west of the access road. The Radome 0 well is approximately 440 feet deep. There are other non-potable water supply wells located outside of Building #85 near the southwest side of the building under a manhole cover and another that was buried under pavement. Contaminants of concern for on-site wells include turbidity, iron, manganese, gross alpha particles, hydrogen sulfide, and radon. According to the Potable Water System Study, the radionuclides detected in the water supply wells are considered to be naturally occurring.

There are two leachfields constructed southwest of Building #85. One of the leachfields was older and replaced with a second leachfield that was located further south and west of the original leachfield.

One incinerator was located in Building #85. Ash from the incinerator was reported to have been removed by a waste contractor for off-site disposal.

Stormwater is reported to discharge to the drainage ditch that surrounds the CDAA. It is assumed that the strormwater eventually discharges to the ocean west of the Subject Property.

1.2 Geology and Hydrogeology Summary

Table 3
Site Geology and Hydrogeology

Feature	Source	Description
Nearest Water Body	USGS Topographic Map	Un-named stream originating at the Subject Property flowing westerly and discharging to Prospect Harbor
Bedrock Geology	Maine Geologic Survey	Devonian aged granite, but gray highly fractured basalt was encountered near the Wullenweber Array
Surficial Geology	Maine Geologic Survey	The surficial soil near the coast consists of undifferentiated thin drift. The soil thickness ranges from 0 to 7.5 feet thick and is mostly sand and gravel with some areas also containing silt. Soils toward the interior consist of swamp and marsh. Groundwater was encountered west of Building #153 at a depth of 52 to 79 inches below grade and south of Building #85 at 0 to 16 inches below grade.
Wetlands	National Wetland Inventory (NWI), EDR Report	Wetlands are scattered along the perimeter of the Subject Property.
Flood Zone	(FEMA) 100-year flood plain panel for Hancock County Panel 230283 0020 B	Subject property is designated as coastal flooding with base elevation of 13 feet
Drinking Water Source	Maine Public Water Resource Information System, MEDEP EGAD	Nearest public water supply well approximately 1.8 miles northwest of Subject Property across Prospect Harbor.
Sand & Gravel (S&G) Aquifer	MGS Hartland Quadrangle 2001, open File No. 01-86	Nearest sand & gravel aquifer is approximately 5 miles northeast of Subject Property

1.3 Potential Contaminants of Concern

Based on historical information, the following contaminants of concern (COCs) are anticipated:

Table 1-3 Contaminants of Concern	
Recognized Environmental Condition (REC)	**Contaminants of Concern (COCs)**
REC #1-Lead paint was verified on the exterior of Building #85 during a 1996 inspection and assumed to be in the interior of many of the buildings as well. The paint is significantly chipping with large sections completely peeled from the walls, doors, and other surfaces.	• Lead from paint chips impacting soil near exterior walls
REC #2- There are large areas within the Wullenweber CDAA that contain no plant life. The cause of this could be related to geotextile located directly below the surface, excessive amounts of herbicides, or another unrelated reason.	• Historical use of herbicides/ pesticides could be the cause of no plant growth within the CDAA
REC #3- Potential fuel spills related to the abandoned #2 fuel oil UST southwest of Building #85. In addition CEG is not certain that the adjacent 8,000-gallon diesel fuel UST has been removed.	• #2 fuel oil constituents from the UST
REC #4- The presence of broken fluorescent light bulbs particularly in Building #85 may have resulted in the release of mercury to the floors and walls.	• Mercury vapor from broken fluorescent bulbs may have impacted air quality and condensate mercury may have impacted porous building material
REC #5- The pesticide mixing area west of Building #103 has not been tested for herbicides or pesticides.	• Pesticides and herbicides from the pesticide mixing area
REC #6- There is a floor drain located in Building #156 that has an unknown discharge point. A significant amount of solvents were used in this building and if releases occurred it is likely that they were discharged to the floor drain and its discharge point.	• VOCs and RCRA metals suspected from solvents discharged to floor drain
REC #7- Asbestos was detected in Building #85 and should be reevaluated to determine if the condition of the floor has deteriorated enough to be considered friable.	• Asbestos based on visual observation of friable material and date of buildings constructed
REC #8- Based on the construction date of Buildings #85 and #103, the presence of poly-chlorinated biphenyls (PCBs) containing caulk and paint should be investigated	• PCBs in air and building material. PCB containing caulk and paint consists primarily of aroclors 1254 and 1242

Contaminants of concern for on-site wells include turbidity, iron, manganese, gross alpha particles, hydrogen sulfide, and radon. According to the Potable Water System Study, the radionuclides detected in the water supply wells are considered to be naturally occurring.

1.4 Contaminant Migration

There are several mechanisms by which contaminants may migrate at the Site. These transport mechanisms include air, groundwater, and surface runoff. Contaminant migration into air can occur by volatilization of contaminants or dust generation. Mercury vapor released from a fluorescent bulb is readily dispersed in air and absorbed through the lungs. This exposure pathway is the most immediate health concern. But the mercury vapor condenses to a liquid and typically absorbed into porous material. The liquid mercury can remain in place and vaporize over time, contributing to ongoing indoor exposure. Another contaminant that may pose a health risk through inhalation is PCBs from building materials, specifically PCB containing caulk or paint, if present.

Migration into groundwater can occur by percolating surface water or groundwater through waste materials or contaminated soils, which enters the groundwater in a dissolved-phase. Contaminants may also enter the groundwater directly through the floor drain. If the contaminant has a specific gravity less than 1.0 (the specific gravity of water), associated liquid-phase product will float on the groundwater surface. Conversely, if the specific gravity of the contaminant is greater than 1.0 (such as with chlorinated solvents), associated liquid-phase product will sink to the bottom of the groundwater column. Liquid-phase product is a continued source of dissolved-phase contamination. Once dissolved-phase contamination enters the groundwater, it will move with the groundwater in the direction of flow. Groundwater flow is anticipated to be in a westerly direction based on topography.

VOCs are typically volatile and soluble. Once released to the environment these compounds are expected to volatilize into the atmosphere, infiltrate into the soil, and dissolve into surface and groundwater. Upon entering the soil, the VOCs are expected to adsorb to the soil in the unsaturated zone and volatilize into the soil pore space. VOCs may also dissolve in rainwater, percolate through the soil column, and enter the groundwater. Some VOCs, such as toluene and xylene, have a greater persistence than other VOCs, and are, therefore, expected to remain in the soil profile for a longer duration. Once in solution, dissolved-phase VOCs will become mobile and migrate with the groundwater. Processes that naturally reduce VOC concentrations include volatilization, biodegradation, and dilution. Collectively, these processes are known as natural attenuation and given favorable conditions, can reduce the concentrations of VOCs over time.

The fate and transport of metals, PCBs, and SVOCs in the environment are similar. In general, PCBs, SVOCs, and metals tend to have very low solubility and mobility and will, therefore, tend to stay adsorbed to the soil particles and not migrate substantially into the water column. Metal solubility is affected by environmental factors including soil pH and soil temperature. The solubility of SVOCs is affected by their molecular weights. As a result, it is likely that some metals and SVOCs will mobilize more readily. In the case of the broken fluorescent light bulbs, mercury may be released once the bulbs have been broken. Liquid mercury may be released to the concrete floor or wall materials. Mercury released as a vapor may condense onto the same surfaces. It is assumed that the mercury will be concentrated on the surfaces of these materials. Some rainwater was present in particular parts of the buildings, if rainwater pooled or flowed within the buildings, it could potentially act as a transport mechanism to spread the mercury. PCBs may be present in some of the building materials and it is anticipated they would not migrate beyond their immediate location. In some instances, however, PCB impacted caulk could also migrate into the concrete surfaces in which the caulk is placed.

Once a contaminant has entered the groundwater, the rate of contaminant movement is influenced by many factors. These factors include physical and chemical properties of the contaminant and the aquifer. In general, once a contaminant reaches the groundwater, the contaminant will move as the groundwater moves through the process of advection (travel in the direction of groundwater flow) and dispersion (spreading vertically and horizontally).

1.5 Potential Receptors

Potential sensitive receptors at or in the vicinity of the Subject Property include the adjacent heath and groundwater. Additionally, human receptors could be impacted through dermal contact, inhalation, or ingestion of Site soils, dust, and groundwater.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY FLOW CHART

This section summarizes the organizational structure for this project.

2.1 Project Organizational Chart

Table 2-1 consists of a Project Organization Chart depicting the agencies and companies involved with this project. **Table 2-2** describes each participant's responsibilities for this project.

In addition to the project responsibilities outlined in **Table 2-1** and **Table 2-2**, CEG anticipates hiring subcontractors including an excavator operator and analytical laboratory, as described in **Section 5.0** of this Work Plan and Cost Estimate.

Table 2-1 Project Organization Chart

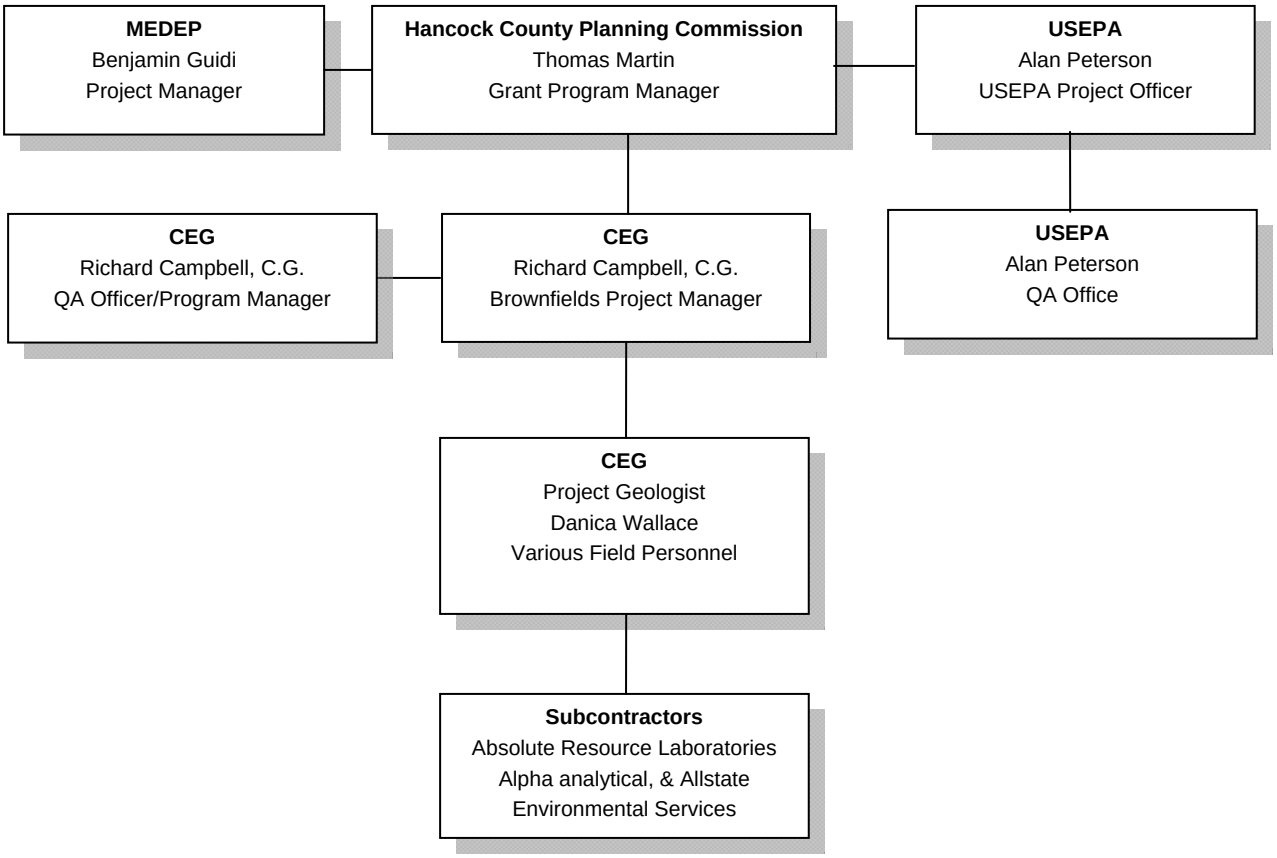


Table 2-2
Project Personnel Responsibilities

Name	Title	Organizational Affiliation	Responsibilities
Thomas Martin	Grant Program Manager	Hancock County Planning Commission	Administers Brownfields grant. Provides technical oversight.
Alan Peterson	EPA Project Officer	USEPA	Project oversight and approval.
Alan Peterson	EPA QA Officer	USEPA	Provides QA/QC project oversight.
Benjamin Guidi	MEDEP Project Manager	MEDEP	Provides technical oversight and reviews technical documents.
Richard Campbell	Brownfields Project Manager	CEG	Provides overall technical and project direction for the consultant.
Danica Wallace	Task Manager/ Field Leader	CEG	Day-to-day technical lead; oversees and coordinates data collection; participates in data interpretation and preparation of deliverables; communicates and coordinates with subcontractors.
Richard Campbell, C.G.	Quality Assurance Officer	CEG	Develops project QA/QC objectives and implements checks for QAPP adherence.
Field Staff	Scientists/ Engineers	CEG	Conduct field activities with oversight from Project Manager; oversee subcontractor field activities; communicates and coordinates with Project Manager.

3.0 SCOPE OF WORK SUMMARY

CEG proposes the following scope of work to investigate the identified AOCs:

- Prepare and submit this work scope and cost estimate for your approval;
- Prepare a Site Specific Health and Safety Plan (HASP);
- Mark the Site for Dig Safe;
- Collect ambient air samples in specific buildings for polychlorinated biphenyls (PCBs) and or mercury based on suspect building materials and areas of broken fluorescent bulbs;
- Investigate the floor drain piping to determine the discharge point;
- Sample inlet and outlet of drain for contaminants of concern;
- Conduct test pitting to explore subsurface conditions in the vicinity of the current/former fuel oil and diesel fuel USTs;
- Screen soils for VOCs using a photoionization detector (PID) and headspace technique;
- Screen soils for metals using an x-ray fluorescence (XRF) detector;
- Screen soils for petroleum hydrocarbons using oleophilic dye tests;

- Collect surficial and shallow soil samples based on soil screening and analyze for contaminants of concern as identified each REC;
- Collect building material samples for analysis of PCBs;
- Screen building surfaces and ambient air for mercury using a mercury vapor analyzer;
- Collect representative sample of broken fluorescent bulbs for characterization to determine if material is hazardous;
- Collect wipe samples of building material below broken fluorescent bulbs to determine impacts from condensed mercury to porous materials;
- Collect building material such as concrete flooring below broken fluorescent bulbs as confirmation samples to the wipe samples;
- Subcontract an asbestos investigation;
- Conduct a location survey of each analytical sample location using global positioning system (GPS); and
- Prepare a report summarizing the methods and results of the investigation.

4.0 SITE SPECIFIC QAPP ADDENDUM

This section represents the Building #85 Complex and Wullenweber CDAA Phase II ESA Site Specific QAPP Addendum (SSQAPPA). Information presented in the CEG Generic QAPP includes the quality assurance and quality control requirements for the HET Phase II ESA. Table 4-1 presents CEGs Standard Operating Procedures (SOPs) for this project that are located with the Generic QAPP and Table 4-2 presents SOPs that are not included in our Generic QAPP, but are attached to this work plan.

Table 4-1
CEG Standard Operating Procedures

SOP Reference Number	Title, Revision Date and/or Number	Originating Organization
SOP #002	Soil Sampling, Rev #0, August 2008	CEG
SOP #004	Monitoring Well Groundwater Sampling	MEDEP (DR#002)
SOP #007	Chain of Custody and Sample Handling, Rev #0, August 2008	CEG
SOP #008	Field Monitoring Equipment Calibration, Rev #0, August 2008	CEG
SOP #009	Micro-well Installation	MEDEP (DR#009)
SOP #010	Standard Guide for Site Assessments: Phase II ESA Process; ASTM Designation E1903-97, Rev #0, August 2008	ASTM
SOP #011	Field Documentation Protocol, Rev #1, May 2010	MEDEP (DR#013)
SOP #012	Preservation of Soil Samples for Volatile Organic Analysis, Rev #0, August 2008	CEG
SOP #014	MEDEP X-ray Fluorescence Field Screening, Rev #1, May 2010	MEDEP (DR#025)
SOP #019	Indoor Air Protocol	MEDEP
SOP #020	Compendium of Field Testing of Soil Samples for Gasoline and Fuel Oil	MEDEP (TS004)
SOP #022	Equipment Decontamination	MEDEP (DR#017)
SOP #023	GPS	CEG

Additional SOPs that are not currently in CEG's Generic Company QAPP

Table 4-2
Additional CEG Standard Operating Procedures

SOP Reference Number	Title, Revision Date and/or Number	Originating Organization
SOP #024	Collecting a Wipe Sample	CEG
SOP #025	Operating a Mercury Vapor Analyzer	CEG
SOP #026	For Sampling Porous Surfaces for Polychlorinated Biphenyls (PCBs)	EPA

5.0 PHASE II FIELD INVESTIGATION

The following section presents a detailed description of the proposed field investigation tasks used to investigate soil and building materials,. To complete the proposed investigation, CEG will subcontract with:

- Absolute Resource Associates, of Portsmouth, New Hampshire, for analytical services;
- Alpha Analytical, of Westborough, Massachusetts;
- Riverside Lane, of Ellsworth, for asbestos and lead paint sampling; and
- Allstate Environmental Services, of Gorham, Maine, for excavation of test pits and tank excavation if applicable.

Analytical samples collected will be labeled as presented in Section 10.2.1 of the QAPP as follows:

SS	= Surface Soil	TP	= Test Pit
BM	=Building Material	LS	= UST Excavation

5.1 Site Specific HASP

The purpose of the site-specific HASP is to provide guidance, standards, and critical information necessary to address health and safety issues at the Site. CEG will prepare a site-specific HASP to comply with Occupational Safety and Health Administration (OSHA) 29 CFR 1910.120 regulations. The HASP shall include, but not be limited to, personal protective equipment requirements, air monitoring guidelines and action levels, site hazards and controls, emergency telephone numbers, a contingency plan that conforms with 29 CFR 1910.120(1)(1) and (1)(2) for Site emergencies, and Material Safety Data Sheets (MSDS) for potential constituents of concern.

5.2 DigSafe Notification and Utility Clearance

Prior to initiating subsurface investigations, CEG will contact DigSafe to provide utility clearance. If possible, CEG will coordinate with local personnel to mark the Site for DigSafe.

5.3 Decontamination and Investigation Derived Wastes

Decontamination of non-dedicated equipment will be conducted using an alconox wash followed by a distilled water rinse in accordance with Section 5.1 of the QAPP. Investigation derived wastes (IDW), including decontamination fluids and excavated soil, will be discarded directly to the ground surface at the associated sample locations.

5.4 Analytical Program

Analytical samples collected at the site will be submitted to Absolute Resource Laboratories, LLC (ARA) for analysis. A description of analytical methods and CEGs quality assurance plan is included as an appendix to CEG's Generic QAPP. The analytical program consists of the analytical methods presented in **Table 5-1.**

Table 5-1
Absolute Resource Associates
Summary of SOPs, Methods, Glassware and Preservative per Laboratory Analysis

Test	Method	SOP No.	Matrix	Preferred Volume (mL or oz)	Container/ Preservative
Equipment Blank Water Matrix					
Volatile Organics	8260B, 624	QA-5120	Water	2x40mL	G-clear/4°C, HCl
Semi-volatiles	8270D, 625	QA-5515	Water	1000mL	G-Amber/4°C
Extractable Petroleum Hydrocarbons (EPH)	MADEP-2004-1.1	QA-5313	Water	1000 mL	G-Amber/4°C HCl
Individual Metals	6010C	QA-5603	Water	125mL	P/4°C, HNO_3
Herbicides	8151A	627.8151	Water	1 liter	G-Amber/4°C
Pesticides	8081	QA-5304	Water	1 liter	G-Amber/4°C
PCBs	USEPA Method SW8082	QA-5303	Water	2x1000 mL	GA/4°C
Soil and Building Materials Matrix					
Volatile Organics	5035/8260B	QA-5120	Soil	1-40mL Vial (10mL MeOH to 10g Soil)	G-Clear/4°C, MeOH
Semi-volatiles	8270D	QA-5304	Soil	4 oz	G-Amber/4°C
Extractable Petroleum Hydrocarbons	MADEP-2004-1.1	QA-5313	Soils	4 oz	G-Amber/4°C
Volatile Petroleum Hydrocarbons (VPH)	Method VPH 01-1.1,	QA-5130	Soils	1-40mL Vial (25mL MeOH to 25g Soil)	G-Clear/4°C, MeOH
Individual Metals	6010C / 7471B	QA-5603	Soil	4 oz	G-Clear/4°C
TCLP Metals	SW 846 Method 1311	QA-5604	Solid	4 oz	G/4°C
PCBs	USEPA Method SW8082	QA-5303	Building Material	4 oz	G-Amber/4°C
Herbicides	8151A	627.8151	Soil	4 oz	G-Clear/4°C
Pesticides	8081	QA-5304	Soil	4 oz	G-Clear/4°C

Table 5-1
Absolute Resource Associates
Summary of SOPs, Methods, Glassware and Preservative per Laboratory Analysis

Test	Method	SOP No.	Matrix	Preferred Volume (mL or oz)	Container/ Preservative
Air Sample					
Mercury	NIOSH Method 6009	6438	Air	6-liter	Summa canister
PCBs	Method TP-10A	2162	Air	6-liter	Summa canister
Wipe Sample					
Mercury	245.1, 7470A, and 7471B	QA-5600	Wipe	100 cm X 100 cm	G-clear

Quality control samples are presented in **Table 5-2** and will be collected as described in the QAPP.

Table 5-2
Field Quality Control Requirements

QC Sample	No. of Soil Samples	No. of Water Samples	Frequency	Acceptance Criteria	Corrective Action
Field Duplicate	1	1	5% per parameter per matrix	30% relative percent difference for duplicate aqueous samples. 50% relative percent difference for duplicate soil samples.	Compare to appropriate action level and re-sampling or reanalysis
Trip Blank	1	1	1 per cooler containing VOC water samples	No compounds detected	Qualify results or resample if required
Equipment Blank	1	1	One per non-dedicated piece of equipment that comes in contact with sample medium per event	No compounds detected	Qualify results or resample if cross contamination is suspected

Note: Samples will be collected with dedicated sampling equipment.

5.5 Data Quality Objectives

Data Quality Objectives (DQOs) are qualitative and quantitative statements that specify the quality and quantity of data needed to support decisions during site assessments. DQOs are developed by considering the purpose of collecting the data and the intended use of the data. For this project, the DQOs will establish the quality of data required to meet the goal of the site assessments and the intended end use of the data. The data collected will be compared to risk-based standards and screening criteria to evaluate potential risk to human health and the environment. A summary of data quality objectives developed to meet the site specific goals are provided in **Table 5-3**

TABLE 5-3
Summary of Data Quality Objectives

Matrix	Parameters	Methods	Analytical Level [1]	Data Evaluation Tier	Intended Data Use
Field Parameters					
Soil	VOCs	MEDEP SOP: TS004, Determining relative levels of solvents and gasoline in soil with a PID	Level I	NA	As appropriate to meet project goals
Soil	Metals	MEDEP SOP: #14	Level 1	NA	As appropriate to meet project goals
Soil	Fuel Oil	MEDEP SOP: TS004, Screening soils contaminated with kerosene and fuel oil using an oleophilic dye Test	Level 1	NA	As appropriate to meet project goals
Air	Mercury	Mercury Vapor Analyzer; SOP # 025	Level 1	NA	As appropriate to meet project goals

TABLE 5-3
Summary of Data Quality Objectives

Off-Site Laboratory Analysis

Matrix	Parameters	Methods	Analytical Level [1]	Data Evaluation Tier	Intended Data Use
Soil and water	VOCs	USEPA Method 8260B/5035	Level II	Modified Tier I	As appropriate to meet project goals
Soil and water	SVOCs	USEPA Method 8270	Level II	Modified Tier I	As appropriate to meet project goals
Soil and Water	Herbicides	USEPA 8151A	Level II	Modified tier I	As appropriate to meet project goals
Soil and water	Pesticides	USEPA Method 8081	Level II	Modified tier I	As appropriate to meet project goals
Soil, water, and Building Materials	Metals Mercury	USEPA Method SW846 – 6000/7000 Series	Level II	Modified Tier I	As appropriate to meet project goals
Building Materials	TCLP Mercury	SW 846 Method 1311	Level II	Modified Tier I	As Appropriate to meet project goals
Soil	Extractable Petroleum Hydrocarbons	MADEP-2004-1.1	Level II	Modified Tier I	As appropriate to meet project goals
Building Materials	PCBs	EPA Method 8082	Level II	Modified Tier I	As appropriate to meet project goals
Building Materials	Asbestos	EPA 600/R-93/116 PLM	Level II	Modified Tier I	As appropriate to meet project goals
Air	Mercury	NIOSH Method 6009	Level II	Modified Tier I	As appropriate to meet project goals
Air	PCBs	TO-10A	Level II	Modified Tier I	As appropriate to meet project goals
Wipe Sample	Mercury	EPA Method SW846 – 6000/7000 Series	Level II	Modified Tier I	As appropriate to meet project goals

NOTES:

1) Analytical levels (USEPA, October 1988):
Level I, on-site field screening and measurements, use one point calibration.
Level II analyses using standard laboratory QA/QC, including duplicate analyses, suitable calibration standards, sample preparation equipment, and operator training.

The media-specific criteria that may be used to evaluate the various types of data generated are presented in **Table 5-4**.

TABLE 5-4
State Criteria for Evaluating Data

Medium	Criteria for Evaluation
Surficial Soil (0-6" bgs)	Compare data with background concentrations and Appendix 2 Leaching to Groundwater RAGs (January 13, 2010)
Shallow Soil (6"-2' bgs)	Compare data with background concentrations and Appendix 2 Leaching to Groundwater RAGs (January 13, 2010)
Deep Soil Samples (>2')	Compare data with background concentrations and Appendix 2 Leaching to Groundwater RAGs (January 13, 2010)
Lead Paint	Maine Solid Waste Management Rules, Chapter 424
Broken Fluorescent Bulbs	US EPA hazardous material characterization by TCLP analysis for Future Disposal
ACM	USEPA - Asbestos Hazard Emergency Response Act (AHERA)–40 CFR 763. USEPA - National Emission Standard for Hazardous Air Pollutants (NESHAP)–40 CFR 61. OSHA - Asbestos Standard for General Industry–29 CFR 1910.1001. OSHA - Asbestos Standard for Construction Industry–29 CFR 1926.1101. MEDEP - Statutory Sections - Title 38, Chapter 12-A: Asbestos §1271 - §1284 MEDEP - Chapter 425 - Asbestos Management Regulations, Revised February 2011
Air (Mercury Vapor & PCBs)	RAG Table 2: Indoor Air Commercial
Mercury Surface Wipes	Compare with corresponding Building Material samples to establish a correlation for screening purposes
Mercury Building Material	RAG Table 1: Soil Commercial Worker

Notes: ft bgs = feet below ground surface, R AGs = Remedial Action Guidelines For Soil Contaminated With Hazardous Substances, ACM=asbestos containing material, OSHA=Occupational Safety and Health Administration

bgs = below ground surface
MEDEP = Maine Department of Environmental Protection
RAGs = Remedial Action Guidelines For Soil Contaminated With Hazardous Substances
MEGs = Maximum Exposure Guidelines for Drinking Water

CEG reviewed ARAs Method Reporting Limits for the compounds in each of the selected analytical methods to determine if the detection limits for each compound are sufficient to meet the regulatory criteria presented in **Table 5-4**. A list of compounds that do not meet the respective DQOs has been attached to this SSQAPPA in the appendices. In these cases, CEG will ask ARA to report analytical results to the respective laboratory minimum detection limit. If samples from other analyzed media indicate elevated concentrations of these compounds, additional samples may be required based on site specific conditions.

5.6 Air Sampling

For health and safety measures, CEG shall initially collect air samples within Building #85 for mercury vapor using national Institute for Occupational Safety and health (NIOSH) Method 6009. The result of the air sample will determine the personal protective equipment required to conduct the subsequent proposed work. Air sampling shall be conducted using a 24-hour pre-set flow regulator. Personnel conducting the testing shall minimize time within these buildings to reduce exposure. The testing should be conducted approximately 3 weeks prior to any additional work within the designated building.

The indoor air samples will be collected using a 6-liter Summa canister with regulators pre-set by the laboratory for a 24-hour sample collection time. The canisters shall be placed in an area representative of an area with the highest potential for contaminants.

CEG personnel shall document site and weather conditions during the air sampling using a modified format of the *MEDEP Bureau of Remediation Draft Indoor Air Sample Protocol With Indoor Air Sample Information Collection Form*, August, 2, 2009. Results shall be compared with the current MEDEP guidelines per the *Maine Remedial Action Guidelines (RAGs) for Sites Contaminated with Hazardous Substances*, dated May 8, 2013.

Based on the results of the PCB building material bulk samples analyzed for PCBs, CEG shall determine if air sampling in Building #85 and Building #103 are warranted. If warranted, air samples shall be analyzed for PCBs using EPA Method TO-10A. This air sample shall require an 8 hour flow regulator to meet the necessary detection limits.

5.7 Surficial and Shallow Soil Screening and Analytical Sampling

Proposed surficial and shallow soil samples shall be collected at various locations where suspected contaminants of concern may exist. Soil samples will be collected and documented in accordance with the QAPP and SOP #002. The soil samples shall be described by the on-site geologist and include soil color, texture, moisture content, and any other defining characteristics. On-sight soil screening will consist of visual observations of impacted soil; VOCs using a photoionization detector (PID) with a 10.6 eV-lamp; oleophilic dye test in accordance with MEDEP's SOP# TSOO4; and metals using a portable X-Ray fluorescence analyzer (XRF) following MEDEP's SOP# 14.

CEG anticipates up to six surficial or shallow soil samples will be collected at the Site with proposed sampling locations presented in **Figure 2**. Analytical sampling will be conducted based on suspected contaminants of concern and screening results. CEG has proposed the following analysis as outlined in **Table 5-6**.

5.8 Building Material Sampling

Following the results of the air sampling and making any adjustments to the HASP, if necessary, CEG personnel shall collect building material samples for the analysis of lead paint, mercury and PCBs.

CEG is proposing three samples be analyzed for lead paint (BM-1 through BM-3). One sample shall be collected from any residue on the ground surface and/or from the hand railing on the southwest side of Building #85. Two additional samples are proposed to be collected from the interior walls of Building #85. A single representative sample of the broken fluorescent bulbs shall be collected and analyzed for TCLP mercury (BM-4) to determine if the bulk of material exceeds hazardous waste criteria of 0.2 ug/L for future disposal purposes.

CEG shall collect three wipe samples (BM-5A through BM-7A) and three corresponding floor samples from beneath the broken fluorescent bulbs (BM-5B through BM-7B) for the analysis of mercury. CEG personnel shall attempt to push aside the broken bulbs and as much residue as possible using disposable wooden tongs within an approximate 1 foot by 1 foot area per sample location. Approximately one third of the cleared area (10 cm X 10 cm) shall be used for the wipe sample and the remaining area for the bulk floor sample. The bulk floor sample shall be collected using either a chisel or generator powered hammer drill depending on the flooring material. The purpose of collecting the two samples side by side is for determining correlations, if any, between the two samples. If a correlation can be determined, wipe samples may be used either as a screening method or confirmation sample method during future clean-up, if necessary.

CEG shall collect Building Material samples from Buildings #85 and #103 for the analysis of PCBs. The building material shall be representative of caulk material used as a sealant around windows, doors, and/or foundations. One representative sample from each source media per building shall be collected and analyzed for PCBs using EPA Method 8082.

All bulk samples for mercury and PCB analysis shall be collected in accordance with SOP #026. Wipe samples for the analysis of mercury shall follow the intent of SOP # 024. These SOPs are not in CEG's generic Company QAPP and therefore attached.

CEG shall subcontract Riverside Lane to conduct asbestos and lead paint sampling. CEG anticipates three samples from the flooring tile and three samples from the flooring mastic in Building # 85 will be collected and analyzed.

Table 5-6
Surficial Soil Sample Collection and Laboratory Analysis

Samples	Location	Pest	Herb	RCRA Metals	VOC	Rationale
SS-1 thru SS-2	Non-vegetated gravel along perimeter of the CDAA	✓	✓			Determine if application of pesticides and herbicides have impacted soil
SS-3 and SS-4	Floor drain in Bldg 156 and potential discharge location			✓	✓	Determine if any solvents have entered the floor drain
SS-5 and SS-6	Pesticide mixing area near Bldg 103	✓	✓			Determine if spillage or overfill in the area of the mixing of pesticides and herbicides have impacted soils

.

Table 5-7
Building Material Sample Collection and Laboratory Analysis

Samples	Location	Lead	TCLP MERCURY	Mercury	Mercury Wipe	PCBs	ACM	Rationale
BM-1 thru BM-3	Northwest corner of Building #85	✓						Determine if lead paint chips are impacting soils and interior space of bldg 85
BM-4	Broken fluorescent bulbs in Building #85		✓					Determine if broken fluorescent bulbs are considered hazardous for disposal purposes
BM-5A Thru BM-7A	Flooring below broken fluorescent bulbs				✓			Determine if building materials are impacted with mercury
BM-5B thru BM7B	Flooring co-located with wipe samples			✓				Determine if building materials are impacted and if wipe samples can be used as a screening method for determining approximate concentrations of mercury
BM-8 thru BM-13	Caulk around windows, doors, foundation					✓		Determine if caulk contains levels of PCB that may cause a health risk to occupants of building.
BM-14 thru BM-19	Specific rooms within Building #85						✓	Determine if suspect friable asbestos should be addressed

5.7 Test Pit Sampling

CEG will supervise the excavation of approximately 4 test pits to investigate the presence and any impacts from the #2 fuel oil UST on the west side of Building #85. Excavations are estimated to extend to approximately 12 feet below grade. A CEG geologist will collect, log, and screen grab soil samples from the excavated test pits. Soil samples will be screened on-site for metals using an XRF and VOCs using a 10.6 eV-lamp PID. CEG shall collect at least one (1) and up to four (4) soil samples from test pits exhibiting contamination based on visual observations and field screening results. Test pit soil samples may also be selected for laboratory analysis based on their proximity to soil staining (visual observation), olfactory evidence of potential contamination, and or proximity to the suspected sources of constituents of concern. Proposed test pit locations are shown on the attached **Figure 2**.

Table 5-8
Test Pit Soil Sample Collection and Laboratory Analysis

Number of Samples	Location	VOC	SVOC	EPH	VPH	RCRA Metals	Rationale
Up to 4 TP-1 thru TP-4	North, south, east, and west of the current/former fuel oil and diesel fuel USTs			✓			Determine if soil in vicinity of UST exhibit evidence of a release and meet the appropriate RAGs

5.8 Surveying

CEG will conduct a horizontal survey of all sampling points using a Trimble GPS and any prior existing wells with access. The survey data and approximate sampling locations will be documented on the site map.

5.9 Sample Collection Summary

Table 5-9
Sample Collection Summary by Media

	VOCs	Pesticides	RCRA Metals	Herbicides	EPH	Lead	Mercury	TCLP Mercury	Mercury wipe	PCBs
Project Soil Samples	2	4	2	4	4	0	0	0	0	0
QC Soil Duplicates	1	1	1	1	1	0	0	0	0	0
Equipment Blank	1	1	1	1	1	0	0	0	0	0
Trip Blank	1	0	0	0	0	0	0	0	0	0
Total Solid samples	**5**	**6**	**4**	**6**	**6**	**0**	**0**	**0**	**0**	**0**
Project Building Material (BM) Samples	0	0	0	0	0	3	3	1	3	6
QC BM Duplicates	0	0	0	0	0	1	1	1	1	1
Equipment Blank	0	0	0	0	0	1	1	1	0	1
Trip Blank	0	0	0	0	0	0	0	0	0	0
Total BM samples	**0**	**0**	**0**	**0**	**0**	**5**	**5**	**3**	**4**	**8**
Project Air Samples	0	0	0	0	0	0	2*	0	0	1**
QC Duplicates	0	0	0	0	0	0	1*	0	0	1**
Total Air Samples	**0**	**0**	**0**	**0**	**0**	**0**	**3**	**0**	**0**	**2**
*-NIOSH Method 6009, **-EPA Method TO-10A										

6.0 PROJECT REPORTING

The Site investigation data will be compiled into a letter report documenting the methods and results of the investigation tasks. The report will include test pit and boring logs, a site map depicting test pit, soil boring, and monitoring well locations, groundwater contour maps, soil and groundwater quality maps, a revised conceptual model, and a data table comparing the data to applicable guidelines or standards. The report will describe adjustments to the work plan, and will also include recommendations, if any, based on the results of the investigation.

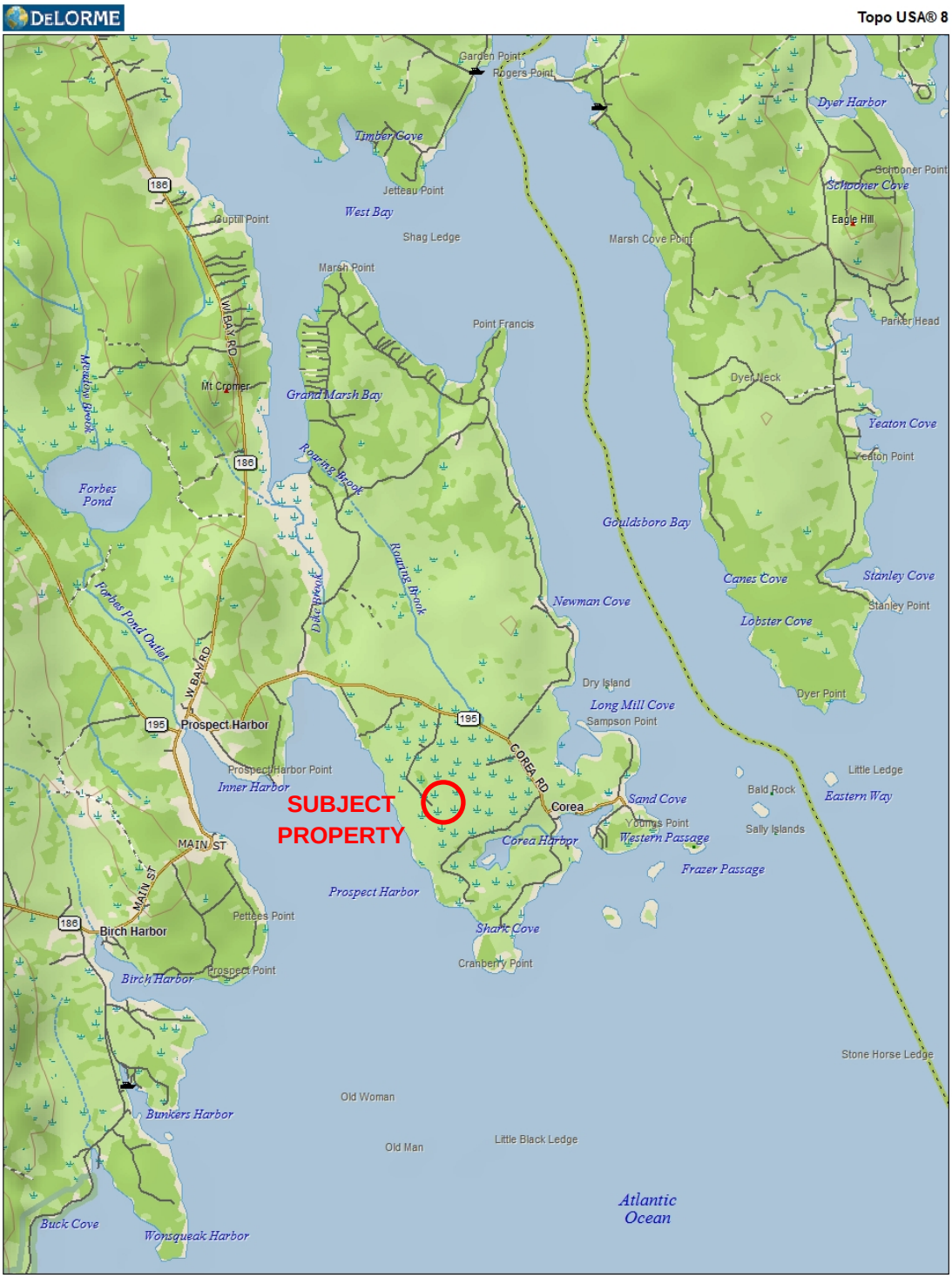
7.0 TENTATIVE SCHEDULE

CEG is prepared to initiate work upon approval of this work plan. The field investigation is scheduled to be completed within one day of mobilization to the Site. Analytical data is anticipated from ARA within three weeks of sample submittal and a Draft Phase II Report is anticipated to be delivered to the HCPC within four weeks of CEG's receipt of the analytical data.

8.0 COSTING

The estimated cost to conduct this proposed scope of work is $28,506.96. A detailed cost estimate is attached.

Figure 1
Locus Map
Naval Security Group Activity Facility
Corea Village, Gouldsboro, Maine



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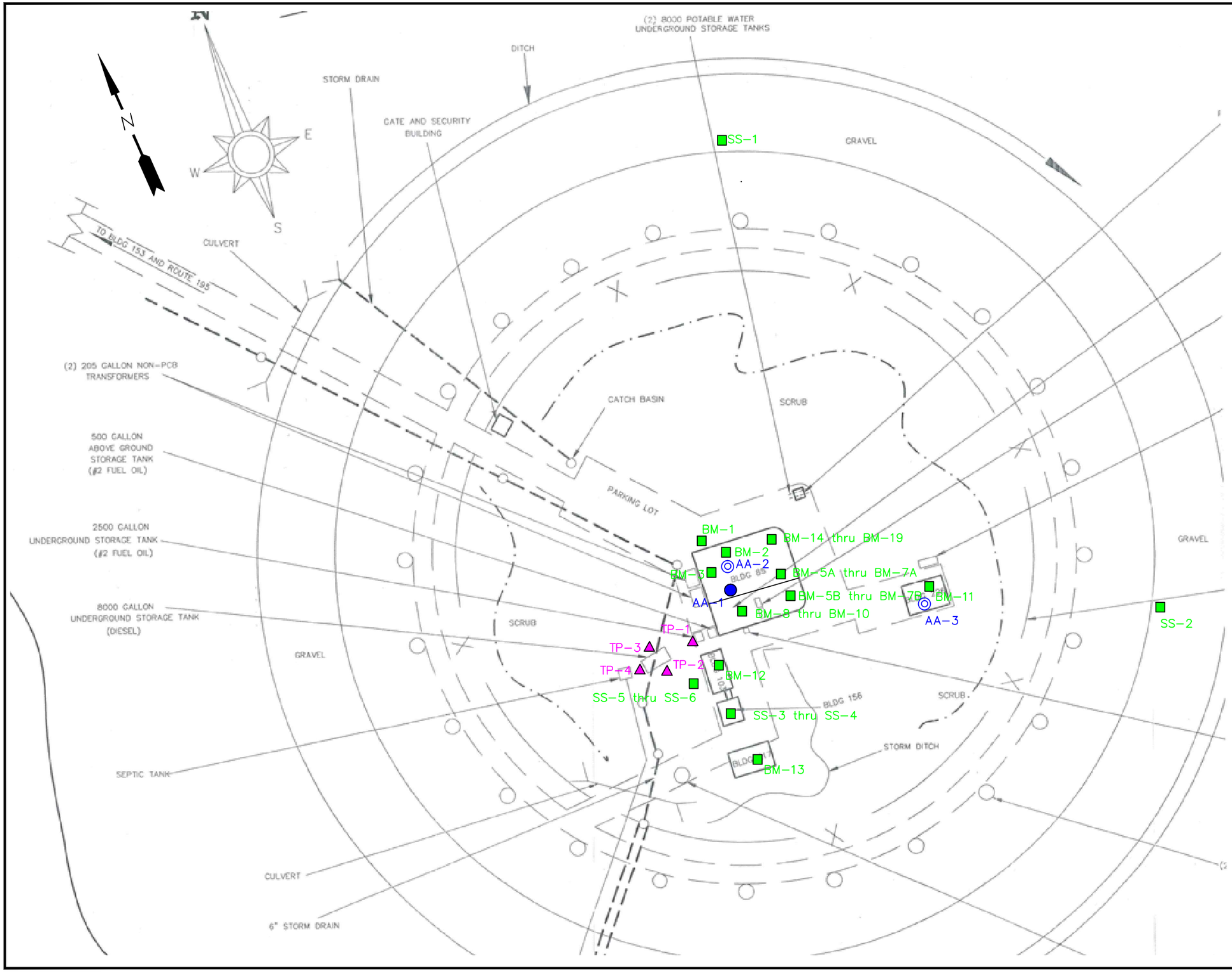

LEGEND

- TEST PIT
- AMBIENT AIR SAMPLE FOR MERCURY
- OPTIONAL AIR SAMPLE FOR POLYCHLORINATED BIPHENYLS (PCBs)
- SHALLOW SOIL SAMPLE OR BUILDING MATERIAL SAMPLE

APPROXIMATE SCALE IN FEET

0 150 300

CLIENT: HANCOCK COUNTY PLANNING COMMISSION

LOCATION: NAVAL SECURITY GROUP ACTIVITY GOULDSBORO, MAINE

PM:	DETAILED:	PROJECT NO.:
DW	DW	1014-257-00
REV. NO.:	DRAWING DATE:	ACAD FILE:
	06/30/15	SITESKETCH

Figure 2

SITE SKETCH BLDG #85

CAMPBELL ENVIRONMENTAL GROUP
173 GRAY ROAD
FALMOUTH, MAINE 04105

As of the date of this site-specific QAPP addendum, the current state and/or federal standards have been incorporated into this table and the reporting limits and standards have been reviewed for accuracy.

TABLE 1-1
Absolute Resource Associates, LLC
2014 Analytical Method Sensitivity and Project Criteria

Analyte	CAS No.	SOIL (mg/kg) 2013 MEDEP REMEDIAL ACTION GUIDELINES (RAGs)		GROUNDWATER (ug/l) 2013 RAGs or 2012 MEGs*	SOIL (mg/kg)		GROUNDWATER (ug/L)	
		RAG	Exposure Scenario*		REPORTING LIMITS (mg/Kg)	MINIMUM DETECTION LIMITS (mg/Kg)	REPORTING LIMITS (ug/L)	MINIMUM DETECTION LIMITS (ug/L)
Metals (Analytical Methods 6010/7000)								
ARSENIC	7440-38-2	1.4	Residential	10	0.5	0.1	8	0.9
BARIUM	7440-39-3	10,000	Residential	1,000	2	0.1	50	0.2
CADMIUM	7440-43-9	11	Residential	1	0.2	0.09	4	0.2
CHROMIUM (+6)	18540-29-9	510	Residential	20	0.4	0.2	10	6
CHROMIUM (+3) (By Calculation)	16065-83-1	10,000	Residential	10,000	2	0.5	50	0.4
LEAD	7439-92-1	340	Residential	10	0.5	0.1	8	0.7
MERCURY	7439-97-6	51	Residential	2	0.02	0.002	0.2	0.03
SELENIUM	7782-49-2	nr	nr	40	2	0.5	50	2
SILVER	7440-22-4	850	Residential	40	0.4	0.04	7	0.4
Volatile Organic Compounds - VOCs (Analytical Method 8260)								
1,1,1,2-TETRACHLOROETHANE	630-20-6	0.20	Leaching to Groundwater	10	0.1	0.02	2	1
1,1,1-TRICHLOROETHANE	71-55-6	520	Leaching to Groundwater	10,000	0.1	0.02	2	1
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.026	Leaching to Groundwater	2	0.1	0.02	2	1
1,1,2-TRICHLOROETHANE	79-00-5	250	Residential	6	0.1	0.02	2	0.9
1,1-DICHLOROETHANE	75-34-3	1.0	Leaching to Groundwater	60	0.1	0.02	2	0.9
CIS-1,2-DICHLOROETHENE	156-59-2	0.14	Leaching to Groundwater	10	0.1	0.02	2	0.9
TRANS-1,2-DICHLOROETHENE	156-60-5	2.4	Leaching to Groundwater	100	0.1	0.03	2	1
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	3.2	Residential	0.4	0.1	0.04	2	0.8
1,2-DIBROMO-3-CHLOROPROPANE (Low Level SIM analysis)	96-12-8	3.2	Residential	0.4	NA	NA	0.2	0.02
1,2-DIBROMOETHANE/ETHYLENE DIBROMIDE	106-93-4	7.1	Residential	0.2	0.1	0.009	2	0.8
1,2-DIBROMOETHANE (Low Level SIM analysis)	106-93-4	7.1	Residential	0.2	NA	NA	0.05	0.05
1,2-DICHLOROBENZENE	95-50-1	11	Leaching to Groundwater	200	0.1	0.02	2	0.9
1,2-DICHLOROETHANE	107-06-02	0.036	Leaching to Groundwater	4	0.1	0.02	2	0.9
1,2-DICHLOROPROPANE	78-87-5	390	Residential	10	0.1	0.03	2	0.9
1,2,3-TRICHLOROBENZENE	87-61-6	420	Excavation or Construction Worker	nr	0.1	0.02	2	0.9
1,2,3-TRICHLOROPROPANE	96-18-4	nr	nr	0.01	0.1	0.02	2	1
1,2,4-TRICHLOROBENZENE	120-82-1	8.6	Leaching to Groundwater	70	0.1	0.009	2	0.9
1,3-DICHLOROPROPANE	142-28-9	3,400	Residential	100	0.1	0.02	2	0.9
1,3-DICHLOROBENZENE	541-73-1	0.075	Leaching to Groundwater	1	0.1	0.01	2	0.9
CIS 1,3-DICHLOROPROPENE	1006-01-5	140	Residential	4	0.1	0.03	2	1
TRANS 1,3-DICHLOROPROPENE	1006-02-6	140	Residential	4	0.1	0.03	2	0.8
1,4-DICHLOROBENZENE	106-46-7	4.3	Leaching to Groundwater	70	0.1	0.01	2	0.9

As of the date of this site-specific QAPP addendum, the current state and/or federal standards have been incorporated into this table and the reporting limits and standards have been reviewed for accuracy.

TABLE 1-1
Absolute Resource Associates, LLC
2014 Analytical Method Sensitivity and Project Criteria

Analyte	CAS No.	SOIL (mg/kg) 2013 MEDEP REMEDIAL ACTION GUIDELINES (RAGs)		GROUNDWATER (ug/l) 2013 RAGs or 2012 MEGs*	SOIL (mg/kg)		GROUNDWATER (ug/L)	
		RAG	Exposure Scenario*		REPORTING LIMITS (mg/Kg)	MINIMUM DETECTION LIMITS (mg/Kg)	REPORTING LIMITS (ug/L)	MINIMUM DETECTION LIMITS (ug/L)
2-BUTANONE/METHYL ETHYL KETONE	78-93-3	10,000	Residential	4,000	0.3	0.04	10	0.6
2-CHLOROTOLUENE	95-49-8	nr	nr	100	0.1	0.03	2	0.7
4-CHLOROTOLUENE	106-43-4	0.69	Residential	500	0.1	0.02	2	1
4-ISOPROPYLTOLUENE	99-87-6	nr	nr	70	0.1	0.01	2	1
ACETONE	67-64-1	10,000	Residential	6,000	2	0.05	50	2
BENZENE	71-43-2	0.51	Leaching to Groundwater	4	0.1	0.04	2	0.9
BROMOCHLOROMETHANE	74-97-5	nr	nr	100	0.1	0.03	2	0.8
BROMODICHLOROMETHANE	75-27-4	230	Residential	6	0.1	0.03	0.6	0.06
BROMOFORM	75-25-2	1,400	Residential	40	0.1	0.02	2	1
BROMOMETHANE	74-83-9	240	Residential	10	0.2	0.02	2	0.9
CARBON DISULFIDE	75-15-0	10,000	Residential	600	0.1	0.03	2	0.9
CARBON TETRACHLORIDE	56-23-5	0.55	Leaching to Groundwater	5	0.1	0.03	2	0.9
CHLOROBENZENE	108-90-7	1.1	Leaching to Groundwater	100	0.1	0.02	2	1
CHLOROFORM	67-66-3	460	Residential	70	0.1	0.02	2	0.9
CHLOROMETHANE	74-87-3	10,000	Residential	20	0.1	0.03	2	1
DIBROMOCHLOROMETHANE	124-48-1	170	Residential	4	0.1	0.02	2	0.9
DICHLORODIFLUOROMETHANE	75-71-8	10,000	Residential	1,000	0.1	0.03	2	1
ETHYLBENZENE	100-41-4	0.81	Leaching to Groundwater	30	0.1	0.02	2	1
CHLOROETHANE	75-00-3	1,700	Residential	7	0.1	0.02	2	1
HEXACHLOROBUTADIENE	87-68-3	130	Residential	4	0.1	0.04	0.5	0.5
4-METHYL-2-PENTANONE (MIBK)	108-10-1	10,000	Residential	500	0.4	0.03	10	0.9
METHYL TERT-BUTYL ETHER	1634-04-4	0.19	Leaching to Groundwater	35	0.1	0.02	2	0.8
METHYLENE CHLORIDE/DICHLOROMETHANE	75-09-2	1,000	Residential	40	0.1	0.02	5	0.9
NAPHTHALENE	91-20-3	1.7	Leaching to Groundwater	10	0.1	0.01	5	0.9
STYRENE	100-42-5	10,000	Residential	100	0.1	0.008	2	0.9
TETRACHLOROETHENE	127-18-4	2.7	Leaching to Groundwater	40	0.1	0.02	2	1
TETRAHYDROFURAN	109-99-9	nr	nr	600	0.5	0.03	10	1
TOLUENE	108-88-3	8.1	Leaching to Groundwater	600	0.1	0.09	2	0.9
TRICHLOROETHENE	79-01-6	0.23	Leaching to Groundwater	4	0.1	0.03	2	0.8
TRICHLOROFLUOROMETHANE	75-69-4	10,000	Residential	2,000	0.1	0.03	2	1
VINYL CHLORIDE	75-01-4	0.013	Leaching to Groundwater	0.2	0.1	0.02	2	1
XYLENES	1330-20-7	26	Leaching to Groundwater	1,000	0.1	0.07	2	2
Semi-volatile Organic Compounds - SVOCs (Analytical Method 8270)								

As of the date of this site-specific QAPP addendum, the current state and/or federal standards have been incorporated into this table and the reporting limits and standards have been reviewed for accuracy.

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Absolute Resource Associates, LLC
2014 Analytical Method Sensitivity and Project Criteria

Analyte	CAS No.	SOIL (mg/kg) 2013 MEDEP REMEDIAL ACTION GUIDELINES (RAGs)		GROUNDWATER (ug/l) 2013 RAGs or 2012 MEGs*	SOIL (mg/kg)		GROUNDWATER (ug/L)	
		RAG	Exposure Scenario*		REPORTING LIMITS (mg/Kg)	MINIMUM DETECTION LIMITS (mg/Kg)	REPORTING LIMITS (ug/L)	MINIMUM DETECTION LIMITS (ug/L)
1,2-DICHLOROBENZENE	95-50-1	11	Leaching to Groundwater	200	0.2	0.1	2	2
1,2,4-TRICHLOROBENZENE	120-82-1	8.6	Leaching to Groundwater	70	0.5	0.1	5	3
1,3-DICHLOROBENZENE	541-73-1	0.075	Leaching to Groundwater	1	0.2	0.1	2	2
1,4-DICHLOROBENZENE	106-46-7	4.3	Leaching to Groundwater	70	0.2	0.1	2	2
2-CHLOROPHENOL	95-57-8	850	Residential	40	0.5	0.1	5	4
2-Cresol	95-48-7	6,700	Residential	40	0.2	0.1	2	1
2-METHYLNAPHTHALENE	91-57-6	3.6	Leaching to Groundwater	30	0.05	0.01	0.5	0.3
2,4-DICHLOROPHENOL	120-83-2	400	Residential	20	0.5	0.1	5	3
2,4-DIMETHYLPHENOL	105-67-9	2,700	Residential	100	0.2	0.1	2	2
2,4-DINITROPHENOL	51-28-5	270	Residential	10	5	5	50	0.6
2,4-DINITROTOLUENE	121-14-2	35	Residential	1	0.2	0.1	2	2
2,4,5-TRICHLOROPHENOL	95-95-4	10,000	Residential	700	0.2	0.1	2	2
2,4,6-TRICHLOROPHENOL	88-06-2	130	Residential	7	0.2	0.1	2	2
2,6-DINITROTOLUENE	606-20-2	16	Residential	0.5	0.2	0.1	2	2
3,3-DICHLOROBENZIDINE	91-94-1	24	Residential	0.8	3	0.04	30	0.7
4-CHLOROANILINE	106-47-8	54	Residential	2	0.2	0.1	2	1
4-METHYLPHENOL	106-44-5	670	Residential	4	0.2	0.2	2	1
4-NITROPHENOL	100-02-7	nr	nr	60	2	2	10	1
ACENAPHTHENE	83-32-9	170	Leaching to Groundwater	400	0.05	0.05	0.5	0.4
ACENAPHTHYLENE	208-96-8	68	Leaching to Groundwater	nr	0.05	0.05	0.5	0.3
ANTHRACENE	120-12-7	2,400	Leaching to Groundwater	2,000	0.05	0.05	0.5	0.3
BENZO(a)ANTHRACENE	56-55-3	2.6	Residential	0.5	0.05	0.05	0.5	0.3
BENZO(A)PYRENE	50-32-8	0.26	Residential	0.05	0.05	0.05	0.2	0.2
BENZO(b)FLUORANTHENE	205-99-2	0.26	Residential	0.5	0.05	0.05	0.5	0.2
BENZO(g,h,i)PERYLENE	191-24-2	3,700	Residential	nr	0.05	0.05	0.5	0.2
BENZO(k)FLUORANTHENE	207-08-9	26	Residential	5	0.05	0.05	0.5	0.2
BENZOIC ACID	65-85-0	11	Leaching to Groundwater	30,000	5	5	50	20
BIS(2-CHLOROETHYL)ETHER	111-44-4	10	Residential	0.3	0.2	0.1	2	1
BIS-2-CHLORO ISOPROPYL ETHER	108-60-1	nr	nr	300	NA	NA	2	2
BIS(2-ETHYLHEXYL) PHTHALATE	117-81-7	770	Residential	nr	0.5	0.1	5	2
BUTYL BENZYL PHTHALATE	85-68-7	5,700	Residential	200	0.5	0.1	5	2
CARBAZOLE	86-74-8	540	Residential	110,000	0.2	0.05	2	2
CHRYSENE	218-01-9	260	Residential	50	0.05	0.05	0.5	0.3
DIBENZ(a,h)ANTHRACENE	53-70-3	0.26	Residential	0.05	0.05	0.05	0.5	0.2
DIBENZOFURAN	132-64-9	130	Residential	3,700	0.05	0.05	0.5	0.1

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		RAG	Exposure Scenario*		REPORTING LIMITS (mg/Kg)	MINIMUM DETECTION LIMITS (mg/Kg)	REPORTING LIMITS (ug/L)	MINIMUM DETECTION LIMITS (ug/L)
DIETHYLPHTHALATE	84-66-2	10,000	Residential	6,000	0.5	0.1	5	3
DI-N-BUTYLPHTHALATE	84-74-2	10,000	Residential	700	0.5	0.04	5	5
DI-N-OCTY PHTHALATE	117-84-0	1,600	Residential	120	0.5	0.05	2	1
FLUORANTHENE	206-44-0	5,000	Residential	300	0.05	0.05	0.5	0.4
FLUORENE	86-73-7	120	Leaching to Groundwater	300	0.05	0.05	0.5	0.3
HEXACHLOROBENZENE	118-74-1	6.8	Residential	0.2	0.2	0.1	2	2
HEXACHLOROBUTADIENE	87-68-3	130	Residential	4	0.2	0.2	2	2
HEXACHLOROCYCLOPENTADIENE	77-47-4	nr	nr	40	1	0.1	10	0.07
HEXACHLOROETHANE	67-72-1	93	Residential	5	0.2	0.04	2	1
INDENO(1,2,3-cd)PYRENE	193-39-5	0.26	Residential	0.5	0.05	0.05	0.5	0.2
ISOPHORONE	78-59-1	nr	nr	400	0.5	0.1	5	3
NAPHTHALENE	91-20-3	1.7	Leaching to Groundwater	10	0.05	0.05	0.5	0.3
NITROBENZENE	98-95-3	nr	nr	1	0.2	0.2	2	2
PENTACHLOROPHENOL	87-86-5	20	Residential	0.9	1	0.1	10	1
PHENANTHRENE	85-01-8	97	Leaching to Groundwater	nr	0.05	0.05	0.5	0.3
PHENOL	108-95-2	10,000	Residential	2,000	0.2	0.1	2	1
PYRENE	129-00-0	3,700	Residential	200	0.05	0.05	0.5	0.4
Polychlorinated bihenyls - PCBs (Analytical Method 8082)								
Aroclor 1016	12674-11-2	4.9	Residential	39	0.2	0.005	0.3	0.1
Aroclor 1221	11104-28-2	nr	nr	nr	0.2	0.005	0.3	0.1
Aroclor 1232	11141-16-5	nr	nr	nr	0.2	0.005	0.3	0.1
Aroclor 1242	53469-21-9	nr	nr	nr	0.2	0.005	0.3	0.1
Aroclor 1248	12672-29-6	nr	nr	nr	0.2	0.005	0.3	0.1
Aroclor 1254	11097-69-1	nr	nr	nr	0.2	0.005	0.3	0.1
Aroclor 1260	11096-82-5	nr	nr	nr	0.2	0.003	0.3	0.1
Total PCBs	1336-36-3	2.4	Residential	0.5	0.2	nr	0.3	nr
Pesticides (Analytical Method 8081)								
alpha-BHC	319-84-6	1.7	Residential	0.06	0.04	0.008	0.05	0.007
beta-BHC	319-85-7	6.0	Residential	0.06	0.04	0.008	0.05	0.008
delta-BHC	319-86-8	nr	nr	0.06	0.04	0.008	0.05	0.007
LINDANE	58-89-9	0.61	Residential	0.03	0.04	0.008	0.05	0.008
HEPTACHLOR	76-44-8	1.3	Residential	0.07	0.04	0.007	0.05	0.02
ALDRIN	309-00-2	0.64	Residential	0.02	0.04	0.009	0.05	0.009
HEPTACHLOR EPOXIDE	1024-57-3	1.2	Residential	0.04	0.04	0.009	0.05	0.01
ENDOSULFAN I (Endosulfan)	959-98-8	nr	nr	40	0.04	0.01	0.05	0.005

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		RAG	Exposure Scenario*		REPORTING LIMITS (mg/Kg)	MINIMUM DETECTION LIMITS (mg/Kg)	REPORTING LIMITS (ug/L)	MINIMUM DETECTION LIMITS (ug/L)
DIELDRIN	60-57-1	0.68	Residential	0.02	0.04	0.01	0.05	0.03
4,4'-DDE	72-55-9	nr	nr	1	0.04	0.02	0.05	0.02
ENDRIN	72-20-8	40	Residential	2	0.04	0.009	0.05	0.01
ENDOSULFAN II (Endosulfan)	33213-65-9	nr	nr	40	0.04	0.02	0.05	0.03
4,4'-DDD	72-54-8	45	Residential	1	0.04	0.04	0.05	0.009
ENDOSULFAN SULFATE	1031-07-8	nr	nr	nr	0.04	0.01	0.05	0.02
4,4'-DDT	50-29-3	38	Residential	1	0.04	0.01	0.05	0.02
METHOXYCHLOR	72-43-5	670	Residential	40	0.04	0.01	0.05	0.05
ENDRIN KETONE	53494-70-5	nr	nr	nr	0.04	0.02	0.05	0.02
ENDRIN ALDEHYDE	7421-93-4	nr	nr	nr	0.04	0.02	0.05	0.02
ALPHA-CHLORDANE	5103-71-9	nr	nr	nr	0.04	0.01	0.05	0.008
GAMMA-CHLORDANE	5103-74-2	nr	nr	nr	0.04	0.01	0.05	0.009
TOXAPHENE	8001-35-2	nr	nr	0.3	0.2	0.02	0.4	0.4
Herbicides (Analytical Method 8151A)								
2,4,5-T	93765	1,300	Residential	70	0.049	0.049	0.13	0.13
2,4,5-TP (Silvex)	93721	1,100	Residential	60	0.049	0.049	0.13	0.13
2,4-D	94757	nr	nr	nr	0.049	0.049	0.13	0.13
2,4-DB	94826	nr	nr	nr	0.49	0.49	1.3	1.3
Dalapon	75990	nr	nr	200	0.049	0.049	0.13	0.13
Dicamba	1918009	nr	nr	200	0.098	0.098	0.25	0.25
Dichloroprop	120365	nr	nr	nr	0.049	0.049	0.13	0.13
Dinoseb	88857	130	Residential	7	0.098	0.098	0.25	0.25
Volatile Petroleum Hydrocarbons - VPH (Analytical Method MADEPVPH-04-1.1)								
BENZENE	71-43-2	0.51	Leaching to Groundwater	4	0.1	0.01	1	0.2
ETHYLBENZENE	100-41-4	0.81	Leaching to Groundwater	30	0.1	0.004	2	0.2
METHYL TERT-BUTYL ETHER	1634-04-4	0.19	Leaching to Groundwater	35	0.1	0.02	2	0.2
NAPHTHALENE	91-20-3	1.7	Leaching to Groundwater	10	0.2	0.01	5	0.1
TOLUENE	108-88-3	8.1	Leaching to Groundwater	600	0.1	0.02	2	0.3
m&p-XYLENES (xylenes)	1330-20-7	26	Leaching to Groundwater	790	0.1	0.02	2	0.6
o-XYLENES (xylenes)	95-47-6	26	Leaching to Groundwater	790	0.1	0.01	2	0.2
C5-C8 ALIPHATICS	DEP2038	1,400	Residential	300	5	2	100	60
C9-C12 ALIPHATICS	DEP2039	2,700	Residential	700	5	1	100	50
C9-C10 AROMATICS	DEP2040	75	Leaching to Groundwater	200	5	0.05	100	5

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		RAG	Exposure Scenario*		REPORTING LIMITS (mg/Kg)	MINIMUM DETECTION LIMITS (mg/Kg)	REPORTING LIMITS (ug/L)	MINIMUM DETECTION LIMITS (ug/L)
Extractable Petroleum Hydrocarbons - EPH (Analytical Method MADEPEPH 2004-1.1)								
EPH_C9-C18 ALIPHATICS	DEP2043	2,700	Residential	700	10	3	50	10
EPH_C19-C36 ALIPHATICS	DEP2042	10,000	Residential	10,000	10	9	50	30
EPH_C11-C22 AROMATICS	DEP2041	460	Leaching to Groundwater	200	10	6	50	40
2-METHYLNAPHTHALENE	91-57-6	3.6	Leaching to Groundwater	10	0.1	0.06	0.5	0.3
ACENAPHTHENE	83-32-9	170	Leaching to Groundwater	12	0.1	0.04	0.5	0.4
ACENAPHTHYLENE	208-96-8	68	Leaching to Groundwater	14	0.1	0.05	0.5	0.3
ANTHRACENE	120-12-7	2,400	Leaching to Groundwater	20	0.1	0.03	0.5	0.3
BENZO(a)ANTHRACENE	56-55-3	2.6	Residential	0.5	0.1	0.03	0.5	0.3
BENZO(a)PYRENE	50-32-8	0.26	Residential	0.05	0.1	0.03	0.2	0.1
BENZO(b)FLUORANTHENE	205-99-2	2.6	Residential	0.5	0.1	0.03	0.5	0.2
BENZO(k)FLUORANTHENE	207-08-9	26	Residential	5	0.1	0.04	0.5	0.2
BENZO(g,h,i)PERYLENE	191-24-2	3,700	Residential	14,000	0.1	0.04	0.5	0.2
CHRYSENE	218-01-9	260	Residential	50	0.1	0.03	0.5	0.3
DIBENZO(a,h)ANTHRACENE	53-70-3	0.26	Residential	0.05	0.1	0.05	0.5	0.2
FLUORANTHENE	206-44-0	5,000	Residential	300	0.1	0.06	0.5	0.4
FLUORENE	86-73-7	120	Leaching to Groundwater	15	0.1	0.04	0.5	0.3
INDENO(1,2,3-cd)PYRENE	193-39-5	2.6	Residential	0.5	0.1	0.04	0.5	0.2
NAPHTHALENE	91-20-3	1.7	Leaching to Groundwater	9.7	0.1	0.06	0.5	0.3
PHENANTHRENE	85-01-8	97	Leaching to Groundwater	23	0.1	0.08	0.5	0.3
PYRENE	129-00-0	3,700	Residential	200	0.1	0.04	0.5	0.4

Notes:

* Most conservative (lowest concentration) applicable regulatory guideline selected for the purposes of this table, mg/kg = milligrams per kilogram, ug/L = micrograms per liter, nr = not regulated

Regulatory guideline exceeds corresponding Reporting Limit or Maximum Detection Limit for aqueous sample

Regulatory guideline exceeds corresponding Reporting Limit or Maximum Detection Limit for soil or sediment sample

MEDEP = Maine Department of Environmental Protection, MEDHS = Maine Department of Human Services, Red Font = Laboratory MDL OR RL exceeds corresponding regulatory standard.

2013 MEDEP RAGs = Maine Remedial Action Guidelines for Sites Contaminated with Hazardous Substances, Revised May 8, 2013

2012 MEGs = Maine Center for Disease Control Maximum Exposure Guidelines for Drinking Water, dated October 19, 2012

CHIP, WIPE, AND SWEEP SAMPLING

SOP#: 2011
DATE: 11/16/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) outlines the recommended protocol and equipment for collection of representative chip, wipe, and sweep samples to monitor potential surficial contamination.

This method of sampling is appropriate for surfaces contaminated with non-volatile species of analytes (i.e., PCB, PCDD, PCDF, metals, cyanide, etc.) Detection limits are analyte specific. Sample size should be determined based upon the detection limit desired and the amount of sample requested by the analytical laboratory. Typical sample area is one square foot. However, based upon sampling location, the sample size may need modification due to area configuration.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure or other procedure limitations. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Since surface situations vary widely, no universal sampling method can be recommended. Rather, the method and implements used must be tailored to suit a specific sampling site. The sampling location should be selected based upon the potential for contamination as a result of manufacturing processes or personnel practices.

Chip sampling is appropriate for porous surfaces and is generally accomplished with either a hammer and chisel, or an electric hammer. The sampling device should be laboratory cleaned and wrapped in clean, autoclaved aluminum foil until ready for use. To collect the sample, a measured and marked off area is chipped both horizontally and vertically to an even depth of 1/8 inch. The sample is then transferred to the proper sample container.

Wipe samples are collected from smooth surfaces to indicate surficial contamination; a sample location is measured and marked off. While wearing a new pair of surgical gloves, a sterile gauze pad is opened, and soaked with solvent. The solvent used is dependent on the surface being sampled. This pad is then stroked firmly over the sample surface, first vertically, then horizontally, to ensure complete coverage. The pad is then transferred to the sample container.

Sweep sampling is an effective method for the collection of dust or residue on porous or non-porous surfaces. To collect such a sample, an appropriate area is measured off. Then, while wearing a new pair of disposable surgical gloves, a dedicated brush is used to sweep material into a dedicated dust pan. The sample is then transferred to the proper sample container.

Samples collected by all three methods are then sent to the laboratory for analysis.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Samples should be stored out of direct sunlight to reduce photodegredation, cooled to 4°C and shipped to the laboratory performing the analysis. Appropriately sized laboratory cleaned, glass sample jars should be used for sample collection. The amount of sample required will be determined in concert with the analytical laboratory.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

This method has few significant interferences or problems. Typical problems result from rough porous

surfaces which may be difficult to wipe, chip, or sweep.

5.0 EQUIPMENT

Equipment required for performing chip, wipe, or sweep sampling is as follows:

- Lab clean sample containers of proper size and composition
- Site logbook
- Sample analysis request forms
- Chain of Custody records
- Custody seals
- Field data sheets
- Sample labels
- Disposable surgical gloves
- Sterile wrapped gauze pad (3 in. x 3 in.)
- Appropriate pesticide (HPLC) grade solvent
- Medium sized laboratory cleaned paint brush
- Medium sized laboratory cleaned chisel
- Autoclaved aluminum foil
- Camera
- Hexane (pesticide/HPLC grade)
- Iso-octane
- Distilled/deionized water

6.0 REAGENTS

Reagents are not required for preservation of chip, wipe or sweep samples. However, reagents will be utilized for decontamination of sampling equipment.

7.0 PROCEDURES

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain necessary sampling and monitoring equipment.

3. Decontaminate or preclean equipment, and ensure that it is in working order.

4. Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate.

5. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.

6. Mark all sampling locations. If required the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

7.2 Chip Sample Collection

Sampling of porous surfaces is generally accomplished by using a chisel and hammer or electric hammer. The sampling device should be laboratory cleaned or field decontaminated as per the Sampling Equipment Decontamination SOP. It is then wrapped in cleaned, autoclaved aluminum foil. The sampler should remain in this wrapping until it is needed. Each sampling device should be used for only one sample.

1. Choose appropriate sampling points; measure off the designated area. Photo documentation is optional.

2. Record surface area to be chipped.

3. Don a new pair of disposable surgical gloves.

4. Open a laboratory-cleaned chisel or equivalent sampling device.

5. Chip the sample area horizontally, then vertically to an even depth of approximately 1/8 inch.

6. Place the sample in an appropriately prepared sample container with a Teflon lined cap.

7. Cap the sample container, attach the label and custody seal, and place in a plastic bag. Record all pertinent data in the site logbook and on field data sheets. Complete the sampling analysis request form and chain of custody record before taking the next sample.

8. Store samples out of direct sunlight and cool to 4EC.

9. Follow proper decontamination procedures then deliver sample(s) to the laboratory for analysis.

7.3 Wipe Sample Collection

Wipe sampling is accomplished by using a sterile

gauze pad, adding a solvent in which the contaminant is most soluble, then wiping a pre-determined, pre-measured area. The sample is packaged in an amber jar to prevent photodegradation and packed in coolers for shipment to the lab. Each gauze pad is used for only one wipe sample.

1. Choose appropriate sampling points; measure off the designated area. Photo documentation is optional.

2. Record surface area to be wiped.

3. Don a new pair of disposable surgical gloves.

4. Open new sterile package of gauze pad.

5. Soak the pad with solvent of choice.

6. Wipe the marked surface area using firm strokes. Wipe vertically, then horizontally to insure complete surface coverage.

7. Place the gauze pad in an appropriately prepared sample container with a Teflon-lined cap.

8. Cap the sample container, attach the label and custody seal, and place in a plastic bag. Record all pertinent data in the site logbook and on field data sheets. Complete the sampling analysis request form and chain of custody record before taking the next sample.

9. Store samples out of direct sunlight and cool to 4ºC.

10. Follow proper decontamination procedures, then deliver sample(s) to the laboratory for analysis.

7.4 Sweep Sample Collection

Sweep sampling is appropriate for bulk contamination. This procedure utilizes a dedicated, hand held sweeper brush to acquire a sample from a pre-measured area.

1. Choose appropriate sampling points; measure off the designated area. Photo documentation is optional.

2. Record the surface area to be swept.

3. Don new pair of disposable surgical gloves.

4. Sweep the measured area using a dedicated brush; collect the sample in a dedicated dust pan.

5. Transfer sample from dust pan to sample container.

6. Cap the sample container, attach the label and custody seal, and place in a plastic bag. Record all pertinent data in the site log book and on field data sheets. Complete the sampling analysis request form and chain of custody record before taking the next sample.

7. Store samples out of direct sunlight and cool to 4EC.

8. Leave contaminated sampling device in the sample material, unless decontamination is practical.

9. Follow proper decontamination procedures, then deliver sample(s) to the laboratory for analysis.

8.0 CALCULATIONS

Results are usually provided in mg/g, µg/g, mass per unit area, or other appropriate measurement. Calculations are typically done by the laboratory.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

The following general quality assurance procedures apply:

1. All data must be documented on standard chain of custody forms, field data sheets or within the site logbook.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

The following specific quality assurance activities apply to wipe samples:

For wipe samples, a blank should be collected for each sampling event. This consists of a sterile gauze pad, wet with the appropriate solvent, and placed in a prepared sample container. The blank will help identify potential introduction of contaminants via the sampling methods, the pad, solvent or sample container. Spiked wipe samples can also be collected to better assess the data being generated. These are prepared by spiking a piece of foil of known area with a standard of the analyte of choice. The solvent containing the standard is allowed to evaporate, and the foil is wiped in a manner identical to the other wipe samples.

Specific quality assurance activities for chip and sweep samples should be determined on a site specific basis.

10.0 DATA VALIDATION

A review of the quality control samples will be conducted and the data utilized to qualify the environmental results.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow EPA, OSHA and corporate health and safety procedures.

12.0 REFERENCES

U.S. EPA, A Compendium of Superfund Field Operation Methods. EPA/540/5-87/001.

NJDEP Field Sampling Procedures Manual, February, 1988.

UBC School of Population & Public Health Laboratory

Standard Operating Procedure: Mercury Analyzer

Introduction
The Jerome 431-X Mercury Vapor Analyzer is designed for easy and accurate analysis of mercury vapor in the workplace environment, and to aid in locating and evaluating mercury spills. Mercury is highly toxic and high exposures can result in death. It is important to remember that mercury has a very low vapor pressure. This means that at room temperature, dangerous levels of vapor can develop.

The mercury analyzer works by drawing a precise amount of air across a thin gold film. The mercury interacts with the gold, altering its resistance to the sensor. The change in resistance is proportionate to the mercury concentration in the environment. *(Refer to Manual for more detailed description).*

Measurement Range:	0.001 to 0.999 mg/m3
Limit of Detection:	0.001 mg/m3
Warnings of Interferences:	Does not perform well in sewage treatment facilities, or other environments with high levels of ammonia.

Regenerating and Zeroing the Instrument
Sensor regeneration and zeroing is required at the beginning of the sampling day, the end of the sampling day, or whenever the instrument is saturated. See the "Sampling" section for details on saturation measurement.

1. Press the "ON" button to turn on the analyzer and allow 1 minute warm up time.
2. Connect the analyzer to an AC power source and press the "REGEN" button to start the 10-minute sensor regeneration cycle. **Do not interrupt the cycle.**
3. Once regeneration has completed, wait for 30 minutes prior to zeroing the analyzer. *(In the case of an emergency such as a mercury spill, the unit can be rezeroed immediately following regeneration; however, results will not be as accurate.)*
4. While pressing the "ZERO" button, adjust the "zero adjust" potentiometer using the trimmer tool until the digital meter reads 0.
5. Attach the long black intake tube to the inlet on the front of the analyzer, and attach the zero filter to the intake tube with the flexible tubing provided.
6. Press the "SAMPLE" button to check if analyzer is properly zeroed. If mercury reading does not read 0.00 at the end of the run, repeat step 4 again.
7. Disconnect the zero filter. The mercury analyzer is now ready for sampling.

Single Readings
This mode produces the greatest accuracy of measurement (± 5% at 0.100 mg/m^3.)

1. Remove the zero intake filter and flexible tubing from the black intake tube.
2. Press the "SAMPLE" button once.
3. The mercury concentration will be displayed (in mg/m^3) after a 12-second cycle.
4. Press the "OFF" button to turn off the analyzer.

Multiple Readings (Survey Mode)
This mode is used to locate mercury spills or to assess areas of potentially high concentrations of mercury. The current mercury concentration is taken and displayed every 3 seconds.

***Note**: the sampling in this mode is not as accurate due to decreased sample volume (± 20% at 0.100 mg/m^3.)*

1. Remove the zero intake filter and flexible tubing from the black intake tube.
2. Press and hold the "SAMPLE" button.
   - ***To lock the analyzer*** in the survey mode, hold the "SAMPLE" button down until the display shows "- 0.00" with the "-" sign flashing. Press the "ZERO" button, then release the "SAMPLE" button.
3. The analyzer will display the concentration in 12 seconds and updated every 3 seconds.
4. When you are finished surveying, release the "SAMPLE" button
5. Press the "OFF" button to turn off the analyzer.

At the end of the day, instrument regeneration is required. It is vital that mercury contamination is not allowed to stay on the gold film sensor overnight.

Note: During sampling, the display will indicate the level of saturation of the sensor as follows:
.- = 0-25% sensor saturation
.-- = 25-50% sensor saturation
.--- = 50-75% sensor saturation
-.--- = 75-100% sensor saturation

Fully assembled instrument

1.0 INTRODUCTION

Porous media, such as concrete, adsorbs liquids impacted with polychlorinated biphenyls from discharges and spills. Wipe sampling and coring are the two primary methodologies for collecting representative samples of this medium.

1.1 PURPOSE

This Standard Operating Procedure (SOP) is a general reference for the proper equipment and techniques for concrete sampling. These techniques should be followed whenever applicable, although site-specific conditions or project-specific plans may require adjustments in methodology.

1.2 SCOPE

The purpose of these procedures is to enable the user to collect representative and defensible wipe samples and to facilitate planning of the field sampling effort.

2.0 WIPE SAMPLING GUIDELINES

Collection of wipe samples will follow the guidelines as set forth in 40 CFR 761.123 Standard Wipe Test.

2.1 WIPE SAMPLE COLLECTION METHOD

Prior to sampling, the area should be cleared of any debris or potential cross-contamination from other activities. One wipe sample should be taken from a 100-square centimeter (cm) surface area (10 cm by 10 cm) at each point for sample collection. The surface should be flat. Wipes are usually composed of filter paper, gauze or glass wool. Wipe samples should be collected from flat, smooth surface areas of at least 10 cm by 10 cm. Wipe samples should be collected from areas expected to be representative of contaminant distribution, such as areas with visible staining.

Samples collected for volatile organic analyses should be collected using a wipe that is wet with the appropriate solvent for the analyte to be collected. Samples collected for metals analysis should be collected using a wipe that is wet with deionized water. One 10-cm square template should be used for each sample location. With the sampling media, wipe downward and then across the template.

Upon completion of the sample collection, the wipe sample is placed in a glass jar and the appropriate preservative is added to the sample container. The wipe samples shall be placed in separate containers and stored in a cooler packed with ice for transportation to the laboratory.

2.2 SAMPLE CONTAINERS

A complete set of sample containers should be prepared by the laboratory prior to going into the field. The laboratory should provide the proper containers with the required preservatives. The laboratory's quality manual should provide a complete description of the procedures used to clean and prepare the containers. The containers should be labeled in the field with the date, sample

identification, project name, collectors' name, time of collection, parameters to be analyzed, and preservative. The sample containers should be kept in a cooler (at zero plus or minus 2 degrees centigrade) until they are received by the laboratory. One cooler should be used to store the unfilled bottles and another to store the samples. All sample bottles and equipment will be kept away from fuels and solvents.

When wipe samples are to be analyzed for volatile organic analyses, samples will be carefully collected in a manner than minimizes volatilization.

3.0 CONCRETE CORE

The collection of concrete core samples are specified in 40 CRF 761.286 to be a minimum of 1 inch in diameter (≤2 centimeters and ≤3 centimeters) and a maximum of 3 inches long (7.5 centimeters) from within a sampling grid.

EPA Region I developed an SOP for sampling concrete which will be followed in all TIMET field events unless otherwise noted in the project-specific sampling and analysis plan (see attached).

DISCLAIMER
This SOP provided general guidance for TIMET contractors and subcontractors for technical issues addressed during environmental site investigation and remediation activities. It is noted, however, that each site and project is unique and these guidelines are not a substitute for common sense and good management practices based on professional training and experience. In addition, individual contract terms may affect the implementation of this SOP. TIMET contractors reserve the unrestricted right to change, modify or not apply these guidelines in their sole, complete, and unrestricted discretion to meet certain circumstances, contractual requirements, site conditions, or job requirements.

REGION I, EPA-NEW ENGLAND

DRAFT
STANDARD OPERATING PROCEDURE
FOR SAMPLING CONCRETE IN THE FIELD

U.S. EPA-NEW ENGLAND
Region I
Quality Assurance Unit Staff
Office of Environmental Measurement and Evaluation

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Region I, EPA New England

Standard Operating Procedure for Sampling Concrete in the Field

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Region I, EPA New England

Standard Operating Procedure for Sampling Concrete in the Field

1.0 Scope and Application

The following Standard Operating Procedure (SOP) describes a concrete sampling technique which uses an impact hammer drill to generate a uniform, finely ground, powder which is easily homogenized, extracted and analyzed. This procedure is primarily geared at providing enough sample for one or two different analyses at a time. That is, the time required to generate sufficient sample for a full suite of analyses may be impractical. The concrete powder is suitable for all types of environmental analyses, with the exception of volatile compounds, and may be analyzed in the field or at a fixed laboratory. This procedure is applicable for the collection of samples from concrete floors, walls, and ceilings.

The impact hammer drill is far less labor intensive than previous techniques using coring devices, or hammers and chisels. It allows for easy selection of sample location and sample depth. Not only can the project planner control the depth to sample into the concrete, from surface samples (0 - ½ inch) down to a core of the entire slab, but the technique can also be modified to collect samples at discrete depths within the concrete slab.

Another issue with concrete sampling is the fact that the amount of time spent drilling translates into the weight of sample produced. Thus, to maximize sampling time, it is important to know the minimum amount of sample required for each analysis. To do this, the project planner should take the following steps: 1) Use the Data Quality Objective (DQO) process and familiarity with the site to develop the objectives of the sampling project and the depth(s) of sample to be collected. 2) Review the site history and any previous data collected to determined possible contaminants of concern. 3) Establish the action levels for those possible contaminants and determine the appropriate analytical methods (both field and/or fixed laboratory) to meet the DQOs of the project. 4) Based on the detection limits of these methods, determine the amount of sample required for each analysis and the total sample weight require for each sample location (including quality control samples).

As with any environmental data collection project, all aspects of a concrete sampling episode should be well thought out, prior to going out in the field, and thoroughly described in a Quality Assurance Project Plan (QAPP). The QAPP should clearly state the DQOs of the project and document a complete Quality Assurance/Quality Control program to reconcile the data generated with the established DQOs. For more information on these subjects, refer to EPA documents QA/R-5, EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, and QA/G-4, Guidance for the Data Quality Objective Process.

2.0 Method Summary

A one-inch diameter carbide drill bit is used in a rotary impact hammer drill to generate a fine concrete powder suitable for analysis. The powder is placed in a sample container and homogenized for field or fixed laboratory analysis. The procedure can be used to sample a single depth into the concrete, or may be modified to sample the concrete at distinctly different depth zones. The modified depth sampling procedure is designed to minimize any cross contamination between the sampling zones. If different sampling depths are required, two different diameter drill bits and a vacuum sampling apparatus are employed.

3.0 Health and Safety

Eye and hearing protection are required at all times during sample drilling. A small amount of dust is generated during the drilling process. Proper respiratory protection and/or a dust control system must be in place at all times during sampling.

4.0 Interferences and Potential Problems

Since this sampling technique produces a finely ground uniform powder, physical matrix effects from variations in the sample consistency (i.e., particle size, uniformity, homogeneity, and surface condition) are minimized. Matrix spike analysis of a sample is highly recommended to monitor for any matrix related interferences.

As stated in Section 1.0 above, this sampling procedure is not recommended for volatile organic compound (VOC) analysis. The combination of heat generated during drilling and the exposure of a large amount of surface area will greatly reduce VOC recovery. If low boiling point semi-volatile compounds (i.e., naphthalene) are being analyzed, then the drill speed should be reduced to minimize heat build-up.

5.0 Equipment and Supplies

5.1 Single Depth Concrete Sampling

5.1.1 Rotary impact hammer drill
5.1.2 1-inch diameter carbide drill bits
5.1.3 Stainless steel scoopulas
5.1.4 Stainless steel spoonulas (for collecting sample in deeper holes, >2-inches)
5.1.5 Rectangular aluminum pans (to catch concrete during wall and ceiling sampling)
5.1.6 Gasoline powered generator (if alternative power source is required)

5.2 Multiple Depth Sampling (in addition to all the above)

5.2.1 ½ inch diameter carbide drill bits
5.2.2 Vacuum/sample trap assembly (see Section 7.2 and Figure 1)

5.2.2.1 Vacuum pump
5.2.2.2 2-hole rubber stopper
5.2.2.3 Glass tubing (to fit stopper)
5.2.2.4 Large glass test tubes, or Erlenmeyer flasks, for sample trap (several are suggested)
5.2.2.5 Polyethylene tubing for trap inlet (Tygon tubing may be used for the trap outlet)
5.2.2.6 Pasture pipets
5.2.2.7 Pipe cleaners
5.2.2.8 In-line dust filter (glass fiber filter, or equivalent)

6.0 Sample Containers, Preservation, and Storage

Concrete samples must be collected in glass containers for organic analyses, and may be collected in either glass or plastic containers for inorganic analyses. In general, a 2-ounce sample container with Teflon-lined cap (wide-mouth jars are preferred) will hold sufficient volume for most analyses. A 2-

ounce jar can hold roughly 90 grams sample. Note, samples which require duplicate and/or matrix spike/matrix spike duplicate analyses may require a larger sample container, or additional 2-ounce sample containers.

Organic samples are to be shipped on ice and maintained at 4°C (± 2°C) until the time of extraction and analysis. Inorganic samples may be shipped and stored at room temperature. Refer to 40 CFR Part 136 for guidelines on analysis holding times.

To maintain sample integrity, chain-of-custody procedures must be implemented at the time of sampling to 1) document all sample locations and associated field sample identification numbers, 2) document all quality control samples taken, including field duplicates, split samples for confirmatory analyses, and PE samples, and 3) document the transfer of field samples from field sampler to field chemist or fixed laboratory.

7.0 Procedure

7.1 Single Depth Concrete Sampling

Lock a 1-inch diameter carbide drill bit into the impact hammer drill and plug the drill into an appropriate power source. (A gasoline generator will be needed if electricity is not available.) For easy identification, sample locations may be pre-marked using a crayon or a non-contaminating spray paint. (Note, the actual drilling point must not be marked.) Depending on the appearance of the sample location, or the objectives of the sampling project, it may be desired to wipe the concrete surface with a clean dry cloth prior to drilling. All sampling decisions of this nature should be noted in the sampling logbook. Begin drilling in the designated location. Apply steady even pressure and let the drill do the work. Applying too much pressure will generate excessive heat and dull the drill bit prematurely. The drill will provide a finely ground concrete powder that can be easily collected, homogenized and analyzed. Having several decontaminated impact drill bits on hand will help expedite sampling when numerous sample locations are to be drilled.

<u>Sample Collection</u>

A ½-inch deep hole (using a 1-inch diameter drill bit) generates about 10 grams of concrete powder. Based on this and the action levels for the project, determine the sampling depth, and/or the number of sample holes to be composited, to generate sufficient sample volume for all of the required analyses. (Note, with the absorbency of concrete, a ½-inch deep hole can be considered a surface sample.)

A decontaminated stainless steel scoopula can be used to collect the sample. The powder can either be collected directly from the surface of the concrete and/or the concrete powder can be scraped back into the hole and the less rounded back edge of the scoopula can be used to collect the sample. For holes greater than 2-inches in depth, a stainless steel spoonula will make it easier to collect the sample from the bottom of the hole.

To ensure collection of a representative sample when multiple analyses are required, a concrete sample should always be collected and homogenized in a single container and then divided up into the individual containers for the various analyses or split samples. This is particularly important when sample holes are deep, or when several holes are drilled adjacent to each other to form a sample composite.

Wall and Ceiling Sampling

A team of two samplers will be required for wall and ceiling sampling. The second person will be needed to hold a clean catch surface (i.e., an aluminum pan) below the drill to collect the falling powder. For wall samples, a scoopula, or spoonula, can be used to collect remaining concrete powder from within the hole. For ceiling holes, it may be necessary to drill the hole at an angle so the concrete powder can fall freely in the collection plan (and avoid falling on the drill). Another alternative might be to use the chuck-end of the drill bit and punch a hole through the center of the collection pan. The drill bit is then mounted through the pan and into the drill. Thus, the driller can be drilling straight up while the assistant steadies the pan to catch the falling dust. As a precaution, it may be advantageous to tape a piece of plastic around the drill, just below the chuck, to avoid dust contaminating the body of the drill and entering the mechanical vents. (Note, the plastic should deflect dust from the drill, but be loose enough underneath to allow for proper ventilation.)

7.2 Multiple Depth Concrete Sampling

The above method for concrete sampling can also be used to collect samples from different depths within the concrete. To do this, two different sized drill bits (i.e., ½ inch and 1 inch) and a simple vacuum pump with a vacuum trap assembly is required (see Figure 1). First, the 1 inch drill bit is used to drill to the first level and the concrete sample is collected as described in Section 7.1. The vacuum pump is then turned on and the hole is cleaned out using the vacuum trap assembly. The drill bit is then changed to the ½ inch bit and the next depth is drilled out (the ½ inch bit is used to avoid contact with the sides of the first hole). A clean tube or flask is placed on the vacuum trap, and the sample from the second drilling is collected. To go further, the 1 inch drill is used to open up the hole to the second level, the hole is cleared, and then the ½ inch drill is used again to go to a third level, etc. Note, the holes and concrete surface should be vacuumed thoroughly to minimize any cross-contamination between sample depths.

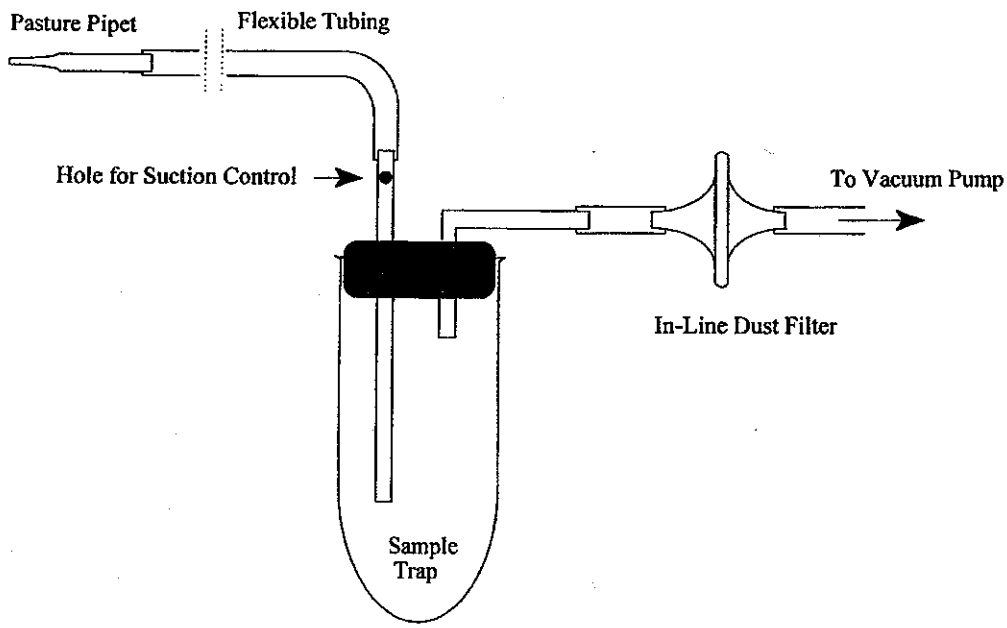
Vacuum Trap Design and Clean-out

The trap presented in Figure 1 is a convenient and thorough way for collecting and removing concrete powder from drilled holes. The trap system is designed to allow for control of the suction from the vacuum pump and easy trap clean-out between samples. Note, by placing a hole in the inlet tube (see Figure 1), a finger on the hand holding the trap can be used to control the suction at the sampling tip. Thus, when this hole is left completely open, there will be no suction, and the sampler can have complete control over where and what to sample. To change-out between samples the following steps should be taken: 1) The pasture pipet and piece of polyethylene tubing at the sample inlet should be replaced with new materials, 2) the portion of the rubber stopper and glass tubing that was in the trap should be wiped down with a clean damp paper towel (wetted with deionized water) and then dried with a fresh paper towel, 3) a clean pipe cleaner should be drawn through the glass inlet tube to remove any concrete dust present, and 4) the glass tube or flask used to collect the sample should swapped out with a clean decontaminated sample trap. Having several clean tubes or flasks on hand will facilitate change-out between samples.

7.3 Decontamination Procedure

Necessary supplies for decontamination include: two small buckets, a scrub brush, potable water, deionized water, a squirt bottle for the deionized water, and paper towels. The first bucket contains a soap and potable water solution, and the second bucket contains just potable water. Place all used drill bits and

Figure 1



utensils in the soap and water bucket. Scrub each piece thoroughly using the scrub brush. Note, the concrete powder does cling to the metal surfaces, so care should be taken during this step, especially with the twists and curves of the drill bits. Next, rinse each piece in the potable water bucket, and follow with a deionized water rinse from the squirt bottle. Place the deionized water rinsed pieces on clean paper towels and individually dry and inspect each piece. Note, all pieces should be dry prior to reuse.

8.0 Field Documentation

All Site related documentation and reports generated from concrete sampling should be maintained in the central Site file. If personal logbooks are used, legible copies of all pertinent pages must be placed in the Site file.

8.1 Field Logbooks

All field documentation should be maintained in bound logbooks with numbered pages. If loose-leaf logsheets are used to document site activities, extra care should be taken in keep track of all logsheets. The original copy of all logsheets should be maintained in the central Site file. Note, all sample locations must be documented by tying in their location to a detailed site map, or by using two or more permanent landmarks. The following information should be documented in the field logbooks:

- Site name and location,
- EPA Site Manager,
- Name and affiliation of field samplers (EPA, Contractor company name, etc.),
- Sampling date,
- Sample locations and IDs,
- Sampling times and depths, and
- Other pertinent information or comments

8.2 Sample Labeling and Chain-of-Custody

8.2.1 Sample Labels

Sample labels will be affixed to all sample containers. Labels must contain the following information:

- Project name,
- Sample number, and/or location
- Date and time of sampling,
- Analysis,
- Preservation, and
- Sampler's name.

8.2.2 Chain-of-Custody

All samples must be traced from collection, to shipment, to laboratory receipt and laboratory custody. The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. The COC form is signed by all individuals responsible for sampling, sample transport, and laboratory receipt. (Note, overnight deliver services, often used with sample transport, are exempt from having to sign the COC form. However, copies of all shipping invoices must be kept with the COC documentation.) One copy of the COC is retained by the field sampling crew, while the original (top, signed copy) and remaining carbonless copies are placed in a zip-lock bag and taped to the inside lid of the shipping cooler. If multiple coolers are required for a sample shipment to a single laboratory, the COC need only be sent with one of the coolers. The COC should state how many coolers are included with the shipment. All sample shipments to different laboratories require individual COC forms. The original COC form accompanies the samples until the project is complete, and is then kept in the permanent project file. A copy of the COC is also kept with the project manager, the laboratory manager, and attached to the data package.

8.2.3 Custody Seal

The Custody seal is an adhesive-backed label which is also part of the chain-of-custody process. The custody seal is used to prevent tampering with the samples after they have been collected in the field and sealed in coolers for transit to the laboratory. The Custody seals are signed and dated by a sampler and affixed across the opening edges of each cooler containing samples. Clear packing tape should be wrapped around the cooler, and over the Custody seal, to secure the cooler and avoid accidental tampering with the Custody seal.

9.0 Quality Assurance and Quality Control (QA/QC)

A solid QA/QC program is essential to establishing the quality of the data generated so that proper project decisions can be made. The following are key quality control elements which should be incorporated into a concrete sampling and analytical program.

9.1 Equipment Blanks

An equipment blank should be performed on decontaminated drill bits and collection utensils at a frequency of 1 per 20 samples or 1 per day, whichever is greater. To prepare the equipment blank, place the decontaminated drill bit and utensils in a large clean stainless steel bowl. Pour sufficient deionized water into the bowl to fill all of the required sample containers. Next, stir the drill bit and utensils in the bowl with a clean utensil to thoroughly mix the blank. Finally, decant off the equipment blank into the sample containers. Note, a clean funnel may help to pour off the equipment blank into the containers.

9.2 Field Duplicates

Field duplicates are samples collected adjacent to each other (collocated) at the same sample location (not two aliquots of the same sample). Field duplicates not only help provide an indicator of overall precision, but measure the cumulative effects of both the field and analytical precision, and also measure the representativeness of the sample. Field duplicates must be prepared and analyzed at a frequency of 1 per 20 samples or 1 per non-related concrete matrix, whichever is greater. An example of a non-related concrete matrix might be the investigation of two different types of chemical spills.

Calculate the Relative Percent Difference (RPD) between the sample and its duplicate using Equation 1.

Equation 1

$$RPD = \frac{|S - D|}{\frac{(S + D)}{2}} \times 100$$

Where:

S = Original sample result
D = Duplicate sample result

The following general guidelines have been established for field duplicate criteria:

- If both the original and field duplicate values are ≥ practical quantitation limit (PQL), then the control limit for RPD is ≤50%,
- If one or both values are < PQL, then do not assess the RPD.

If more rigorous field duplicate criteria are needed to achieve project DQOs, then that criteria should be documented in the project QAPP.

If the field duplicate criteria specified above are not met, then flag that target element with an "*" on the final report for both the original and field duplicate samples. Report both the original and field duplicate

analyses; do not report the average. Field duplicate samples should be indicated on the sample ID. For example, the sample ID can contain the suffix "FD."

9.3 Laboratory Duplicates

Laboratory duplicates are two aliquots of the same sample that are prepared, homogenized and analyzed in the same manner. (Note, proper sample homogenization is critical in producing meaningful results.) The precision of the sample preparation and analytical methods is determined by performing a laboratory duplicate analysis. Laboratory duplicates can be prepared in the field and submitted as blind samples, or the laboratory can be requested to perform the laboratory duplicate analysis. In the case of laboratory prepared duplicates, the field sampling team must be sure to provide sufficient sample volume. Laboratory duplicates must be prepared and analyzed at a frequency of 1 per 20 samples or 1 per non-related concrete matrix, whichever is greater.

Calculate the RPD between the sample and its duplicate using Equation 1. The following general guidelines have been established for laboratory duplicate criteria:

- If both the original and laboratory duplicate values are ≥ PQL, then the control limit for RPD is ≤25%,
- If one or both values are < PQL, then do not assess the RPD.

If duplicate criteria are not met, then flag that target element with an "*" on the final report for both the original and duplicate samples. Report both the original and duplicate analyses; do not report the average.

9.4 Matrix Spike/Matrix Spike Duplicate Samples

Matrix spike/matrix spike duplicate samples (MS/MSDs) are two additional aliquots of a sample which are spiked with the appropriate compound(s) or analyte(s) of concern and then prepared and analyzed along with the original sample. (Note, proper sample homogenization, prior to spiking, is critical in producing meaningful results.) MS/MSDs help evaluate the effects of sample matrix on the analytical methods being used. The field sampling team must provide sufficient sample volume such that the field or fixed laboratory can prepare and analyze MS/MSDs at a frequency of 1 per 20 samples or 1 per non-related concrete matrix, whichever is greater.

Calculate the recovery of each matrix spike compound or analyte using Equation 2.

Equation 2

$$MSR = \frac{SSR - SR}{SA} \times 100$$

Where,

MSR	=	Matrix Spike Recovery,	SA	=	Spike Added
SSR	=	Spiked Sample Result,	SR	=	Sample Result

Calculate the relative percent difference (RPD) between the recoveries of each compound or analyte in the matrix spike and matrix spike duplicate using Equation 3.

Equation 3

$$RPD = \frac{|MSR - MSRD|}{\frac{(MSR + MSRD)}{2}} \times 100$$

Where,

MSR = Matrix Spike Recovery
MSRD = Matrix Spike Duplicate Recovery

9.5 Performance Evaluation Samples

In accordance with the EPA Region I Performance Evaluation Program Guidance, performance evaluation (PE) samples should be submitted for each type of analysis to be performed in the field or by the fixed laboratory performing full protocol EPA methods. PE samples provide information on the quality of the individual data packages. PE samples are certified standard reference materials (SRMs) from a source other than that used to calibrate the instrument. If both field and fixed laboratories are being used to analyze samples, at least one solid PE sample should undergo both field analysis and confirmatory full protocol EPA method analysis to facilitate data comparability. A copy of the certified values for the SRM must be submitted with the final data packages to facilitate data evaluation.

9.6 Data Verification and Validation

All field data and supporting information (including chain-of-custody) that is collected during a concrete sampling episode should be verified daily, by a person other than that performing the work, to check for possible errors.

During the project planning process, a plan for data validation should be established for all data, both for field and fixed laboratories. All data must be validated to assure that it is of a quality suitable to make project decisions. For help in developing a data validation program refer to Region I, EPA New England,

Data Validation Functional Guidelines for Evaluating Environmental Analyses.

9.7 Audits

9.7.1 Internal Audits

As part of the Quality Assurance/Quality Control Program for any sampling project, a series of internal audit checks should be instituted to monitor and maintain the integrity of the sample collection process. Timely internal reviews will insure that proper sampling, decontamination, chain-of-custody and quality control procedures are being followed. Also, the internal audit review is there to monitor any corrective actions taken, and/or institute corrective actions that should have been taken and were not. All corrective actions taken must be documented in an appropriate logbook, and if any corrective actions impact the final data reported, then they must also be documented in the final report narrative. The results of all internal audits must be documented in a report, and copies of the report issued to the Project Manager and the Quality Assurance Manager. The original copy of any audit report must remain with the main project file and be available for review.

9.7.2 External Audits

The Agency reserves the right to perform periodic field audits to ensure compliance with this SOP.

10.0 References

1) Guidance for the Data Quality Objective Process, QA/G-4, EPA/600/R-96/055, September 1994.

2) EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, QA/R-5, Interim Final, October 1997.

3) Guidance for the Preparation of Standard Operating Procedures for Quality-related Operations, QA/G-6, EPA/600/R-96/027, November 1995.

4) Region I, EPA-New England Data Validation Functional Guidelines for Evaluating Environmental Analyses, July 1996.

5) EPA Region I Performance Evaluation Program Guidance, July 1996.

6) U.S. EPA Code of Federal Regulations, 40 CFR, Part 136, Appendix B, Revised as of July 1995.

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SOP Number: 627.8151

Title: Chlorinated Herbicides by GC Using Methylation Derivatization

1.0 Scope and Application

This is a capillary gas chromatographic (GC) method for determining certain chlorinated acid herbicides and related compounds in aqueous, soil, and waste matrices. The following compounds can be determined:

Analyte	CAS Number
2,4,5-T	93-75-5
2,4,5-TP (Silvex)	93-72-1
2,4-D	94-75-7
2,4-DB	94-82-6
Dalapon	75-99-0
Dicamba	1918-00-9
Dichloroprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	7085-19-0
Pentachlorophenol	87-86-5

2.0 Summary of Method

2.1 Water Samples

A measured volume of sample, approximately 1L is adjusted to pH less than 2 with 12 N sulfuric acid and extracted with diethyl ether and then esterified with diazomethane. The derivatives are determined by gas chromatography with electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.2 Soil Samples

A measured mass of sample, approximately 30 g is sonicated with acetone and diethyl ether. The extract pH is adjusted to less than 2 with 12 N sulfuric acid and the diethyl ether is extracted and the esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

3.0 Interferences

3.1 Interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. The use of high purity reagents and solvents helps to minimize interference problems. A blank is prepared daily for each batch of extractions.

3.2 Matrix interference may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from source to source, depending upon the nature and diversity of the waste being analyzed.

4.0 Sample Collection, Preservation and Storage

4.1 Collect 1 liter of unpreserved sample in an amber glass container with a Teflon-lined cap.
4.2 Samples must be kept at 4°C until time of extraction.
4.3 Samples must be extracted within 7 days from time of collection.
4.4 Sample extracts must be stored at 4°C in the dark and analyzed within 28 days of extraction.

5.0 Equipment and Supplies

5.1 Refer to sample preparation SOP 217.8151 for preparation equipment.
5.2 Gas Chromatograph
- 5.2.1 Perkin Elmer 8500 or Autosystem with Turbochrom software
- 5.2.2 Column 1 (Primary column) – Restek CLP Pest II
- 5.2.3 Column 2 (Confirmation column) – Restek CLP-Pest
- 5.2.4 Detector – Dual Electron capture

6.0 Reagents and Standards

6.1 Refer to sample preparation 247.sonc herb

6.2 Hexane - Burdick and Jackson 217-4

6.3 Stock Standard Solutions- purchased. Concentrations below:

	Absolute #	Conc. ug/ml
Dalapon	#81505	200
Dicamba	#81506	200
Dichloroprop	#81507	200
2,4-D	#81501	200
Pentachlorophenol	#30038	200
2,4,5-TP	#81504	200
2,4,5-T	#81503	200
2,4 DB	#70531	1000
Pichloram	#30039	200
Dinoseb	#81508	200
MCPA / MCPP	#CUS12459	30,000

6.3.1 Working Calibration Standards:

	Volume (ul) of Stock Solution	Final Volume (hexane)
Dalapon	250	10 ml
Dicamba	500	10 ml
Dichloroprop	250	10 ml
2,4-D	250	10 ml
Pentachlorophenol	250	10 ml
2,4,5-TP	250	10 ml
2,4,5-T	250	10 ml
2,4 DB	500	10 ml
Pichloram	500	10 ml
Dinoseb	500	10 ml
MCPA / MCPP	500	10 ml

6.3.2 Calibration Check Standard: Dilute 100 ul of working standard (6.3.1) to 10 ml final volume in hexane:

	Conc. ug/ml
Dalapon	50
Dicamba	100
Dichloroprop	50
2,4-D	50
Pentachlorophenol	50
2,4,5-TP	50
2,4,5-T	50
2,4 DB	500

Pichloram	100
Dinoseb	100
MCPA / MCPP	15000

6.3.4 Herbicide Surrogate: Ultra #PPS-166, DCAA methyl ester solution at 100 ug/ml.

6.3.5 Calibration Verification Standard (CVS)- Herbicide Custom Stock, Absolute part #97477. Dilute 100 ul of custom stock + 100uL internal standard to 10mL in hexane.

6.3.6 Internal Standard (IS)- Ultra part # PPS-351-1. Dilute 100uL 1-bomo-2-nitrobenzene to 100uL hexane.

Note: Stock standard solutions should be replaced after six months or sooner if comparison with laboratory fortified blanks, or QC samples indicate a problem.

6.4 Gas- Ultra-high purity liquid nitrogen

7.0 Definitions

7.1 Calibration Standard (ICV, CCV)- A solution of procedure analytes used to calibrate the mass spectrometer response.

7.2 Laboratory Control Sample (LCS)- A separate source standard containing known concentrations of analytes that is added prior to sample preparation and then analyzed by the laboratory to demonstrate that it can obtain acceptable identifications and measurements.

7.3 Surrogate Compound- A compound that is not expected to be found in the sample that is added to a sample aliquot before extraction and is measured with the same procedures used to measure sample components. The purpose of a surrogate compound is to monitor procedure performance with each sample.

7.4 Laboratory Reagent Blank- An aliquot of DI water or solid reference material that is treated as a sample. It is exposed to all glassware and apparatus, and all procedure solvents, reagents and surrogate compounds. The extract is concentrated to the final volume used for samples and is analyzed the same as a sample extract. The method blank is used to determine if method analytes are present in the lab environment, glassware or reagents.

7.5 Internal Standard -- A pure analyte(s) added to a solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same solution. The internal standard must be an analyte that is not a sample component.

8.0 Procedure

8.1 Gas Chromatograph Conditions

8.1.1 Injector Temp: 250°C

8.1.2 Detector Temp: 375°C

8.1.3 Carrier Gas: Nitrogen at 20 ml/minute

8.1.4 Temperature program: 100°C for 1 min., 12.5°C per minute to 300°C, hold 1 min. Total analysis time: 18 minutes.

8.2 External Standard Calibration Procedure

8.2.1 Prepare calibration standards as outlined below.

8.2.2 Herbicide Calibration Curve:

STD#	Herbicide Working Std	µL DCAA	Final Vol.	µL Internal Standard
1	50	10	10 mL	100
2	75	20	10 mL	100
3	100	30	10 mL	100
4	150	40	10 mL	100
5	200	50	10 mL	100

8.2.3 Calibration Verification Standard; see Section 6.3.2

Log the standard preparation into logbook. Be sure to include the date, recipe, lot numbers, initials and expiration date.

8.3 Initial Calibration

8.3.1 A five level calibration curve must be performed prior to the analysis of any samples. Properly identify each surrogate and compound peak.

Calibrate the instrument. The RSD must be ≤20% for each compound's curve.

8.3.3 Immediately following calibration, a calibration verification standard is analyzed to determine the accuracy of the calibration standards. All analyte recoveries should fall within +/- 15%. If these criteria are not met, fresh calibration standards should be made a new calibration performed, followed by the analysis of the calibration verification standard.

8.4 Initial Calibration Verification

8.4.1 If a calibration curve has already been established from a previous analysis day, the analyst may analyze an initial calibration verification in place of the calibration curve. The third or fourth level standard of each curve can serve this purpose. Recoveries must be within the range of +/- 15% to continue analysis. If the recoveries fail to meet these criteria, the standard may be re-analyzed once. If criteria again are not met, a new calibration must be performed.

8.5 Initial Calibration Blank

8.5.1 A solvent blank is analyzed following the initial calibration or the initial calibration verification to ensure no carry-over occurs which may cause false positive results.

8.6 Continuing Calibration Verification

8.6.1 Every ten samples a continuing calibration standard must be analyzed to monitor instrument performance. The third or fourth level standard of each curve can serve this purpose. The standard level used should be alternated every ten samples. Recoveries must be within the range of +/- 15% to continue analysis. If the recoveries fail to meet these criteria, the standard may be re-analyzed once. If the criteria are again not met, the instrument is considered out of calibration and no data prior to the failing CCV may not be reported.

8.7 Continuing Calibration Blank

8.7.1 A solvent blank is analyzed following the initial calibration or the initial calibration verification to ensure no carry-over occurs which may cause false positive results.

8.8 Analysis Sequence

Solvent Blank
Calibration or ICV
CVS (if new calibration)
Solvent Blank
Extraction Blank
LCS
MS
MSD
Samples 1-6
Cal Check
Solvent Blank
Samples 1-10
Cal Check
Solvent Blank

Note: If the response for any peak exceeds the working range of the system, the sample is diluted and reanalyzed.

9.0 Quality Control

9.1 Initial Demonstration of Capability

9.1.1 Prior to the analysis of any samples, the analyst must demonstrate they understand and can perform the method. Four fortified samples with each analyte of interest at a concentration around the midpoint of the calibration curve must be prepared using SOP 214.515.1 and analyzed.

9.1.2 The recovery of each analyte for all four samples must fall in the range of +/- 30%. For those compounds that meet the acceptance criteria, performance is considered acceptable and sample analysis may begin. For those compounds that fail these criteria, this procedure must be repeated using four fresh samples until satisfactory performance has been demonstrated.

9.2 Method Detection Limit Study

9.2.1 Analyze seven consecutive, prepared aliquots containing analyte concentrations 2 to 5x the expected MDL. Evaluate using procedure in 40CFR part 136 Appendix B.

9.3 Laboratory Reagent Blanks

9.3.1 Before processing any samples, the analyst must demonstrate that all glassware and reagent interferences are under control. Each time a set of samples is extracted or reagents are changed, a laboratory reagent blank (LRB) must be analyzed. If within the retention time window of any analyte of interest the LRB produces a peak that would prevent the determination of that analyte, determine the source of contamination and eliminate the interference before processing samples.

9.4 Assessing Surrogate Recovery

9.4.1 When surrogate recovery from a sample or method blank is <50% or >130%, check (1) calculations to locate possible errors, (2) fortifying solutions for degradation, (3) contamination or other obvious abnormalities, and (4) instrument performance. If the recovery is <50%, have the sample re-prepped. If the recovery is >130%, it must be narrated.

9.4.2 If sample extract re-analysis meets the surrogate recovery criterion, report only data for the reanalyzed extract. If sample extract re-analysis continues to fail the surrogate recovery criterion, report all data for that sample as suspect.

9.5 Matrix Spike and Matrix Spike Duplicate

9.5.1 Two fortified samples (labeled MS and MSD) are analyzed every batch of 20 samples. Thus 10% of all samples are represented by a fortified sample.

9.5.2 Calculate the percent recovery for each analyte by dividing the result by the true value. The recovery should fall within laboratory established control limits for MS/MSD.

9.5.3 If the recovery of any such analyte falls outside the 30-130% control limits and the LCS for that analyte is shown to be in control, the recovery problem encountered with the MS/MSD is judged to be matrix related, not system related. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.5.4 Calculate the relative percent difference (RPD) between the MS and MSD

recoveries. If the RPD is greater than 20% for waters or 30% for soils, evaluate the LCS/LCSD RPD to determine if sample homogeneity is the likely cause.

9.6 Laboratory Control Samples

9.6.1 The fortified blank samples labeled as LCS and LCSD are analyzed with each batch of 20 samples. Calculate the percent recovery for each analyte by dividing the result by the true value. The recovery must be between 40-130% recovery. If the recoveries fall outside these limits, the batch must be re-prepped. The only exception will be when recoveries are above 130% recovery but all sample results are non-detect. Results are then reported with narration.

9.6.2 Calculate the relative percent difference (RPD) between the LCS and LCSD recoveries. If the RPD is greater than 20% for waters or 30% for soils, narrate the nonconformance if both recoveries are within criteria.

9.7 Compound Identification

9.7.1 Identify a sample component by comparison of its retention time to the retention time of a reference chromatogram. If the retention time of an unknown compound corresponds, within limits, to the retention time of a standard compound, then identification is considered positive.

9.7.2 Retention time windows are established quarterly by recording the retention time of each compound in a standard three times over a 72-hour period. The retention time window is calculated as three times the standard deviation of these times. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

9.7.3 Identification requires expert judgment when sample components are not resolved chromatographically. Use of the secondary column data minimizes problems associated with identification such as co-eluting peaks.

10.0 Calculations

10.1 The turbochrom software is utilized to compare the response of the sample to that of the calibration designated. Further calculations may include multiplication of dilution factors and/or adjusting for sample weight.

11.0 Safety

11.1 The toxicity or carcinogenicity of each reagent used in this procedure 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 possible level by whatever means available. Refer to the Phoenix Environmental Laboratory Chemical Hygiene Plan. Also a reference file of material data handling sheets is available to all personnel in the general laboratory area.

12.0 Pollution Prevention

12.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

12.2 Reagents and standards should be purchased and/or prepared in volumes consistent with laboratory use to minimize the volume of disposal.

13.0 Waste Management

13.1 It is the laboratories responsibility to comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

14.0 Method Performance

14.1 This method was validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, initial and continuing calibration verifications, blank analysis, laboratory control samples and matrix spikes and duplicates.

14.2 See Section 9.0 Quality Control in this SOP for acceptable limits.

15.0 Corrective Action for Out-of-Control or Unacceptable Data

15.1 See Section 9.0 Quality Control in this SOP for corrective actions.

16.0 References

16.1 Method 8151A- "Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization", SW-846, Revision 1, December 1996.

Revision History Log for SOP #627.8151

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
3/15/13	7	Updated standard concentrations	Jeff Bucko	Kathy Cressia	3/15/13
10/22/14	8	Updated PCP std conc., slight changes to conditions	Brian Bilodeau	Keith Aloisa	10/22/14

Mercury Determination in Sorbent Tubes by Cold Vapor Atomic Absorption Technique (CVAA)

References: USEPA, "Method 7471B Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)," in Test Methods for Evaluating Solid Waste, SW846, Revision 2, January 1998

USEPA SOP "Analysis of Mercury in Air with a Modified NIOSH 6009 Method" , Rev 2.0, 5/13/1999

1. Scope and Application

Matrices: This cold-vapor atomic absorption method is applicable to the determination of total mercury (organic and inorganic) in sorbent tubes.

Definitions: Refer to Alpha Analytical Quality Manual.

This digestion and analytical procedure measures total mercury (organic & inorganic) in sorbent tubes. Mercury can accurately be determined in the range of 0.01 to 0.25 ug/ tube that do not require dilution.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the AA and in the interpretation of AA data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability, analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the Quality Assurance Officer and/or Laboratory Director on a case-by-case basis.

Parameter	CAS
Mercury	7439-97-6

2. Summary of Method

Air samples of elemental Hg are collected on Hopcalite sorbent material contained in glass tubes. Air is pumped through the sorbent. The sorbent material from the collection tube is quantitatively transferred to a digestion tube. The sample is digested by first adding 2.5 mL of concentrated nitric acid followed by 2.5 mL of concentrated hydrochloric acid. After digestion, the sample is diluted with deionized water. Once the samples have been digested, they are ready for analysis by the cold-vapor atomic absorption technique (CVAA).

The CVAA technique is based on the absorption of radiation at 253.7-nm by mercury vapor. The addition of the hydroxylamine-hydrochloride solution to the digestate, reduces the excess potassium permanganate without reducing the mercury and transforms the oxidized mercury to non-volatile $HgCL_2$. Mercury is then reduced with stannous chloride from $HgCL_2$ to elemental mercury, [Hg (0)]. The elemental mercury is aerated as mercury vapor from the digestate in a closed system where the vapor passes through a cell positioned in the light path of an atomic absorption spectrometer. Absorbance is measured as a function of mercury concentration based on the peak height measured.

2.1 Method Modifications from Reference

Method 7471B specifies use of 300 mL BOD bottles for sample and standard preparation and a final volume of approximately 100 mL. This method has been modified for use of 50 mL plastic digestion tubes. Reagent volumes have been reduced proportionately.

For convenience, samples are digested in 50 mL digestion tubes and diluted 1:2 at the instrument, where USEPA SOP 1827 states to dilute to final volume of 100 mL.

3. Reporting Limits

The mercury solid RL is 0.01 ug/tube.

4. Interferences

4.1 Potassium permanganate is added to eliminate potential interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from distilled water.

4.2 High concentrations of copper may cause interference; however copper concentrations as high as 10 mg/L have no effect on the recovery of spiked mercury samples.

4.3 Additional portions of potassium permanganate may need to be added until the purple color persists due to interference from chloride or organic matter. During the oxidation step, chlorides are converted to free chlorine which absorbs radiation at 253.7-nm. Care must be taken to ensure that free chlorine is absent before mercury is reduced to its elemental state and mercury vapor is swept into the cell. This may be accomplished by using additional portions of sodium chloride-hydroxylamine hydrochloride solution.

Note: Chloride interference is not as common in solid matrices as aqueous matrices.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. All digestion and analysis procedures must be conducted in a laboratory exhaust hood.

5.2 Care must be taken when handling all samples, digestates, and standards since they are preserved to a pH <2. In addition, the digestate solutions contain strong oxidizing reagents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

10-200 L of air collected through the tube.

6.2 Sample Preservation

The samples are stored at 25°C until digestion and analysis.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

All sorbent tube samples must be analyzed within 30 days from date of collection.

7. Equipment and Supplies

7.1 PSA 10.035 MILLENNIUM MERLIN MERCURY ANALYZER

7.1.1 Lamp: Mercury, low pressure

7.1.2 HP Laser Jet P2015dn or equivalent, compatible with AA and AF software

7.1.3 Millennium AAS Detector:

7.1.4 Pumps: Two variable speed and independently controlled peristaltic pumps to deliver reagent and sample solutions as well as remove waste.

7.1.5 Drying Tube: Gas liquid separator and dryer tube to remove mercury vapor from solution and prevent water vapor from entering the absorption cell.

7.2 Disposable Digestion tubes, 50mL

7.3 Air Displacement pipettes: Digital pipettes capable of delivering volumes ranging from 0.1 to 5000 µL with an assortment of high quality disposable pipette tips.

7.4 Analytical balance: Capable of accurate measurement to the nearest 0.0001 g

7.5 Top-loading balance: Capable of accurate measurement to the nearest 0.01 g

8. Reagents and Standards

ACS Trace Metal grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

Solutions below expire six months from preparation unless noted.

Stock standard solutions are stored in a cabinet, out of direct light

8.1 Deionized (DI) water: The Barnstead NANO-pure system provides Type I water used in the preparation of samples and standards.

8.2 Concentrated hydrochloric acid (HCl). Fisher trace metals grade or equivalent. *(Digestion reagent).* Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.3 Concentrated nitric acid (HNO_3). Fisher trace metals grade or equivalent. *(Digestion reagent).* Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.4 Stannous Chloride ($SnCl_2 \bullet 2H_2O$). Fisher T142-500, or equivalent. *(Analytical reagent)*

8.5 Stannous Chloride Reduction Solution (2%). Add 20 g of $SnCl_2 \bullet 2H_2O$ to 100mL of concentrated HCl and dilute to 1L with deionized water. Prepare fresh daily. *(Analytical reagent)*

8.6 HCl Rinse Solution (10%). Dilute 900mL of concentrated HCl to 9L with deionized water. *(Analytical reagent)*

8.7 1000mg/L Mercury Stock Standard from two different sources. One source is used to prepare the calibration curve (Ultra Scientific ICP-080) and the other is for verification of the calibration curve (Inorganic ventures CGHG1-1). Use the vendor's expiration date. *(Standards preparation)*

8.8 Mercury Working Standards. Three mercury working standards are prepared from successive dilutions of the mercury stock standard and are used to prepare the calibration curve. Acidity of the working standards must be maintained at 1% HCl acid. Add 1mL of concentrated HCl to a 100mL volumetric flask. A 1.0mg/L working standard is prepared by adding 0.1mL of the 1000mg/L Ultra Scientific stock standard to a final volume of 100mL. This standard is diluted 1:10 to make a 0.1mg/L working standard and 1:100 to make a 0.01 mg/L working standard. The 0.1 and 0.01mg/L working standard is diluted prior to preparation of the calibration standards. The 1.0mg/L working standard is prepared monthly. See Section 10.0, for specific details regarding working standard preparations. *(Standards preparation)*

8.9 1.0mg/L LCS and Matrix Spike Solution. Prepare from the 1000 mg/L stock standard (8.13) by adding a 0.1mL aliquot to a final volume of 100mL. This solution is stable for 1 month. *(Spiking Solution)*

8.10 Sorbent Matrix Blank Sample: Purchased from SKC, Catalog #226-17-1A sorbent tubes, used for all QC.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

9.1.1 A method blank must be prepared from the blank sorbent material and analyzed once per every 20 samples or per mercury digestion batch, whichever is more frequent.

9.1.2 Mercury concentrations must not be detectable in the method blank at values greater than the reporting limit.

9.1.3 Corrective Action: Digestion of the method blank and all associated samples must be performed until the method blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Section Head if the samples associated with the method blank are non-detect for mercury or if the concentration of mercury is greater than 10x the blank level in the samples. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified in the project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)

9.2.1 The LCS is digested along with the samples. A LCS must be digested using blank sorbent material and analyzed once per every 20 samples or per mercury digestion batch, whichever is more frequent.

9.2.2 The acceptable recovery QC limits are 90%-110%.

9.2.3 Corrective Action: An explanation of this out of control LCS recovery must be included in the project narrative to the client and the sample data reported noting the acceptable MS results as batch QC.

9.3 Initial Calibration Verification (ICV)

9.3.1 Initial Calibration Curve

9.3.1.1 The correlation coefficient (r) of the initial calibration curve must be ≥0.995. The initial calibration curve consists of five standards and a blank.

9.3.1.2 Corrective Action: If the correlation coefficient does not exceed 0.995, individual standards or the entire curve may be re-analyzed until the correlation coefficient is in control. If the correlation coefficient is still not in control, the initial calibration curve must be re-prepared and re-analyzed until the correlation coefficient is acceptable.

9.3.2 Initial Calibration Verification (ICV) Check Standard

9.3.2.1 The initial calibration verification check standard must be from a second source or lot number to verify the accuracy of the standard curve. The concentration of the ICV is at approximately the mid-level of the calibration curve.

9.3.2.2 The acceptable recovery QC limits for the ICV is 90%-110%.

9.3.2.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the ICV still exceeds the control limits, re-calibrate the instrument.

9.3.3 Initial Calibration Blank (ICB)

9.3.3.1 An ICB must be analyzed immediately following the ICV.

9.3.3.2 The ICB concentration must not be greater than the reporting limit.

9.3.3.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the ICB still exceeds the control limits, re-calibrate the instrument and re-analyze a fresh blank.

9.4 Continuing Calibration Verification (CCV)

9.4.1 Continuing Calibration Verification (CCV) Check Standard

9.4.1.1 A CCV must be analyzed at a minimum of every 10 samples and at the close of an analytical sequence. The concentration of the CCV is at approximately the mid-level of the calibration curve. This standard monitors instrument performance throughout the duration of the analytical run.

9.4.1.2 The acceptable recovery QC limits for the CCV is 90%-110%.

9.4.1.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CCV still exceeds the control limits, re-calibrate and re-analyze all samples since the last acceptable CCV.

9.4.2 Continuing Calibration Blank (CCB)

9.4.2.1 A CCB must be analyzed immediately after every CCV.

9.4.2.2 The CCB concentration must not be greater than the reporting limit.

9.4.2.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CCB still exceeds the control limits, re-calibrate and/or re-analyze a fresh blank. All samples associated with the out of control CCB must be re-analyzed (since the last acceptable CCB). Exceptions may be made with approval of the Section Head if the samples associated with the out of control CCB are non-detect for mercury or if sample concentrations for the affected metals are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high CCB and the client is notified in a project narrative associated with the sample results.

9.5 Laboratory Duplicate

9.5.1 Duplicate analyses (matrix duplicate) must be performed if provided by the client once per 20 samples (5% frequency).

9.5.2 Acceptable relative percent difference (RPD) for duplicate analysis is ≤ 20 % for solid matrices. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

The RPD limits are continuously monitored and documented in-house through control charts.

9.5.3 Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to client.

9.6 Method-specific Quality Control Samples

9.6.1 MDL check standard

9.6.1.1 MDL check standard is prepared by spiking blank sorbent material with mercury at the MDL.

9.6.1.2 The acceptance criteria is 80-120% of the true value.

9.6.1.3 Corrective Action: May repeat analysis once to see if an analytical error has occurred. Include a project narrative with the results to the client.

9.6.2 Lot Blank

One unopened sampling tube must be prepared and analyzed as a lot blank with each sample lot of twenty tubes or less per project. The lot blank tube is delivered to the laboratory with the samples, broken up without disturbing the contents, and then prepared using the procedure outlined in Section 10.

9.6.3 Trip Blank

One blank tube from the same lot is included as the trip blank. The tube is broken and carried to the site in the same type of container as the samples. It is delivered to the laboratory with the samples and is prepared using the procedure outlined in Section 10.

9.6.4 Field Blanks

One or more blank tubes from the same lot are included as field blanks. The tube is broken and carried to the sampling stations at the site but no air is sampled. Field blanks are delivered to the laboratory with the samples and are prepared using the procedure outlined in Section 10.

9.7 Method Sequence

- Initial calibration curve (STD0, STD1, etc., to STD5)
- ICV
- ICB
- MDL check standard
- Method Blank (The assigned LIMS batch name)
- LCS (The assigned LIMS batch name)

- Sample analysis (samples 1-5 including SD, BS and BSD samples)
- CCV
- CCB
- Sample analysis (6-15)
- CCV
- CCB

The calibration curve must meet the criteria for linearity (Correlation Coefficient 0.995 or better).

10. Procedure

10.1 Equipment Set-up

10.1.1 Digestion Procedure

10.1.1.1 Quantitatively transfer the sorbent material and the front glass wool plug from each sampler tube into a 50 mL digestion tube.

10.1.1.2 All QC and calibration standards are digested along with the samples.

10.1.1.3 Add 2.5mL of concentrated HNO_3 to all tubes, followed by 2.5mL of concentrated HCl.

10.1.1.4 Spike Laboratory Control Sample with 0.125mL of the Hg 1 mg/L spiking solution (see 8.9).

10.1.1.5 Allow samples to digest for 1 hour or until the sorbent material is dissolved. The solution will be dark and may contain undissolved material.

10.1.1.6 Dilute to 50 mL with DI water.

10.1.1.7 Dilute the samples 1:2 at the instrument.

10.2 Initial Calibration

A series of five calibration standards are prepared by pipetting suitable volumes of standard solution into 50mL centrifuge tubes. The preparation date of these standards, the initials of the analyst, the lot number of the source material, stock concentrations, volumes used, final volumes, final concentrations, and manufacturer, and expiration date must be recorded in the *Mercury CVAF, Mercury CVAA and Amalgam Working Standards Preparation Logbook*. The PSA MILLENIUM MERLIN is calibrated using a multi-point calibration curve consisting of a blank and six standards.

10.2.1 The working standards are prepared as follows:

10.2.1.1 Mercury Stock Solution (1000mg/L). See Section 8.13. Check expiration date to ensure the standard has not expired.

10.2.1.2 Mercury Working Standard (1.0mg/L). See Section 8.14. Check expiration date to ensure the standard has not expired.

10.2.1.3 Diluted Mercury Working Standard (0.1mg/L): Dilute 0.1mL of working standard (10.2.1.2) to 1.0mL. This solution must be prepared fresh daily.

10.2.1.4 Diluted Mercury Working Standard (0.01mg/L): Dilute 0.1 mL of working standard (10.2.1.3) to 1.0mL. This solution must be prepared fresh daily.

10.2.1.5 The calibration standards may be prepared in 50mL screw cap plastic digestion tubes. Each tube must contain approximately 25mL of deionized water, which is then spiked with the volume listed in the table below. Prepare a five-point mercury calibration curve daily as follows

Volume of the Working Standard (mL)	Concentration of Working Standard (mg/L)	Concentration of Calibration Standard (µg/L)
0	None	Blank
0.05	0.1	0.05
0.1	0.1	0.1
0.25	0.1	0.25
0.1	1	1
0.25	1	2.5

10.2.1.6 Digest all standards along with the samples as described in Section 10.1, Equipment Set-up. Calibration standards and all QC are diluted 1:2 at the instrument.

10.2.2 The PSA MILLENIUM MERLIN computer will calculate a linear regression, correlation coefficient ("r"), and slope of standard curve. The correlation coefficient must be greater than $\geq$ 0.995 for linearity. The linear regression is computed on a multipoint calibration.

10.3 Equipment Operation and Sample Processing

10.3.1 Before turning the instrument on, unplug and power off on rear panel. Connect the millennium AAS detector, plug back in and power on the back panel. The pump windings must be inspected for wear, rotated and/or changed. Secure the pump windings to the pump. Turn the gas on to 52 P.S.I. Turn on the mercury analyzer and the computer. Allow the instrument to warm up for 30 to 60 minutes.

10.3.2 Place the rinse line in the 10% HCl rinse solution. The $SnCl_2$ line from the instrument must be immersed into the 2% stannous chloride reduction solution.

10.3.3 All standard and sample information is entered into a sample information, or instrument sequence file. The following information is entered: sample ID, dilution and units. A hard copy of the sequence file is printed, given a page number, and becomes part of the permanent instrument run log. After one month, or more if suitable, of mercury analyses, the sequence printouts are bound and given an internal log identification number.

10.3.4 Place the calibration standards and samples onto the autosampler rack in the appropriate positions according to the sample information sequence file.

10.3.5 Begin the run sequence with the initial calibration curve and evaluate it for QC acceptance. Evaluate the ICV, ICB, Method Blank and LCS for QC acceptance prior to the analysis of the samples. The typical analytical sequence is listed in Section 9.8.

10.3.6 The instrument detection limits (IDLs) may be performed if required by the regulatory program or the client.

10.3.7 Dilute and reanalyze any samples that exceed the linear calibration range for mercury. Report the mercury result from the dilution analysis.

10.3.8 The *Mercury Data Review Checklist* must accompany all acceptable mercury results for primary and secondary review.

10.4 Continuing Calibration

The same time must be elapsed between CCVs and CCBs as is allowed between samples. Analyze the continuing calibration verification (CCV) and the continuing calibration blank (CCB) after each 10 samples and at the end of the analytical sequence.

10.5 Preventive Maintenance

Pump windings should be inspected for wear on a daily basis and replaced when they appear flattened (approximately once per month depending on use). The gas-liquid separator should be cleaned periodically when it appears coated with a yellow film. Add 30% KOH solution and let set for approximately 30 minutes. Rinse with DI water. The dryer tube should be replaced approximately once per year. (Part number H003S001 PS Analytical)

11. Data Evaluation, Calculations and Reporting

11.1 The mercury results are calculated by the following equation:

$$\text{Mercury result in ug/M}^3 = (\underline{A \times B} \times DF)/V$$

Where:

A = Digestion final volume in mL, typically 50 mL
B = Concentration of sample from instrument read-out in µg/L
DF = Dilution Factor
V = Volume in M^3

The client must provide the sampling volume.
If the sampling volume provided is in Liters, divide L by 1000 to convert to M^3.

11.2 All mercury results must be reported to three significant figures.

11.3 The primary analyst does the batching and data upload into the LIMS system.

11.4 A secondary review is performed on all.

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

Section 9, Quality Control, defines the corrective actions that must be taken in instances where QC outliers exist.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2124 Chemical Hygiene Plan

1732 DL/LOD/LOQ Generation SOP

1739 IDC/DOC Generation SOP

1797 Waste Management and Disposal SOP

16. Attachments

None.

Determination of PCB Homologs, 136/209 Individual Congeners, and Pesticides Confirmation by GC/MS - SIM

References: **Method 680,** "*Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography / Mass Spectrometry*", USEPA, Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio; November 1985

Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4, February 2007, Test Methods for Evaluating Solid Waste, SW-846,

Method 8082A Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Rev. 1, February 2007, Test Methods for Evaluating Solid Waste, SW-846

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-10 / TO-4A

Determination Of Pesticides And Polychlorinated Biphenyls In Ambient Air Using Low/High Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD), Center for Environmental Research Information, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268, January1999. EPA/625/R-96/010b.

NOAA Technical Memorandum NMFS-NWFSC-59, Extraction, Cleanup, and Gas Chromatography/Mass Spectrometry Analysis of Sediment and Tissue for Organic Contaminants, March 2004, U.S. of Department of Commerce, National Oceanic and Atmospheric Administration

1. Scope and Application

Matrices: This method is applicable to the quantification of Polychlorinated Biphenyls (PCBs) as Homologs, Aroclors and/or individual Congeners as well as Pesticides in water, soil, sediment, tissue (either animal or vegetable) and in ambient air using PUF sampling followed by Gas Chromatography/Mass Spectrometry with Selected Ion Monitoring (GC/MS-SIM).

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the analysis and quantification of sample extracts for PCBs as single Congeners, Homologs (isomer groups by the level of chlorination) and/or commercial Aroclors as well as Pesticides by Gas Chromatography/Mass Spectrometry with Selected Ion Monitoring (GC/MS-SIM). Additionally, this method can provide a "total" PCB result for a given sample extract. Target analytes include selected PCB congeners from BZ1 to BZ209, the Homolog groups, PCB Aroclors and Pesticides listed below. Individual congeners, homolog groups and pesticides are determined and measured in the concentration range of 0.5 to 500 parts per trillion (ng/L) for water samples, 0.033 to 200 parts per billion (ug/Kg) for soil/sediment and tissue samples, and 5-5000 ng/puf for PUF cartridges. These ranges are determined based on the lowest and highest levels of the calibration curve, extracted amount and final volume therefore detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed. Detection limits for Homolog groups are equal to the lowest detection limit of the individual congeners detected within that group. In general, analytes detected over these concentration ranges will be diluted and re-analyzed for accurate quantitation.

The following extraction and cleanup methods may apply, prior to sample analysis:

- *Extraction of Water Samples by Separatory Funnel-Method 3510C* (SOP 2165),
- *Tissue Preparation and Homogenization* (SOP 2166),
- *Shaker Table Extraction* (SOP 2261),
- *Sulfur Cleanup with Copper-Method 3660B* (SOP 2168),
- *Gel Permeation Chromatography-GPC (SOP 2167)*
- *Sulfuric Acid Cleanup-Method 3665A* (SOP 2169),
- *Silica Gel Cleanup* (SOP 2170),
- *Microscale Solvent Extraction – Method 3570* (SOP 2172)
- *Soxhlet Extraction – Method 3540C (SOP 2173),*
- *Soxhlet Extraction of PUF Cartridges (SOP 2174)*

The existence of 209 possible PCB congeners makes it impractical to list each potential method analyte and Chemical Abstract Service (CAS) number. Because in some cases, depending upon client request, PCBs are identified and measured as isomer groups, the non-specific CAS number for each level of chlorination is used to describe the method analytes. See below for this listing.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS and in the interpretation of GC/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability, analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the Quality Assurance Officer and/or Laboratory Director on a case-by-case basis.

PCB Homolog Group	Formula	CAS #
Monochlorobiphenyl	$C_{12}H_9Cl$	27323-18-8
Dichlorobiphenyl	$C_{12}H_8Cl_2$	25512-42-9
Trichlorobiphenyl	$C_{12}H_7Cl_3$	25323-68-6
Tetrachlorobiphenyl	$C_{12}H_6Cl_4$	26914-33-0
Pentachlorobiphenyl	$C_{12}H_5Cl_5$	25429-29-2
Hexachlorobiphenyl	$C_{12}H_4Cl_6$	26601-64-9
Heptachlorobiphenyl	$C_{12}H_3Cl_7$	28655-71-2
Octachlorobiphenyl	$C_{12}H_2Cl_8$	31472-83-0
Nonachlorobiphenyl	$C_{12}H_1Cl_9$	53742-07-7
Decachlorobiphenyl	$C_{12}Cl_{10}$	2051-24-3

Pesticides	Formula	CAS #
4,4'-DDD	$C_{14}H_{10}Cl_4$	72-54-8

4,4'-DDE	$C_{14}H_8Cl_4$	72-55-9
4,4'-DDT	$C_{14}H_9Cl_5$	50-29-3
Aldrin	$C_{12}H_8Cl_6$	309-00-2
Alpha-BHC	$C_6H_6Cl_6$	319-84-6
Alpha-Chlordane	$C_{10}H_6Cl_8$	5103-71-9
Beta-BHC	$C_6H_6Cl_6$	319-85-7
Delta-BHC	$C_6H_6Cl_6$	319-86-8
Dieldrin	$C_{12}H_8Cl_6O$	60-57-1
Endosulfan I	$C_9H_6Cl_6O_3S$	959-98-8
Endosulfan II	$C_8H_6Cl_6O_3S$	33213-65-9
Endosulfan Sulfate	$C_9H_4Cl_6O_4S$	1031-07-8
Endrin	$C_{12}H_8Cl_6O$	72-20-8
Endrin Aldehyde	$C_{12}H_8Cl_6O$	7421-93-4
Endrin Ketone	$C_{12}H_8Cl_6O$	53494-70-5
Gamma-BHC (Lindane)	$C_6H_6Cl_6$	58-89-9
Gamma Chlordane	$C_{10}H_6Cl_8$	5103-74-2
Heptachlor	$C_{10}H_5Cl_7$	76-44-8
Heptachlor Epoxide	$C_{10}H_5Cl_7O$	1024-57-3
Methoxychlor	$C_{16}H_{15}Cl_3O_2$	72-43-5

2. Summary of Method

An aliquot of a well mixed, homogeneous aqueous, solid, or tissue sample is accurately measured or weighed for sample preparation. Generally, 1L of water sample, 1-10g of tissue sample, 5-30g of sediment/soil sample for *Microscale Solvent Extraction - 3570 (*SOP 2172*) and 15-30g* of sediment/soil sample *for Soxhlet Extraction (SOP 2173)*. The PUF cartridge is extracted via Soxhlet, with the appropriate solvent. Water, soil/sediment, and tissue samples as well as PUF cartridges are spiked with surrogate compounds and extracted using methylene chloride,methylene chloride/acetone mixture, or hexane/ether mixture. The extract is dried and exchanged to hexane during sample concentration to a 1-10mL final volume. If necessary, the sample may be copper cleaned to remove sulfur, and/or GPC, silica or acid cleaned to lessen sample matrix interferences, prior to sample analysis.

After cleanup, the extracts are spiked with internal standards, and analyzed by GC/MS-SIM. Analytes are introduced into the GC/MS by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC equipped with a narrow-bore capillary column. The GC column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer operating in the selective ion mode (SIM). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of the calibration standards. Concentrations are determined using mean relative response factors from a multi-level calibration curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards. Multi-component analytes (PCB Homologs) are assigned the response factor of a representative PCB congener from that chlorination group. For PCB Aroclors single point calibration factors are used.

2.1 Method Modifications from Reference

This method exhibits some modification from the reference methods.

SIM data acquisition parameters, GC separation/operating conditions and MS sensitivity/calibration ratios for ions, and column type differ from the one described in Method 680 due to changes in technology since 1985.

Different compounds are utilized as Internal Standards and Surrogates then those specified in Method 680.

Different Calibration Congeners are used for Homologs group than the ones specified in Method 680.

Corrections are not made to any data for Homolog groups Cl2 – Cl8 for interferences resulting from M+35 or M+70 ions.

Acceptance criteria for ICAL, ICV, CCV, Surrogates and Internal Standards have been adopted from the guidance in Method 8270D,

Different DFTPP criteria then specified in method 8270D. Maximum Sensitivity Criteria are used.

Method Modifications from NOAA Technical Memorandum:

- Different extraction method then specified, MSE versus ASE
- Different requirements for batch quality control samples: method blank and LCS/LCSD versus method blank and SRM
- Different cleanup techniques: silica columns only versus silica/alumina columns and size-exclusion high-performance liquid chromatography
- Different GC settings: PTV/splitless injection versus COC (cool on-column) injection; oven temperature program differs, no guard column
- Different MS settings: electron ionization versus chemical ionization
- Different Calibration model: Average Response Factor obtain from Multi Level Calibration versus Point-to-Point Calibration
- Different Quality Controls and Measurements

3. Reporting Limits

Analytes are determined and measured in the concentration range of 0.5 to 500 parts per trillion (ng/L) for water samples, 0.033 to 200 parts per billion (ug/Kg) for soil/sediment and tissue samples, and 5-5000 ng/puf for PUF cartridges. The detection limit for Homolog groups is equal to the lowest detection limit of the individual congener detected within that group. Detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed.

4. Interferences

4.1 Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause interferences that lead to discrete artifacts and/or elevated baselines in the ion current profiles. Demonstrate that all of these materials are free from interferences under the conditions of the preparation and analysis by extracting and analyzing a laboratory method blank with each batch of up to 20 samples.

4.2 Contaminants co-extracted from the sample may cause matrix interferences. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature of the environment being investigated. An interference, which is unique to SIM techniques, can arise from the presence of co-eluting compounds, which contain the same quantification mass ion, or the same number of chlorine atoms. This event results in a positive interference to the reported value for the compound of interest. This interference is controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantification ion and the

confirmation ion are not within the specified limits, then interferences may be present. Quantification and confirmation ion criteria can be found in Table II.

4.3 With the isomer, or Homolog group quantification approach, co-eluting PCBs that contain the same number of chlorines, are identified and measured together. Therefore, co-eluting PCBs are only a problem if they contain a *different* number of chlorine atoms.

4.4 An interference can arise from the presence of co-eluting congeners containing one or two additional chlorines, which will contribute to the quant ion due to the fragmentation that occurs during the analysis. This event results in a positive interference to the reported value for the compound of interest. PCBs listed below are co-eluting with other congeners containing two additional chlorines:
- when analyzed on BNA5 for 136 Congeners – BZ#77, BZ#126, BZ#157
- when analyzed on BNA2 for 209 Congeners – BZ#37, BZ#60, BZ#78, BZ#81, BZ#107/123, BZ#105, BZ#127, BZ#126, BZ#167,

The interference from another PCB containing one additional chlorine is small and can be neglected except when measuring the area of a small amount of the congener co-eluting with a large amount of another congener containing one more chlorine.

4.5 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

***Aqueous* samples:** Collect in 1L or 2L amber glass bottles. The minimum amount of sample needed to reach the reporting limits in Section 3.0 for this method for aqueous samples is 1L. Additional sample is needed (approximately 3X the minimum amount) if MS/MSD analyses are to be performed.

***Soil/sediment* samples:** Collect in glass soil jars. The minimum amount of sample needed to reach the reporting limits in Section 3.0 for this method for solid and tissue matrices is 5g or 30g. Additional sample is needed (approximately 3X the minimum amount) if MS/MSD analyses are to be performed.

***Air Samples*:** Collected with appropriate air sampling techniques described in the Reference Method for collecting PUF cartridge air samples.

6.2 Sample Preservation

***Aqueous* samples:** Store without preservative at 4°C.

***Soil/sediment* samples:** Stored at 4°C, or if desired, frozen.

Air Samples: Stored at 4°C without preservation.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

The hold time for this method is 7 days for the extraction of aqueous samples and 14 days for the extraction of solid and tissue samples. If sediment or tissue samples are frozen, this suspends the holding time until removal from the freezer. Air PUF cartridges must be extracted within 7 days of collection.

All extracts must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph: The instrumentation includes a temperature-programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for splitless injection onto a capillary column. The injection port includes a silanized glass liner containing a plug of silanized glass wool to reduce high-molecular-weight mass discrimination. The model is HP6890 or equivalent. The injection port will require maintenance on an as needed basis if degradation or contamination is apparent.

7.2 Large volume injector, PTV - Gerstel, or equivalent: Temperature and flow programmable and capable of injecting 1 to 50 uL of standards and sample extracts onto the GC column in a split or splitless mode.

7.3 Column: For 209 Congener and Homolog Analysis - Restek 60-m x 0.18 mm ID, 0.18 um film thickness, fused-silica capillary column with Crossbond phase, or equivalent.

For 136 Congener, Homolog, Pesticide Analysis – Restek 60-m x 0.25 mm ID0.25 um film thickness, fused-silica capillary column with Crossbond phase, or equivalent.

7.4 Mass Spectrometer: The mass spectrometer must operate at 70ev (nominal) electron energy in the electron impact ionization mode and be tuned to optimize the sensitivity of the instrument to the maximum in the mass range being monitored (45 - 525 amu). The GC capillary column is fed directly into the ion source of the mass spectrometer. The model is HP5973, or equivalent. The source will require cleaning and/or filament replacement on an as needed basis. Please refer to the instrument hardware manual, located in the laboratory, for detailed procedures.

7.5 Auto sampler: Adapted onto the Gas Chromatograph. The model is HP 6890 series autosampler with a GC autosampler controller, or equivalent.

7.6 Computer: With Windows XP operating software utilizing HP Enviroquant G1701DA Version D.01.02 software; Audit Trail: audit.txt function is used for audit trail purposes.

7.7 Helium: Ultra high purity grade (99.9999% pure) or hydrogen of equivalent purity.

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specification of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. See SOP Reagent, Solvent, and Standard Control (SOP 1816) for additional details regarding solvent purity. All solvent expirations determined as indicated by manufacturer guidelines

Analytical Standards are stored according to manufacturer's recommended procedure. Stock standards, and calibration curve standards are stored in either 10mL or 40mL glass vials and kept in a standards freezer at -10^0 — -20^0C in the GC Instrumentation Lab. Primary standards are discarded as indicated by the vendor expiration. Stock standards are given one year expiration from the preparation date or the expiration of the primary vendor solution, whichever occurs first. Working standards are given six month expiration from the preparation date or the expiration of the primary solution whichever occurs first. If breakdown of a solution is observed the solution will be discarded. All analytical standards are prepared in Hexane. All extraction standards (surrogates, laboratory control spikes and matrix spikes) are prepared in acetone or hexane for Soxhlet extraction - *Method 3540C*. All spiking solutions must be assayed for use by analysis before release to the preparation lab If there is a significant amount of spiking solution remaining after the six-month period, the lab will re-QC the solution. If the QC of the solution still passes the lab will extend the expiration date by another month.

8.1 Methylene Chloride: ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (SOP 1816) for additional details regarding solvent purity.

8.2 Acetone: ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (SOP 1816) for additional details regarding solvent purity.

8.3 Hexane: ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (SOP 1816) for additional details regarding solvent purity.

8.4 Methanol: Purge and Trap grade, see SOP *Reagent, Solvent, and Standard Control* (SOP 1816) for additional details regarding solvent purity.

8.5 Ether, see SOP *Reagent, Solvent, and Standard Control* (SOP 1816) for additional details regarding solvent purity.

8.6 DFTPP (decafluorotriphenylphosphene) tuning solution: Prepare by diluting 0.025mLs of a 2000 ug/mL standard into 10mL of Methylene Chloride for a 5 ug/mL tuning standard. Working solution is further diluted 1:10 prior to analysis.

8.7 Individual Analytes (BZ-1 to BZ-209) and pesticides: Obtained from AccuStandard or equivalent at a concentration of 100 ug/mL.

8.8 209 Congeners Custom Calibration Set: Obtained from Accustandard as 9 separate mixes at the concentration of 10 ug/mL each. The solution consists of 209 Congeners. Prepare

the Stock Solution by diluting the calibration mixes to a stock concentration of 1000 ng/mL. See Section 8.16 for calibration preparation information.

8.9 Custom 136 PCB Congeners Set/ Homolog Custom Mix, Retention Time Window / Calibration Standard: Obtained from Accustandard as 7 separate mixes at the concentration of 4 ug/mL each. The solution consists of at least one representative PCB congener used for quantitation from each Homolog group and the first and last eluting congener of each Homolog group used to identify the start and stop time of each SIM window. Prepare the Stock Solution by diluting the calibration mixes and the Carbon-labeled Surrogate Stock solution to a 136 Cong/Surr Stock concentration of 400 ng/mL. See Section 8.16 for calibration preparation information.

Pesticide Calibration Standard: Commercially obtained primary solutions are used to volumetrically create stock solutions. All stock solutions are made by adding the appropriate amount of primary standard to a 25 mL volumetric flask to obtain the concentration of 5000 ug/L. The stock solutions are then used in various combinations and serial dilutions to make calibration curve standards (see Table in section 8.16). Calibration standards are always prepared in hexane. The basic calibration curve is made from MCP Pest Stock, Additional Pest Stock and DBOB/BZ198 Surrogate Stock. MCP Pest Stock is derived from primary *Custom Pesticide Standard (PN # S-9553-SS)* commercially obtained from Accustandard, Additional Pest Stock is made from primary *Custom Pesticide Standard (PN#S-14378-R1)* commercially obtained from Accustandard, and DBOB/BZ198 Surrogate Stock is prepared from primary surrogate solutions commercially obtained from Ultra: *DBOB* – at a concentration of 5000 ug/mL *(catalog # PPS-172) and BZ#198* – at a concentration of 100 ug/mL *(catalog # RPC-075S)*.

8.10 Surrogates: 4,4'-Dibromooctafluorobiphenyl (DBOB) and BZ 198, obtained from Ultra Scientific or equivalent.

8.10.1 DBOB/BZ198 stock solution: Add 25 uL of the 5000 ug/mL DBOB primary solution and 1250 uL of the 100 ug/mL BZ 198 primary solution to 25 mL volumetric flask and dilute with hexane for 5000 ug/L stock solution.

8.10.2 Pest/Cong Surrogate spiking solution: add 4 mL of DBOB/BZ198 stock solution to 200 mL volumetric flask and dilute with acetone, for a 100 ug/L final concentration. The solution must be assayed for use by analysis before release to the preparation lab. All compounds must be within 20% of their true value. 1mL is spiked into each QC and field sample. The concentrations may be adjusted to meet project specific needs.

8.11 Carbon-labeled Surrogates: BZ 19 and BZ 202, obtained from Cambridge Isotope or equivalent, at a concentration of 40 ug/mL.

8.11.1 Carbon-labeled Surrogates Stock Solution: Prepare a separate solution by taking 1.25mL of each of the 40 ug/mL surrogate solutions, and add to 10mL of hexane for a stock concentration of 5 ug/mL or 5000 ng/mL.

8.11.2 Homolog Surrogate spiking solution: Add 250 uL of each surrogate to 100 mL volumetric flask and dilute with acetone, for a 100 ug/L final concentration. All compounds must be within 20% of their true value. 1mL is spiked into each QC and field sample. The concentrations and the spiking amount may be adjusted to meet project specific needs.

8.11.3 High Homolog Surrogate spiking solution: Add 1.25 mL of each surrogate to 50 mL volumetric flask and dilute with hexane, for a 1000 ug/L final

concentration. The solution must be assayed for use by analysis before release to the preparation lab. All compounds must be within 20% of their true value. 1mL is spiked into each QC and field sample. The concentrations and the spiking amount may be adjusted to meet project specific needs.

NOTE: PCB High Homolog Surrogate spiking solution is prepared in hexane and used for Soxhlet extraction only.

8.12 Carbon-labeled Internal Standards (IS): BZ 15 and BZ 180, obtained from Cambridge Isotope or equivalent, at a concentration of 40 ug/mL. Add 2.5mL of each internal standard to 10mL volumetric flask and dilute with hexane, for a final concentration of 10 ug/mL. 20uL is spiked into each standard, QC sample, and field sample. The resulting on-column concentration is 200 ug/L in a 1mL sample aliquot. The concentrations may be adjusted to meet project specific needs.

8.13 Laboratory Control Sample, Matrix Spike, and Matrix Spike Duplicate (LCS/MS/MSD) Preparation:

8.13.1 Homolog LCS spiking solution: Prepared using Accustandard C-CSQ-SET. Nine ampules containing all 209 congeners at 10ug/mL. Add 1.0 mL of each mix to 100mL volumetric flask and dilute with *acetone*, for a 100 ug/L final concentration. The solution must be assayed for use by analysis before release to the preparation lab. Recoveries of 90% of the analytes must be +/- 20% of the true values.

8.13.2 High Homolog LCS spiking solution: Prepared using Accustandard C-CSQ-SET. Nine ampules containing all 209 congeners at 10ug/mL. Add 1.0 mL of each mix to 25 mL volumetric flask and dilute with hexane, for a 400 ug/L final concentration. The solution must be assayed for use by analysis before release to the preparation lab. Recoveries of 90% of the analytes must be +/- 20% of the true values.

NOTE: HIGH Homolog LCS/MS/MSD spiking solution is prepared in hexane and used for Soxhlet extraction only

8.13.3 PESTICIDES LCS and Matrix spiking solution: Two Pest LCS Spiking solutions are generally used in this method. Alternatively, solutions with project specific targets may be used. The LCS spiking solutions are stored in the same manner as surrogate solutions. The Pest LCS solutions are made from a combination of *Custom Standard – Pesticide Mix (Quote#110507-082) and Custom Standard – Additional Pesticide Mix (Quote#030207-236)* both commercially obtained from Ultra at 100ug/mL. Two spiking solutions are made from this primary solution. The first spiking solution is labeled "Pest LCS" spike and is prepared by diluting the appropriate aliquot of each of the two primary solutions to obtain a spike solution concentration of 1000ug/L. The second spike solution is the "Pest ULOW LCS" spike and consists of the same two primary mixes diluted appropriately to create a spike solution at a concentration of 100ug/L. All solutions are made volumetrically in Acetone at desired final volume. Generally 100ml of spiking solution standard is made but this amount can vary depending on use, need and preference. Generally 1 mL of LCS spiking solution

is added into each laboratory control spike, matrix spike, and spike duplicate QC samples.

8.13.4 **136 Congener LCS and Matrix spiking solution:** Prepared using Accustandard S-12331-TP-1ML-SET. Seven ampules containing all 136 calibrated congeners at 4.0ug/mL. Add 0.625 mL of each mix to 100mL volumetric flask and dilute with *acetone,* for a 100 ug/L final concentration. The solution must be assayed for use by analysis before release to the preparation lab. Recoveries of 90% of the analytes must be +/- 20% of the true values.

8.14 Independent Calibration Verification (ICV) standard:

8.14.1 **209 Congener ICV Standard:** For the 209 Congener Calibration Curve, the level 5 of the 136 Congeners curve serves as ICV (Accustandard or equivalent)

8.14.2 **136 Congener ICV Standard:** For 136 Congeners Calibration Curve, a different lot of the same solution used to prepare the ICAL standards serves as the ICV, S-12331-TP-1ML-SET.

8.14.3 **Pesticide ICV Standard:** The ICV can be made two ways as a combination of Custom Standard – Pesticide Mix (Quote#041310- 631) and Custom Standard – Additional Pesticide Mix (Quote#041310-632) both commercially obtained from Ultra at a concentration of 100ug/ml or use Restek Pest Mix 8081AB#1 cat#32291 at concentration 200ug/ml and Hexachlorobenzene and Additional Pesticide Mix (Quote#041310-632 from Ultra at a concentration of 100ug/ml. Also cis Nonachlor and Chlorpyriphos obtained from Ultra at concentration 100ug/ml are added to both mixtures to obtain all targets compounds. The ICV is prepared by diluting the appropriate aliquot of each of the two primary solutions to obtain a spike solution at the concentration of 50ug/L. This standard must be spiked with 20uL of the internal standard in 10.7, above. Alternatively LCS solution can be used as an independent check solution by diluting the LCS appropriately to obtain a solution at a concentration of 50ug/L

8.15 SRM 1944/1941b – New York/New Jersey Waterway Sediment, and SRM 1974b – Organics in Mussel Tissue: From National Institute of Standards & Technology (NIST). Please refer to the individual certifications for the assigned true values. These SRMs may be extracted and analyzed with sample batches as part of the overall QC evaluation, if requested by the client. Other certified SRMs may be used on a project specific basis.

8.16 Aroclors Solution: Obtained from Ultra at a concentration of 100 ug/mL for one-point calibration.

8.17 Calibration Preparation Information

Homolog Calibration Congener	Isomer Group	Stock Concentration ng/mL 136Congeners/209Congeners
BZ 1 (1st Mono)	Cl1	400 1000 ng/mL
BZ 8, BZ 5/8	Cl2	400 1000 ng/mL
BZ 29	Cl3	400 1000 ng/mL
BZ 50	Cl4	400 1000 ng/mL
BZ 87, BZ 87/111	Cl5	400 1000 ng/mL
BZ 154	Cl6	400 1000 ng/mL
BZ 188 (1st Hepta)	Cl7	400 1000 ng/mL
BZ 200, BZ 200/204	Cl8	400 1000 ng/mL
BZ 206 (last Nona)	Cl9	400 1000 ng/mL
BZ 209 (Deca)	Cl10	400 1000 ng/mL
Add'l Ret. Window PCB Congeners	**Isomer Group**	**Stock Conc. ng/mL 136Congeners/209Congeners**
BZ 3 (last Mono)	Cl1	400 1000 ng/mL
BZ 10 (1st Di)	Cl2	400 1000 ng/mL
BZ 15 (last Di)	Cl2	400 1000 ng/mL
BZ 19 (1st Tri)	Cl3	400 1000 ng/mL
BZ 37 (last Tri)	Cl3	400 1000 ng/mL
BZ 54 (1st Tetra)	Cl4	400 1000 ng/mL
BZ 77 (last Tetra)	Cl4	400 1000 ng/mL
BZ 104 (1st Penta)	Cl5	400 1000 ng/mL
BZ 126 (last Penta)	Cl5	400 1000 ng/mL
BZ 155 (1st Hexa)	Cl6	400 1000 ng/mL
BZ 169 (last Hexa)	Cl6	400 1000 ng/mL
BZ 189 (last Hepta)	Cl7	400 1000 ng/mL
BZ 202 (1st Octa)	Cl8	400 1000 ng/mL
BZ 205 (last Octa)	Cl8	400 1000 ng/mL
BZ 208 (1st Nona)	Cl9	400 1000 ng/mL
Surrogates		
DBOB	N/A	5000 ng/mL
BZ 198	Cl8	5000 ng/mL
Carbon-labeled Surrogates		
BZ 19	Cl3	5000 ng/mL
BZ 202	Cl8	5000 ng/mL

Note: Any of the above congeners may be reported as individual congeners, as well as within a Homolog group, as this method is not limited to this congener list. Additional congeners may be analyzed via this method by utilizing the MDL and PQL from a congener of the same Homolog class that has been previously established. Parentheses indicate the first and last eluting PCB congener in each chlorination level, used to establish the selective ion monitoring (SIM) retention time windows.

Suggested Curve Preparation for Individual Components (Minimum 5 Levels) for 136 Congeners

Calibration Level	136 Cong. /Surr Stock 400ng/mL	Calibration L6 (200ug/L)	Final Volume
L1 – 0.5 ug/L	-	0.025mL	10 mL
L2 – 1.0 ug/L	-	0.05mL	10 mL
L3 – 10 ug/L	-	0.5mL	10 mL
L4 – 20 ug/L	-	1mL	10 mL
L5 – 50 ug/L	1.25mL	-	10 mL
L6 – 200ug/L	5mL	-	10 mL
L7 – 400 ug/L	-	-	10 mL

Suggested Curve Preparation for Individual Components (Minimum 5 Levels) for 209 Congeners

Calibration Level	Surr. Stock 5000ng/mL	209 Cong. Stock 1000ng/mL	Calibration L6 (200ug/L)	Final Volume
L1 – 0.5 ug/L	-	-	0.025mL	10 mL
L2 – 1.0 ug/L	-	-	0.05mL	10 mL
L3 – 10 ug/L	-	-	0.5mL	10 mL
L4 – 20 ug/L	-	-	1mL	10 mL
L5 – 50 ug/L	0.1mL	0.5mL	-	10 mL
L6 – 200ug/L	0.4mL	2mL	-	10 mL
L7 – 500 ug/L	1ml	5ml	-	10 mL

Pesticides Level Curve Preparation for Individual Components

Calibration Level	Volume of Pesticide/Surrogate Stocks added*	Aliquot of High Calibration level** added	Volume of Hexane added
Level 1 – 0.5 ug/L	N/A	0.025 ml	10 mL final volume
Level 2 – 1.0 ug/L	N/A	0.05 ml	10 mL final volume
Level 3 – 5.0 ug/L	N/A	0.25 ml	10 mL final volume
Level 4 – 10 ug/L	N/A	0.5 ml	10 mL final volume
Level 5 – 20 ug/L	N/A	1.0 ml	10 mL final volume
Level 6 – 50 ug/L	0.25 mL	N/A	25 mL final volume
Level 7 – 100ug/L	0.2 mL	N/A	10 mL final volume
Level 8 – 150ug/L	0.3 mL	N/A	10 mL final volume
Level 9 – 200ug/L	0.4 mL	N/A	10 mL final volume

Up to 10 levels may be analyzed. Standards chosen should bracket the linear range of the instrument. A minimum of a 5-level curve must be analyzed, but 6-levels, or more, may be analyzed and evaluated depending upon client specific project detection limits. This is an example of possible curve levels, the curve used may vary based on project and instrument performance.
* Each calibration level is a combination of three stock solutions prepared at the concentration of 5000ug/L added at the indicated amount.
**High calibration level is 200ug/L.

Note: 20 uL of the 10 ug/mL chosen Internal Standard mix is added to each calibration level for a concentration of 200 ng/mL. *A minimum of a 5-level curve must be analyzed, but 6-levels, or more, may be analyzed and evaluated depending upon client specific project detection limits.*

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Quality Control (QC) samples are necessary to monitor both the sample extraction and instrument analysis procedures. The Quality Control samples described below are considered the method defaults, and are the minimum requirements, except where noted. Client and Project specific Data Quality Objectives (DQOs) supersede the requirements in this section where applicable. Client or Project specified DQOs shall be included, or referenced, in the final report to the client.

9.1 Blank(s)

A method blank must be extracted (spiked with surrogates and internal standards) and analyzed once per every 20 samples or per extraction batch, whichever is more frequent.

An *acceptable* method blank should not contain any individual compound at the concentration of reporting limit, or above. All efforts must be made to identify and eliminate the source of contamination. The presence of analytes at concentrations at or above the reporting limit will warrant application of a "B" qualifier to that target compound(s) on all associated report forms, and perhaps re-extraction of all associated samples. The results are qualified with a "B" for any associated sample concentrations that are less then 10x the blank concentration for the

analyte. Surrogate and internal standard recoveries must meet the QC limits for the method blank, see Sections 9.7.1 and 9.7.2. Re-extraction *corrective action* that would exceed the sample holding time criteria should be discussed with the client, Laboratory Director, QA Manager, and/or Section Supervisor prior to implementation. Exceptions may be made with approval of the Section Supervisor if the samples associated with an out of control method blank are non-detect for the affected compound(s) or if the concentrations of the affected compound(s) are greater than 10x the blank level in the samples. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified, via the project narrative, of any method blank non-compliance associated with the sample results.

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

The laboratory control sample/laboratory control sample duplicate (LCS/LCSD) contains at least 10 PCB congeners that represent each Homolog group. If only PCB Congeners are being evaluated, and if the list includes more than 20 congeners, at least 16 individual congeners will be spiked. If pesticides are being analyzed, the analyte list targeted in most cases will be spiked into the LCS/LCSD. The LCS/LCSD is extracted along with the samples. An LCS/LCSD pair must be extracted and analyzed once per every 20 samples or per extraction batch, whichever is more frequent. The number of PCB congeners in this sample may vary with client specific requests.

The acceptable recovery QC limits are found in Section 12 for an aqueous, solid, tissue and PUF LCS/LCSD.

Corrective Action: Repeat analysis or check to see if an analytical error has occurred. If the LCS recovery is still out of control, re-extract and re-analyze the LCS/LCSD and all associated samples. Samples cannot be analyzed until an acceptable LCS/LCSD is obtained. Exceptions may be made with approval of the Section Supervisor if the samples associated with the out of control LCS/LCSD are also associated with a matrix spike and matrix spike duplicate that is in control which demonstrates an isolated problem pertaining to the LCS and/or LCSD only. An explanation of this out of control LCS and/or LCSD recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS/MSD results as batch QC.

9.3 Initial Calibration Verification (ICV)

Refer to section 10.2.4

9.4 Continuing Calibration Verification (CCV)

Refer to section 10.4

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

Matrix spike and matrix spike duplicate analyses are performed at the client's request.
The acceptable recovery and RPD QC limits are found in Section 12 for an aqueous, solid, tissue, and PUF MS/MSDs.

Corrective Action: Repeat analysis or check to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the control limits and the associated LCS/LCSD is within control, include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the affected results as evidenced by the matrix spike and matrix spike duplicate exceedance.

9.6 Laboratory Duplicate

Laboratory matrix or sample duplicates are analyzed if requested by the client. The QC limit is 30% RPD for target compounds found above 5 times the reporting limit.

Corrective Action: If the %RPD exceeds the 30% control limit and the associated MS/MSD %RPD is within 30%, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results isolated to this sample, as evidenced by the matrix duplicate exceedance and the MS/MSD acceptance. If both the sample/duplicate and the MS/MSD exceed the control limits, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results as evidenced by the sample/duplicate and the MS/MSD exceedences.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Surrogates are monitored for recovery for all matrices. The recovery limits are found in Section 12.

Corrective Action: Check to see if an analytical or dilution error occurred and re-calculate. If only one surrogate falls below the recovery limit, but is above 10% recovery, the exceedance is noted, with approval of the Section Supervisor, and the results are reported to the client with a notation in the case narrative. If all surrogates are recovered below the limit, re-extract the sample and report the re-extract results along with the original results, if re-extraction occurred beyond the holding time, and the re-extract surrogates are within the QC limits. If the surrogates are recovered below the limit in the re-extract, this confirms a suspected matrix interference on the surrogates, and only the original analysis needs to be reported. If the chromatogram shows obvious matrix interference, no re-analysis or re-extraction is necessary. *This decision must be made with approval of the Section Supervisor.* Surrogate outliers and sample re-extracts must be noted in the case narrative to the client.

9.7.2 Internal Standards

Internal standards are added to every field sample, QC sample, standard, and method blank. The acceptance limits are -50% to +100% of the internal standard response (or area) of the daily continuing calibration verification standard.

Corrective Action: Check to see if an analytical, dilution, or spiking error occurred. If the chromatogram shows obvious matrix interference, no re-analysis is necessary. *This decision must be made with approval of the Department Manager.* Note the exceedance in the case narrative to the client. If no obvious interference is present, re-analyze the extract. If internal standards are now within the acceptance limits, report only the re-analysis, as long as the re-analysis occurred within the 40-day analytical hold time. If the re-analysis occurred outside of the 40-day analytical hold time, both the original and re-analysis must be reported. If the internal standards again are outside the acceptance limits, after re-analysis, either within or outside of the 40-day hold time, report only the original analysis, and include a narrative to the client that the suspected matrix interference on the internal standards was confirmed by sample re-analysis.

9.7.3 Standard Reference Materials

Standard reference materials (SRMs) are available from the National Institute of Standards and Technology (NIST) and are extracted and analyzed with samples on a project specific basis. These are not used as controls, but to evaluate potential matrix effects in associated samples for the target compounds being evaluated.

Acceptance criteria for SRM analysis will vary from project to project depending upon client data quality objectives (DQOs). Generally, 40% - 140% recovery of the true certified values of the target compounds of interest, serves as advisory acceptance criteria.

Corrective Action: Repeat analysis and/or check to see if an analytical error has occurred. If the % recovery or %D still exceeds the control limits and the associated LCS/LCSD and/or MS/MSD are within control, include a project narrative with the results to the client noting that the observed recovery exceedences of the SRM are isolated to this sample as evidenced by the LCS/LCSD and/or MS/MSD acceptance.

9.7.4 PEM Evaluation for Pesticide analysis only: Evaluate the percent degradation of 4,4'-DDT (to 4,4'-DDE and 4,4'-DDD) to monitor the integrity of the injection system (see Section 11.18 for calculation). Degradation is not considered to be a problem if the percent degradation of 4,4'-DDT are less than 20%. If compound does exceed 20% breakdown, the analysis must be stopped, the injection port serviced and other maintenance may need to be performed.

9.8 Method Sequence

Tune – (5 ug/mL) full scan using DFTPPBNA2/5 or PESTDFTPPBNA5.m
CCV (BNA5) – (20 or 50 ng/mL) using 136BNA5.m/PESTBNA5.m
CCV (BNA2) – (50 ng/mL) using 136BNA5.m/PCB209BNA2.m
Method Blank – (ID from sample preparation batch)
LCS – (ID from sample preparation batch)
LCSD – (ID from sample preparation batch)
Samples (up to 12-18 hours of analytical time)
Tune – (5 ug/mL) full scan using DFTPPBNA2/5 or PESTDFTPPBNA5.m
CCV (BNA5) – (20 or 50 ng/mL) using 136BNA5.m/PESTBNA5.m
CCV (BNA2) – (50 ng/mL) using 136BNA5.m/PCB209BNA2.m
Samples (up to 12-18 hours of analytical time)
CCV (BNA5) – (20 or 50 ng/mL) using 136BNA5.m/PESTBNA5.m
CCV (BNA2) – (50 ng/mL) using 136BNA5.m/PCB209BNA2.m

10. Procedure

10.1 Equipment Set-up

Prior to the analysis of any standards or samples, the instrument acquisition and processing methods must be set up. This includes the GC run parameters and the SIM mode acquisition ion entries into the different SIM acquisition retention time windows. An initial calibration must be analyzed to establish linearity of the instrument. First, the mass spectrometer must be tuned to the meet the abundance criteria for PFTBA when using maximum sensitivity tuning.

10.1.1 PFTBA Manual Tuning

10.1.1.1 Prior to initial calibration tune the mass spectrometer using PFTBA (Perfluorotributylamine - calibration gas) to maximize the sensitivity of the instrument in the mass range of interest, 45-525 amu. The use of PFTBA for MS tuning maximizes the sensitivity of the analysis within the mass/charge (m/e) range being monitored. *If only the PTFBA tune is required, per client request or project specific DQOs, the DFTPP tune in Section 10.1.2 does not need to be evaluated.*

10.1.1.2 To acquire the PFTBA Tune:

- Click on the "Instrument" icon to open the ChemStation.
- Go into "Instrument Control" in the "GC/MS Top Environmental" screen.
- Go to "View" and select "Manual Tune".
- Go to "File" and select "Load Tune Values". Select the ATUNE.U file.
- Go back into "File" and select "Generate Report". The calibration gas will automatically turn ON, equilibrate for approximately 20 seconds, and generate a report. Evaluate the PFTBA tune against the parameters below.

PFTBA Ion	Relative Abundance
m/e 69	Base Peak with > 150,000 counts
m/e 219	40% to 90% of Base Peak
m/e 502	4% to 10% of Base Peak

If the PFTBA tune meets the criteria, "Save" the tune values, and exit the program.

10.1.1.3 If the PFTBA does not meet the criteria above, an experienced mass spectrometrist may attempt the following corrective actions:

- Adjust the ion focus value up or down while the calibration gas valve is open and continue to scan until the desired abundances are achieved.
- Adjust the entrance lens value up or down while the calibration gas valve is open and continue to scan until the desired abundances are achieved.
- Save the tune parameters under PCBTUNE.U.

10.1.2 DFTPP Tuning: If only DFTPP is required, per client request or project DQOs, PFTBA does not need to be evaluated.

10.1.2.1 Before the analytical standards are analyzed, the mass spectrometer must be evaluated for the proper ion criteria for DFTPP (decafluorotriphenylphosphene), if specifically requested by the client or included in a project specific QAPP. Generally, 1-4uL of a 10x dilution of 50 ug/mL solution is evaluated. A larger volume or lesser concentration may be evaluated if using large volume injections. If the instrument has been adjusted for the maximum sensitivity PFTBA, then the criteria in Section 10.1.2.5 applies. *DFTPP must be injected under full scan mode.*

10.1.2.2 To acquire the DFTPP tune:

- Click on the "Instrument" icon to open the Chem Station, if not already open.
- *Go into the "GC/MS Top Environmental" screen.*
- *Go into "Sequence."*
- Edit the "Sample Table Log" by entering the "Vial" number starting at position 1, the "Data File ID" the acquisition method, DFTPPBNA2/5.m or DFTPPPESTBNA5.m, and finally the "Sample Name" (*i.e.*, "DFTPP 5 ug/mL") When complete, click "OK".
- Go back into "Sequence" and "Save" the sequence as the date, such as, S2060501.s. The ending "01" indicates the first sequence created on 06/05. The first number comes from instrument ID, i.e. BNA2.
- Go back into "Sequence" and "Load and Run" the sequence that was just saved.

10.1.2.3 After the analysis of the DFTPP, evaluate the tune as follows:

- Enter into the "Environmental Data Analysis" (off-line) screen.
- Go to "File" and select the tune data file.
- Go into "Tuner" and select "Eval DFTPP", then select "AutoFind DFTPP to Screen," to evaluate the tune file, based on the pre-set SW-846 criteria. The software will evaluate the tune by selecting three scans of the DFTPP peak and will display the ion intensities on the screen. That is, one scan at the apex, one scan directly preceding the apex and one scan following the apex and averages them, then takes one background subtracted scan, 20 seconds before the beginning of the DFTPP peak. If the criteria below are met, repeat, but select "AutoFind to Printer", for a hardcopy of the tune evaluation for the record.

  Note: the Maximum Sensitivity tune must be evaluated using the correct method to ensure the criteria in Section 10.1.2.5 are met.

10.1.2.4 If the "AutoFind" tune evaluation does not meet the criteria below, manual evaluation of the tune can be performed by attempting either of the options below:

- Blow up the DFTPP peak on the screen and select either one single scan at the apex of the peak, or a scan immediately preceding or following the apex. Go into "Tuner" and select "Evaluate DFTPP to Screen," or "Evaluate DFTPP to Printer," as described above, OR,

- Take the average of the scans across the entire peak. Go into "Tuner" and select "Evaluate DFTPP to Screen," or "Evaluate DFTPP to Printer," as described above.

10.1.2.5 "Maximum Sensitivity" DFTPP is used for the analysis of PCB Congeners by GC/MS. It shows high-end sensitivity for the higher molecular weight ions that are present in, and evaluated for PCBs.

DFTPP KEY MASSES AND ABUNDANCE CRITERIA
(Maximum Sensitivity)

Mass	m/e Abundance criteria
51	N/A
68	N/A
70	N/A
127	30-80 percent of mass 198.
197	Less than 3 percent of mass 198.
198	Greater than 40 percent of mass 442.
199	5-15 percent of mass 198.
275	15-50 percent of mass 198.
365	Greater than 3 percent of mass 198.
441	Present but less than mass 443.
442	Base peak, 100 percent relative abundance.
443	18-30 percent of mass 442.

10.1.2.6 Tune acceptance must be verified at the beginning of every analytical shift, and prior to the analysis of any standards, and again at the beginning of each 12-18 hour tune clock as defined by the injection time of each DFTPP analysis. If the DFTPP tune does not meet the criteria above, the PFTBA must be re-evaluated, and adjustments made by an experienced mass spectrometrist, to obtain an acceptable DFTPP tune, before continuing with any analysis.

10.1.3 Degradation Evaluation – only if Pesticides are targets of interest

10.1.3.1 Prior to initial calibration and at the start of each run a 44DDT degradation must be evaluated in DFTPP as mentioned above in the quality control section 9.7.4.

10.1.4 GC Instrumental Conditions

10.1.4.1 For 136 Congener, Homologs, Pesticide Analysis Inject an aliquot of 1uL to 5uL into the capillary column of the gas chromatograph at the following conditions. Injection volume (using the Large Volume Injector, LVI) amount will be dictated by project specific DQOs.

GC Parameter	*136 Congeners Setting*	*Pesticides Setting*
Injector Temp:	*70 - 300 °C*	*70 - 300 °C*
Transfer Line Temp:	*280 °C*	
Initial Oven Temp:	*50°C*	*70°C*
Initial Hold Time:	*2.5 minutes*	*1 minute*
Ramp Rate 1:	*25 °C / minute*	*25 °C / minute*
Final Temperature 1:	*180 °C*	*190 °C*
Final Hold Time 1:	*0 minute*	*0 minute*
Ramp Rate 2:	*3 °C / minute*	*3 °C / minute*
Final Temperature 2:	*250 °C*	*240 °C*
Final Hold Time 2:	*0 minute*	*0 minute*
Ramp Rate 3	*15 °C / minute*	*15 °C / minute*
Final Temperature 3:	*300 °C*	*300 °C*
Final Hold Time 3:	*11 minutes*	*8.53 minutes*
Total runtime:	*45.37 minutes*	*35 minutes*
Mode:	*Splitless / Constant Flow 1.2 ml/min*	*Splitless / Constant Flow 1.2 ml/min*
Purge:	*25 mL / minute – on at 2.5 minutes*	*25 mL / minute – on at 2.5 minutes*
MS Temperature:	*250 °C, MS Source, 170 °C, MS Quad*	*250 °C, MS Source, 170 °C, MS Quad*

10.1.4.2 For 209 Congener and Homolog Analysis inject an aliquot of 1uL to 50uL into the capillary column of the gas chromatograph at the following conditions. Injection volume (using the Large Volume Injector, LVI) amount will be dictated by project specific DQOs.

GC Parameter	***Setting***
Injector Temp:	*70 - 300 °C*
Transfer Line Temp:	*300 °C*
Initial Oven Temp:	*65°C*
Initial Hold Time:	*2.1 minutes*
Ramp Rate 1:	*25 °C / minute*
Final Temperature 1:	*170 °C*
Final Hold Time 1:	*0 minute*
Ramp Rate 2:	*3 °C / minute*
Final Temperature 2:	*290 °C*
Final Hold Time 2:	*15 minutes*
Total runtime:	*61.3 minutes*
Mode:	*Splitless / Constant Pressure or Solvent vent for LVI*
Purge:	*25 mL / minute – on at 2.00 minutes*
MS Temperature:	*250 °C, MS Source, 170 °C, MS Quad*

10.1.5 Mass Spectrometer Conditions

The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the SIM mode using appropriate retention time windows to include the quantification and confirmation ions for each congener and the interference ions as shown in Table II.

10.1.6 Large Volume Injection (LVI) Parameters

*Gerstel **	*Settings**
Injector Temp:	*70 - 300 °C*
Initial Hold Time:	*30 sec. - 3 minutes*
Flow Rate:	*0.5 - 5.0 mL / minute*
Purge:	*25 mL / minute – on at 2.00 minutes*
Injection Volume:	*1uL - 50uL*

* = The settings listed may vary from project to project, based on client specific DQOs. *Injection temperature, hold time, flow rate, purge time, and injection volume can affect chromatographic resolution and detection limits.* All parameters listed above can be set within the above setting ranges. Only a trained and experienced mass spectrometrist has the authority to change any setting. All standards and samples must be acquired using the same set of parameters. If any parameters are changed, a new initial calibration must be analyzed and accepted before any samples can be analyzed.

10.1.7 Data Acquisition Parameters

10.1.7.1 SIM Windows must be set up that bracket the expected retention times for each target analyte. These windows include the quantitation (primary) and confirmation ions for each congener Homolog group. To establish the expected retention time window ranges, the mid-level retention time window standard containing the first and last eluting congener in each Homolog group, must be analyzed in full scan mode. The resulting full scan analysis will dictate the windows in which the selected ions will be monitored. Depending upon the length of the analytical GC column, the time each window is selectively monitored may vary. The retention time windows must be shifted accordingly, when instrument maintenance is performed, (*i.e.*, the column is clipped).

10.1.7.2 The "dwell" time for each window should be set close to 20 and the resolution should be set to "high." For pesticides it is set to "low".

10.2 Initial Calibration

10.2.1 Before analysis of sample extracts, establish a multi-point response factor calibration curve showing the linear range of the analysis for all target analytes in Table II. Use at least 5 levels of standard concentrations at 0.5, 1.0, 10, 20, 50, 200, and 500 or 400 ng/mL to construct the curve. See Section 8.16 for the preparation of the standard solutions for the initial calibration curve. Up to 9 levels might be analyzed for Pesticides.

10.2.2 For PCB Aroclors single point calibration factors are used. The response of each individual peak in the sample is compared to a calibration standard to determine the analyte concentration in the sample. Using the GC system software, the analyst must choose 3-5 peaks from the pattern which are characteristic for the Aroclor to obtain the response for the component of interest. The peak area is calculated against the mass injected to obtain a Calibration Factor. Calibration Factors are determined for individual peaks. The calibration factors are then used to calculate the concentration of each corresponding peak in the sample. The 3 to 5 resulting concentrations are averaged to provide the final result for the aroclor in the sample.

10.2.3 Construct an analytical sequence using the HP Enviroquant software:

- Click on the "Instrument" icon to open the ChemStation
- *Go into the "GC/MS Top Environmental" screen into "Sequence"*
- Edit the "Sample Table Log" by entering the "Vial" number starting at position 1, the "Data File ID" ,the acquisition method (such as, 136CONGBNA5.m, which indicates the type of method and the instrument ID), and finally the "Sample Name" (such as, "I206051001STD0.5", for the first standard concentration level, etc.).
- Go back into "Sequence" and "Save" the sequence as the date, such as, S2060501.s. The ending "01" indicates the first sequence created on 06/05. The first number comes from instrument ID, i.e. BNA2.
- Go back in to "Sequence" and "Print" the sequence that was just saved. This will become part of the instrument run log. See Section 11.0 for additional instrument run log details.
- Go back into "Sequence" and "Load and Run" the sequence that was just saved.

10.2.4 When the sequence has finished running, the Enviroquant software will generate "Not Reviewed" quantitation reports. All reports must be "Quant Reviewed" before they can become part of the initial calibration processing method for sample analysis.

- Enter into the "Environmental Data Analysis" (off-line) screen.
- Go to "File" and under method, select the processing method (136Cong0605BNA5.m) that the initial calibration standards will be quantitated with.
- Go into "Quant" and select "QEdit Quant Results" to process the data files. See SOP Manual Integration 08-03 for manual integration details and Section 11.0 for processing of PCB Congener standards.
- When processing is complete for the first standard, "Save" the changes and "Exit." Re-print the re-processed data file by "Generating Quant Report," and save the hard copy for each level of the initial calibration.
- Repeat these steps for all initial calibration standards analyzed within the sequence.
- When the appropriate levels have been processed, go into "IntiCal," and select "Update Levels," and enter all levels for the initial calibration at the proper concentrations. Note: The PCB Homolog *group* responses *must be hand entered* into the calibration curve, or utilizing the RF macro, (*i.e.,* for Mono-, Dichlorobiphenyl, etc.) using the response of the appropriate calibration congener (*i.e.,* BZ1, BZ8, etc.).
- After all responses are entered, "Save" the completed method and print the resulting response factor summary by selecting "Response Factors to Printer."
- Acceptance Criteria: 20% RSD for all target compounds, except 10% of the analytes may be >20% RSD but $\leq$ 30% RSD. All calibration standards must be analyzed within 12-18 hours.
- Replace the Qion ratio values from the mid-point concentration level of the ICAL, by checking off the appropriate box, then update and "Save" the new method.
- Replace the reference spectra for the method from the spectra in the mid-point concentration level of the ICAL by going into "ConCal" and select "Update Reference Spectrum." Again, "Save" the method.
- Establish retention time window ranges from the first and last eluting congener within each chlorination level. Set the integration window ranges for each Homolog group (*i.e.,* Monochlorobiphenyl, Dichlorobiphenyl, etc.) by using the Easy ID function in the Enviroquant software.

10.2.5 If using greater than five calibration levels in the initial calibration, standards must only be excluded from either extreme. That is, the low-level standard or the high-level standard may be dropped to generate a five-level initial calibration. However, an intermediate-level calibration standard must not be dropped to convert a failing six-level initial calibration curve into a passing five-level initial calibration curve. Reduction in the number of calibration standards must also reduce the linear dynamic range used to quantify analytes in samples. The resulting average response factor for each target analyte in the

initial calibration curve will be used by the computer software to calculate actual sample concentrations. See Section 11.0 for additional calculation details.

10.2.6 The following *corrective actions* are recommended for failing initial calibrations:

- Perform instrument maintenance and repeat the initial calibration, OR,
- Qualify all results reported for the analyte failing in the initial calibration, including all Homolog chlorination range(s) quantified using the suspect average response, and any non-detects. If the failure of the suspect average response appears related to a loss in MS sensitivity, instrument maintenance and repeat of the initial calibration curve must be performed.

The choice of corrective action must be made in consultation with the Section Supervisor, QA Manager, Project Manager, and/or the client. The reasoning for choosing the second option must be documented in the project narrative to the client.

10.2.7 Alternately, a linear regression model may be employed, provided that the coefficient of determination (COD or r^2) is $\geq$0.99. Otherwise, construct a nonlinear calibration of no more than a third order equation. Statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approach. A quadratic (second order) model requires six standards, and a third order polynomial requires seven standards. In setting model parameters, do not force the line through the origin. The COD or r^2 must be greater than or equal to 0.99. The experienced analyst must select the regression order, which introduces the least calibration error into the quantitation.

10.2.8 Complete the initial calibration by filling out the *Initial Calibration Checklist*. The initial calibration, along with any corresponding continuing calibration data and sample data, is then forwarded for secondary review.

10.2.9 Initial Calibration Verification - ICV (separate source)

The analysis of separate source standard must follow the initial calibration curve.
After final processing, calculate the percent recovery of each congener by using the following calculation:

% Recovery = Found Amount / True Value x 100

Acceptance Criteria: All compounds must agree within +/-30%D with the exception of the partially or fully coeluting congeners that are identified, measured and calibrated together and at least one of the coeluting compounds is not present in the ICV solution. The Compounds listed in section 4.4 should also be excluded from the evaluation due to the described interference.

10.3 Equipment Operation and Sample Processing

10.3.1 Evaluate the PFTBA tune and/or DFTPP tune as described in Sections 10.1.1 and 10.1.2. *The type of tune required will depend upon the client/project DQOs.*

10.3.2 If Pesticides are targets of interest, a 44DDT Degradation must be evaluated as mentioned in Quality Control Section 9.7.4.

10.3.3 Samples are prioritized for analysis by the Organic Section Supervisor or GC/MS Group Leader based on client due date and sample analytical hold time. Samples are retrieved from the sample storage refrigerator, spiked with 20uL of the chosen internal standard solution per 1mL extract from either Section 8.11 or 8.12, and loaded into the instrument autosampler trays following the generalized sequence below.

- Tune – (5 ug/mL) full scan using DFTPPBNA2/5.m or PestDFTPPBNA5.m
- CCV – (50 ng/mL) using 136/PestBNA5.m or PCB209BNA2.m
- Method Blank – (ID from sample preparation batch)
- LCS – (ID from sample preparation batch)
- SRM – (ID from sample preparation batch) if required
- Samples (up to 12-18 hours of analytical time)
- Tune – (5 ug/mL) full scan using DFTPPBNA2/5.m or PestDFTPPBNA5.m
- CCV – (20/50 ng/mL) using 136/PestBNA5.m or PCB209BNA2
- Samples (up to 12-18 hours of analytical time)
- CCV – (20/50 ng/mL) using 136/PestBNA5.m or PCB209BNA2

10.3.4 Samples are processed from "Not Reviewed" data files, to "Quant Reviewed" data files in a similar way the standards were previously processed. See Section 11.0 for details on sample processing. If a CCV fails the criteria outlined in Section 10.4.3, all samples since the last acceptable CCV must be re-analyzed.

10.3.5 If the on-column concentration of any compound exceeds the concentration of the highest calibration standard, the sample must be diluted, re-spiked with the appropriate amount of internal standard and re-analyzed. Assuming all samples are at a 1mL final volume, the following example dilutions would apply. Adjust the volumes accordingly for other sample final volume amounts and other desired dilutions.

- 1:2 dilution = 500uL of sample : 500uL Hexane and 10uL of IS
- 1:4 dilution = 250uL of sample : 750uL Hexane and 15uL of IS
- 1:5 dilution = 200uL of sample : 800uL Hexane and 16uL of IS
- 1:10 dilution = 100uL of sample : 900uL Hexane and 18uL of IS, etc.

10.4 Continuing Calibration

A continuing calibration verification (CCV) standard, at the concentration of the mid-level of the initial calibration curve, must be analyzed at the beginning and end of every analytical sequence, and every 12-18 hours within the sequence, to confirm instrument stability, via response factor, for each calibrated congener.

10.4.1 After successful analysis of the PFTBA or DFTPP tune (Section 10.1.1 or 10.1.2), "Edit" the "Sample Table Log" to include the 50 ng/mL CCV standard and acquire the CCV against the correct initial calibration method. "Save," then "Load and Run" the sequence, as in Section 10.2.3.

10.4.2 When the sequence has finished running, the Enviroquant software will generate a "Not Reviewed" quantitation report. All reports must be "Quant Reviewed" against the processing method for sample analysis.

- Enter into the "Environmental Data Analysis" (off-line) screen.
- Go to "File" and under method, select the method that the CCV was analyzed under, then select the CCV data file.
- Go into "Quant" and select "QEdit Quant Results" to process the CCV file. See SOP Manual Integration 08-03 for manual integration details and Section 11.0 for processing of PCB congener standards.

When processing is complete, go into "ConCal," and select "Evaluate Data File as Continuing Calibration." **Note:** The PCB Homolog group CCV responses may be omitted since the calibration congener associated with the Homolog group, is the quantitation congener for that same Homolog group.

10.4.3 Acceptance Criteria: Compare the CCV resulting response against the average response for the initial calibration for each calibrated congener and/or pesticide, and calculate the % difference (%D). See Section 11.0 for the calculations. The %D for each calibrated congener and/or pesticide must be below 20%D, except up to 20% of the analytes may be > 20%D but ≤ 30%D. If multiple CCVs are analyzed within an analytical sequence, each CCV must be analyzed within 12-18 hours of the previous CCV, and each CCV, including the ending CCV, must meet the acceptance criteria.

Additional Criteria:

1) The area counts of the internal standards must be within 50-200%of the mid-point standard level from the most recent initial calibration .
2) The retention time of the internal standards must be within 30 seconds of the previous daily standard.

If the CCV meets the acceptance criteria, save the hard copy for each CCV standard and include it with the *Continuing Calibration Checklist.*

Go back into "ConCal" and select "Update Continuing Calibration" and "Save" the method updated to the opening CCV of the day.

10.4.4 If the CCV does not meet the criteria for each calibrated analyte, the following *corrective actions* are recommended:

- Perform instrument maintenance and re-analyze the continuing calibration standard and all affected samples, OR,
- Perform instrument maintenance and repeat the initial calibration, and re-analyze all affected samples, OR,
- If the closing CCV does not meet the criteria and the sample chromatograms show obvious matrix interference, no re-analysis is necessary. *This decision must be made with approval of the Section Supervisor.* CCV outliers and affected samples must be noted in the case narrative to the client.
- If the failure of the suspect response appears related to a loss in MS sensitivity or other instrument related issues, instrument maintenance and repeat analysis of all affected samples and/or the initial calibration curve must be performed.

The choice of corrective action must be made in consultation with the Department Manager, QA Manager, Project Manager, and/or the client. The reasoning for choosing the third option must be documented in the project narrative to the client.

10.5 Preventive Maintenance

10.5.1 Preventive maintenance may include the following: replacing glass liner, ferrules, PTV injection port bottom adapter and/or clipping a length of the analytical column.

10.5.2 Additionally, preventive maintenance for GC-MS system may involve baking out the injection port and the oven, cleaning the ion source, and/or replacing the analytical column.

11. Data Evaluation, Calculations and Reporting

11.1 After sample analysis, "Not Reviewed" quantitation reports are generated by the software system. It is expected that situations will arise when the automated quantitation procedures of the chromatographic software provide inappropriate quantitations or integrations. This normally occurs when there is compound co-elution, baseline noise or matrix interference with the PCB congener compounds. However, with PCB Homolog groups, a range or cluster of peaks is evaluated and manual integration must be performed for each PCB Homolog group or chlorination level cluster. See SOP Manual Integration 08-03 for manual integration details.

11.2 Qualitative identification of multicomponent analytes (Aroclors) requires pattern matching between the calibration standards and the response observed in the sample on both columns. Retention time windows should be used as a gauge; however, pattern recognition for the multicomponent analytes is most important. For samples with PCB Aroclors positively identified, compare the responses of the 3 to 5 major peaks in the single point calibration standard for that Aroclor with the responses of the peaks observed in the sample extract. The relative peaks and number of peaks in the sample should be similar to that observed in the standard; however, degradation, weathering and interferences may cause the sample pattern to differ from that observed in the standard.

The peaks chosen for quantitation must be free from interferences. If the interference or co-elution/overlapping with another Aroclor is observer, the analyst has an option to exclude the affected peaks from the final calculation. At least 3 out of 5 peaks must be used to provide the final concentration. Calculate the concentration of each corresponding peak in the sample chromatogram and the 3 to 5 resulting concentrations are averaged to provide the final result for the sample.

11.3 Identification of the PCB congener and pesticide compounds are based on gas chromatographic relative retention times (RRTs) from the analysis of the mid-level initial calibration standard. For these compounds, manual quantitations are performed, if necessary, by integrating the area of the quantitation ion or peak. For the ten *PCB Homolog groups*, the Homolog groupings (*i.e.,* Dichlorobiphenyl) appear in the extracted ion current profiles (EICPs) as a cluster of congeners with the same degree of chlorination. Establish the pattern of each Homolog group by comparing the primary and secondary ion profiles. Refer to the Manual Integration SOP 1731 for details on Manual Integration. Manually integrate candidate peaks by straight-line integration to the baseline, taking into account background noise in the EICPs for each Homolog group within each determined Homolog-specific retention time window. If a discrete peak, either target or non-target, does not have a confirmation ion, or the experienced analyst judges that the confirmation ion does not meet the ratio criteria, the area for that discrete peak can be measured and subtracted from the total area used to calculate the concentration for that Homolog group. The experienced analyst can also choose not to include the interference in the manual integration even if the interference appears within the retention time window established by retention time markers for specific Homolog group. If there is interference observed within Homolog-specific retention time window that cannot be excluded or subtracted out, the results for the affected Homolog group will be qualified with the G flag and narrated in the case narrative to the client. See the most recently generated "detailed" PCB reference spectrum hardcopy that is based on the most recent analysis mid-point standard of the ICAL. Table II, in Section 16.0, lists the representative ion(s) used for quantitation and confirmation of each parent congener and PCB Homolog group.

Note: Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system.

11.4 From EICP of the quantification (primary) mass ions and the confirmatory mass ions, identify all target analytes according to the following criteria:

- Surrogates and internal standards should meet the acceptance criteria in Section 12.0.
- Examine the chromatograms for evidence of saturated ions in mass spectra. Re-analyze the sample(s) at the appropriate dilution(s), see Section 10.3.4, as needed.
- The characteristic ions (primary and secondary) of each pesticide, congener and/or Homolog group of interest should maximize at the same scan, or within one scan of each other.
- The retention time should fall within ± 10 seconds of the retention time of the authentic target compound or PCB Homolog grouping. *Note*: For PCB Homolog groups, the most intense peak within the group may not have the exact retention time of a calibration congener. Analyst judgment and referral to each Homolog groups' retention time window and group-specific pattern is essential for identification. Apply analyst judgment regarding corrective action, as needed, when these criteria are not met.
- The relative peak height of the quantitation ion compared to the confirmation ion for parent analyte should fall within ± 50 percent of the relative intensities of these ions in the reference mass spectrum (*i.e.,* the mid-level standard of the initial calibration curve).

Note: The relative intensities of the quantitation and confirmation ions may vary widely within a given group of PCB Homologs. Thus, the pattern of each PCB Homolog cluster, and the retention time window for the cluster, will be the primary identification criteria for PCB Homologs. In some instances, a parent congener that does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by the experienced mass spectrometrist. Supportive data includes the presence of the secondary ion, but ratio value greater than ± 50 percent of the primary ion, which may be caused by an interference of the secondary ion. See Section 11.6 for interferences.

11.5 In instances where manual integrations have been performed, they are assigned one of the Manual Integration codes, (Refer to Manual Integration SOP 1731) and can be found on the raw data provided within the data deliverable package The "detailed" report, displaying the manual integration, including an "m" qualifier(s) next to the modified or manually integrated compound(s), shall be provided to the LIMS for secondary review. These requirements apply to all standards, QC samples, field samples and blanks.

11.6 To calculate the ***Relative Standard Deviation*** (RSD) of all PCB congeners, Homolog groups, and surrogate compounds for the initial calibration, use the formula below. See Section 10.2 for initial calibration acceptance criteria. Additionally, use the initial multi-point calibration to determine ***Relative Response Factors*** (RRF_Is) at each concentration level, for each PCB congener. Average the RRF_Is from the initial multi-point calibration, to generate mean RRF_Is, for quantification of each PCB congener. Follow the same calculations for each surrogate compound. The RRF_I for the quantification of each Homolog group is the RRF_I of the PCB calibration congener assigned to that Homolog group (*i.e.,* Tricholorbiphenyls are quantified using the RRF_I of the PCB calibration congener BZ29, which is associated with the Trichlorobiphenyl group). The RRF_Is are based on the internal standard compounds, and are calculated using the formula below. (The relative response factors for the continuing calibration verifications (RRF_Cs) are calculated using the same formula). See Section 16.0, Table II, for the listing of target compounds and their associated internal standards for quantification.

$$RSD = SD / \text{mean } RRF_I \times 100$$

where:

SD = Standard deviation between the five points, for that target analyte.

$$RRF_I = (A_c \times C_{IS}) / (A_{IS} \times C_c)$$

where:

A_c = Area of the characteristic ion for the standard compound to be measured.

A_{IS} = Area of the characteristic ion for the representative internal standard compound.

C_{IS} = Concentration of the representative internal standard compound (ng/mL).

C_c = Concentration of the standard compound to be measured (ng/mL).

Note: Assign the response factor of the calibration congener compound to the Homolog group, (*i.e.,* use BZ 5/8 for the Dichlorobiphenyl group).

11.7 Based on the mean RRF_Is, calculate the ***Sample Extract Amount*** for each Pesticide, PCB congener or Homolog group and surrogate in the sample extracts using the following formula:

$$Q_e = (A_a \times Q_{IS}) / (A_{IS} \times RRF_I)$$

where:

Q_e = Sample extract concentration (ng/mL) of target analyte, from quantitation report.

A_a = Area of the characteristic ion for the target analyte.

A_{IS} = Area of the characteristic ion for the representative internal standard compound.

Q_{IS} = Concentration (ng/mL) of representative internal standard compound, from quantitation report.t.

11.8 Calculate the ***Sample Concentration*** (C) for each Pesticide, PCB congener or Homolog group by the following formula:

$$C = (Q_e / V_s) \times FV \times DF$$

where:

C = Concentration in sample (ng/L water, ug/Kg sediment/tissue or ng/cert for PUF).

V_s = Original volume or weight of sample extracted, corrected for % solids, if applicable. (To correct for % solids, multiply the sample weight by the % solid as expressed as a decimal. For example: 15.24g x 0.843 = 12.85, for a sample size of 15.24g at 84.3% solid).

DF = Dilution factor

FV = Sample Final Volume or Final Effective.

If the response of any analyte in a sample exceeds the linear response range, as defined by the initial calibration standards in Section 10.2, dilute the extract so that the concentration of that analyte falls within the range of the calibration curve.

Note: A Homolog group and PCB Aroclor exceed the calibration level if one single peak in the integration group exceeds the linear response range. If no single peak exceeds the range the total integration range may result above the calibration range. If the target compound in a sample is detected below the reporting limit (RL) but above half of the RL, qualify the reported concentration with a "J". If any target compound is found in the method blank and in the associated sample(s), qualify the reported concentration with a "B" if detected less than 10x the blank concentration for this analyte.

11.9 To calculate ***Total PCBs*** when analyzing for *less than 209 PCB Congeners* use the following:

- Each Homolog group will be identified and final concentration will be calculated using the formulas above.
- Sum all Homolog group concentrations.
- If a Homolog group is non-detect, zero is used in the summation. This minimizes the potential for a high bias result.

To calculate ***Total PCBs*** when analyzing for *all 209 PCB Congeners*, sum all identified congeners.

11.10 Calculate the ***Surrogate Recoveries*** relative to the internal standards by the following formula:

$$\%R_{sc} = (A_{sc} / A_{is}) \times (Q_{is} / Q_{sc}) \times (100 / RRF_{sc}) \times DF$$

where:

Q_{is} = Amount of the representative internal standard (ng).

DF = Dilution factor or fraction of the original extract.

11.11 Compare response factors for each PCB congener in the ***Continuing Calibration Verification*** (CCV), to those of the initial calibration curve by determining the percent difference.

$$\text{Percent Difference (\%D)} = ([RRF_I - RRF_C] / RRF_I) \times 100$$

where:

RRF_I = Mean response factor from initial calibration.

RRF_C = Response factor from CCV.

11.12 All results must be reported to three significant figures. All solids including soils, sediments, and sludge must be reported on a dry-weight basis. Tissue results may be reported on a dry-weight, or "as received," basis depending upon client request. PUF samples are reported "as received".

11.13 The primary analyst does data entry, or upload of the data, into the LIMS system. The LIMS is "linked" to the instrument, so the analyst must choose the sample(s) to be reported from that instrument's analytical sequence. All associated preparation and instrumental QC samples and dilutions are also chosen. Once the data/samples have been selected and "associated" with the proper QC samples, the batched data set is sent to print. In addition to the concentration of

each selected PCB congener and Homolog group on the report, the Total PCB concentration is also reported.

11.14 The laboratory generates two types of data packages from the LIMS: "Commercial" or "Standard" for routine projects, and "Full Deliverable" or "CLP-like" for fully data validated projects. A Commercial/Standard package consists of sample results and the associated method blank and LCS/LCSD results. A Full Deliverable/CLP-Like package includes all sample results, all preparation and instrumental QC results and the associated supporting raw data. Check the "Report Type" on the project folder to ensure all required deliverables are included. A secondary review is performed on all data.

11.15 Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP 1729. All records shall be stored in such a manner as to be safe and accessible for at least 10 years.

11.16 Notebooks: Laboratory notebooks are designed to accommodate the specific analysis. Instrument printouts are used to document run sequences, and each daily sequence printout is filed in a three-ring notebook. If a sample requires re-analysis or re-extraction for any reason, a notation is made next to the sample entry on the sequence log. Requests for re-extraction are further documented in the "Request for Re-extraction, Re-clean" logbook. At regular intervals the sequence run log is permanently bound, assigned an internal ID number, and filed accordingly. Such files shall be archived so as to remain available for at least 10 years. All laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* Work Instructions, WI 108-01, and all record keeping and document control practices.

11.17 Electronic records: All data files from computers, attached to instruments, shall be backed up daily onto the proper directory on the server. The backups shall be stored so as to be accessible for 10 years. Movement of the data files to the server is the responsibility of the primary analyst. Server backup and storage is the responsibility of the IT department.

11.18 The percentage breakdown for DDT:

$$\textbf{\%Breakdown DDT} = \frac{(\text{Area DDD} + \text{Area DDE})}{(\text{Area DDD} + \text{Area DDT} + \text{Area DDE})} \times 100$$

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

All PCB congener, Pesticides and Homolog results are reportable without qualification if analytical holding times are met, preservation requirements (including cooler temperatures) are met, and all QC criteria defined in the table below are met. If any of the below QC parameters are not met, all associated samples must be evaluated for re-analysis. See Sections 9.0 and 10.0 for additional QC discussion including corrective actions for any QC outliers.

QC Parameter	Acceptance Criteria
Initial Calibration Curve	20% RSD for all target analytes with exception for 10% of target analytes to be >20%, but ≤ 30%
Independent Check Verification	+/- 30% recovery of the true values
Continuing Calibration Verification	Analyzed every 12-18 hours, 20% D for all target analytes with exception for 20% of target analytes to be >20%, but ≤ 30%
Method Blank	No analyte at or above the reporting limit, The results are qualified with a "B" for any associated sample concentrations that are less than 10x the blank concentration for this analyte.
Laboratory Control Samples (LCS/LCSD)	40-140%; 30% RPD (sporadic marginal failure criteria applies)
Matrix Spike / Matrix Spike Duplicate	Same as for LCS; 30% RPD between the duplicates.
Sample / Sample Duplicate	30% RPD between the duplicates.
Surrogates	50-125%
Internal Standards	50% - 200% of the daily CCV area for the Internal Standards
SRM	40% - 140% recovery

Section 9.0, Quality Control, defines the corrective actions that must be taken in instances where QC outliers exist.

If non-compliant Pesticide, PCB congener or Homolog results are to be reported, the Department Manager, the Laboratory Director, and/or the QA Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1732 MDL/LOD/LOQ Generation

SOP 1739 IDC/DOC Generation

SOP 1797 Waste Management and Disposal

SOP 1731 Manual Integration & Compound Rejection

16. Attachments

Table I: PCB Homolog Groups

Table II: PCB Homolog Quantification, Confirmation Ions

Table I: PCB Homolog Groups

Compound	Surrogate and IS Reference		Compound	Surrogate and IS Reference
	136Cong	209Cong		
Monochlorobiphenyls	B	A	Surrogate Compounds	
Dichlorobiphenyls	B	A	DBOB	A
Trichlorobiphenyls	B	A	BZ 198	B
Tetrachlorobiphenyls	B	A	Carbon-labeled Surrogates	
Pentachlorobiphenyls	B	A	BZ 19	A
Hexachlorobiphenyls	B	A	BZ 202	B
Heptachlorobiphenyls	B	B		
Octachlorobiphenyls	B	B	Carbon-labeled ISs	
Nonachlorobiphenyls	B	B	BZ 15	A
Decachlorobiphenyl	B	B	BZ180	B

Note: Individual congeners may also be reported, as needed, depending upon client/project specific DQOs. Each congener depends on the level of chlorination would use the surrogates and internal standards similar to its chlorination grouping.

Table II: PCB Homolog Quantification, Confirmation Ions

Parameter	1^0 Ion	2^0 Ion	CASRN	Acceptance Ratio
Monochlorobiphenyls	188	190	27323-18-8	2.5-3.5
Dichlorobiphenyls	222	224	25512-42-9	1.3-1.7
Trichlorobiphenyls	256	258	25323-68-6	0.8-1.2
Tetrachlorobiphenyls	292	290	26914-33-0	1.1-1.5
Pentachlorobiphenyls	326	324	25429-29-2	1.4-1.8
Hexachlorobiphenyls	360	362	26601-64-9	1.0-1.4
Heptachlorobiphenyls	394	396	28655-71-2	0.8-1.2
Octachlorobiphenyls	428	430	31472-83-0	0.8-1.1
Nonachlorobiphenyls	464	466	53742-07-7	1.1-1.5
Decachlorobiphenyl	498	500	2051-24-3	0.9-1.3