



CAMPBELL  
ENVIRONMENTAL GROUP

September 18, 2015

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

**Re: Phase II Environmental Site Specific Quality Assurance Project Plan Addendum  
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 Site Specific Quality Assurance Project Plan Addendum 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.

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.

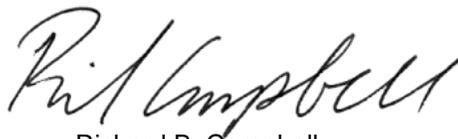
REC#8: Polychlorinated biphenyls may be present in the building materials of Buildings #85 and #103.

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:

<b>Tax Map/Lot</b>	<b>2010 Survey Lot Designation</b>	<b>Acreage</b>
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 stormwater eventually discharges to the ocean west of the Subject Property.

## 1.2 Geology and Hydrogeology Summary

<b>Table 3 Site Geology and Hydrogeology</b>		
<b>Feature</b>	<b>Source</b>	<b>Description</b>
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. Areas 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:

<b>Table 1-3 Contaminants of Concern</b>	
<b>Recognized Environmental Condition (REC)</b>	<b>Contaminants of Concern (COCs)</b>
<b>REC #1-</b> 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.	<ul style="list-style-type: none"> <li>• Lead from paint chips impacting soil near exterior walls</li> </ul>
<b>REC #2-</b> 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.	<ul style="list-style-type: none"> <li>• Historical use of herbicides/ pesticides could be the cause of no plant growth within the CDAA</li> </ul>
<b>REC #3-</b> 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.	<ul style="list-style-type: none"> <li>• #2 fuel oil constituents from the UST</li> </ul>
<b>REC #4-</b> The presence of broken fluorescent light bulbs particularly in Building #85 may have resulted in the release of mercury to the floors and walls.	<ul style="list-style-type: none"> <li>• Mercury vapor from broken fluorescent bulbs may have impacted air quality and condensate mercury may have impacted porous building material</li> </ul>
<b>REC #5-</b> The pesticide mixing area west of Building #103 has not been tested for herbicides or pesticides.	<ul style="list-style-type: none"> <li>• Pesticides and herbicides from the pesticide mixing area</li> </ul>
<b>REC #6-</b> 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.	<ul style="list-style-type: none"> <li>• VOCs and RCRA metals suspected from solvents discharged to floor drain</li> </ul>
<b>REC #7-</b> 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.	<ul style="list-style-type: none"> <li>• Asbestos based on visual observation of friable material and date of buildings constructed</li> </ul>
<b>REC #8-</b> Based on the construction date of Buildings #85 and #103, the presence of poly-chlorinated biphenyls (PCBs) containing caulk and paint should be investigated	<ul style="list-style-type: none"> <li>• PCBs in air and building material. PCB containing caulk and paint consists primarily of aroclors 1254 and 1242</li> </ul>

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 and although PCBs have a low vapor pressure they can migrate into the air and be transported with air movement to other surfaces.

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

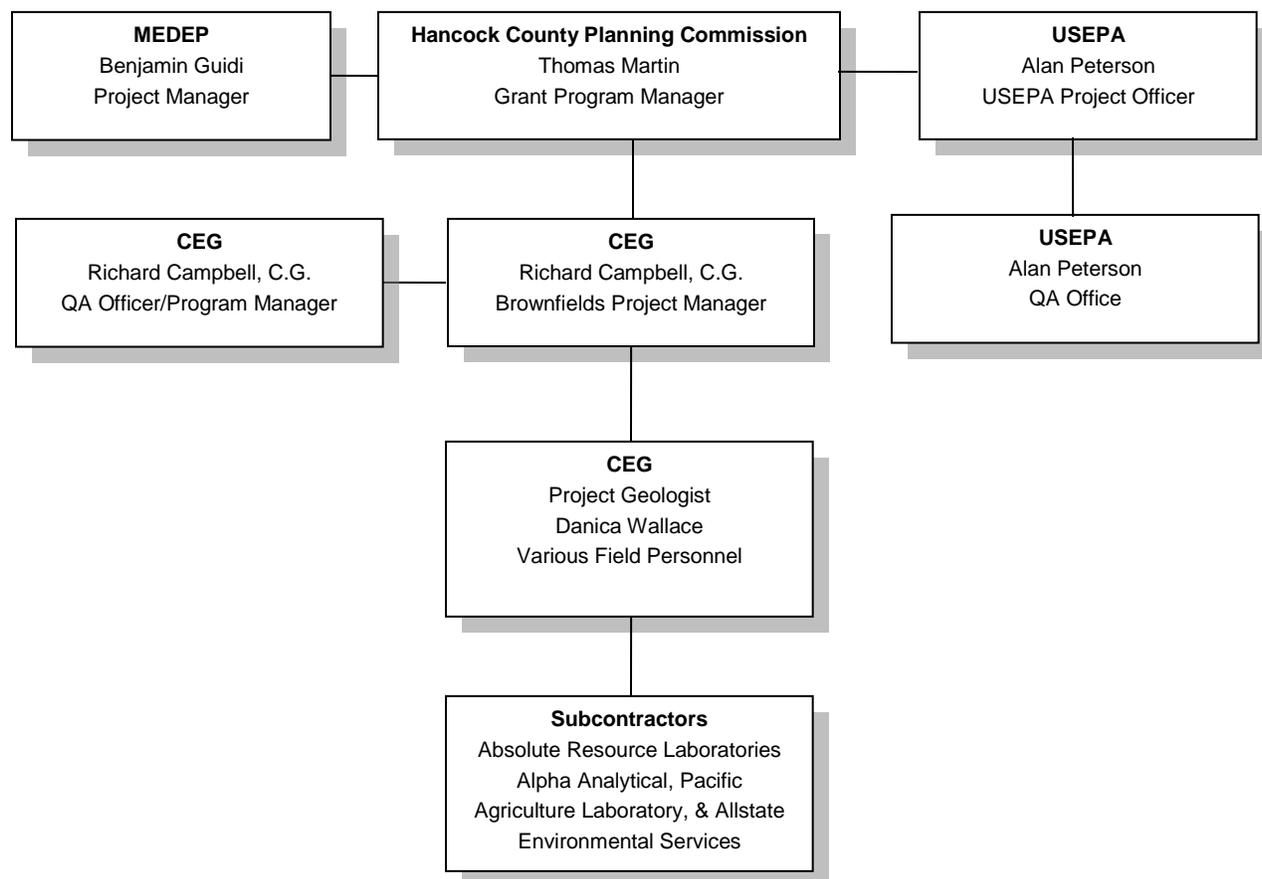
This section summarizes the organizational structure for this project.

### 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**



<b>Table 2-2 Project Personnel Responsibilities</b>			
<b>Name</b>	<b>Title</b>	<b>Organizational Affiliation</b>	<b>Responsibilities</b>
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;
- Mark the Site for Dig Safe;
- Collect building material samples from Building #85 for the analyses of lead, mercury, and PCBs in level C personal protective equipment (PPE) using a respirator cartridge specifically for filtering mercury vapors;
  - Screen building surfaces and ambient air for mercury using a mercury vapor analyzer;
  - Screen building surfaces for lead using an XRF;
  - Collect building material samples for analysis of PCBs;
  - Collect one 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;
- Set 24-hour air sample canister for the analysis of mercury at the time of the building material sampling;
- Investigate the floor drain piping in Building # 156 to determine the discharge point;
- Sample inlet and outlet of Building # 156 drain for contaminants of concern;
- Conduct test pitting to explore subsurface conditions in the vicinity of the current/former fuel oil, diesel fuel USTs, the Wullenweber CDAA, and the herbicide pesticide mixing area;
  - Screen surficial, shallow, and deep soil samples for relevant contaminants of concern using a photoionization detector and headspace technique for VOCs, petroleum hydrocarbons using oleophilic dye tests, and for metals using an x-ray fluorescence (XRF) detector;
- Collect surficial, shallow, and deep soil samples based on soil screening and analyze for contaminants of concern as identified in each REC;
- Collect a soil sample from the herbicide pesticide mixing area and the Wullenweber CDAA based on proximity to suspected contaminants;
- Collect a surface water sample from the drainage ditch around the perimeter of the Wullenweber CDAA and analyze for specific herbicides of concern;
- Conduct a location survey of each analytical sample location using global positioning system (GPS);
- Based on the PCB building material sample results, collect ambient air samples in specific buildings for polychlorinated biphenyls (PCBs);
- Subcontract an asbestos investigation following the results of the ambient air sampling for mercury and PCBs; 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 is 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 #013	Surface Water and Sediment Sampling	MEDEP (DR#004)
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;
- Pacific Agriculture Laboratory, of Portland, Oregon;
- 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
SW	=Surface Water		

### 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 analconox 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
<b>Equipment Blank Water Matrix</b>					
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

<b>Table 5-1</b>					
<b>Absolute Resource Associates</b>					
<b>Summary of SOPs, Methods, Glassware and Preservative per Laboratory Analysis</b>					
<b>Test</b>	<b>Method</b>	<b>SOP No.</b>	<b>Matrix</b>	<b>Preferred Volume (mL or oz)</b>	<b>Container/Preservative</b>
Individual Metals	6010C	QA-5603	Water	125mL	P/4°C, HNO <sub>3</sub>
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
<b>Soil and Building Materials Matrix</b>					
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
Total Organic Carbon	SM 5310C	QA-5160	Soil	1-40mL Vial	G-Clear/4°C Sulfuric Acid
<b>Air Sample</b>					
Mercury	NIOSH Method 6009	6438	Air	6-liter	Summa canister
PCBs	Method TP-10A	2162	Air	6-liter	Summa canister
<b>Wipe Sample</b>					
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.

<b>Table 5-2 Field Quality Control Requirements</b>					
<b>QC Sample</b>	<b>No. of Soil Samples</b>	<b>No. of Water Samples</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>
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**

<b>TABLE 5-3 Summary of Data Quality Objectives</b>					
<b>Matrix</b>	<b>Parameters</b>	<b>Methods</b>	<b>Analytical Level <sup>1</sup></b>	<b>Data Evaluation Tier</b>	<b>Intended Data Use</b>
<b>Field Parameters</b>					
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	Lumex RA-915 Mercury Analyzer; SOP # 025	Level 1	NA	As appropriate to meet project goals

**TABLE 5-3**  
**Summary of Data Quality Objectives**

<b>Off-Site Laboratory Analysis</b>					
<b>Matrix</b>	<b>Parameters</b>	<b>Methods</b>	<b>Analytical Level <sup>1</sup></b>	<b>Data Evaluation Tier</b>	<b>Intended Data Use</b>
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	Organonitrogen Pesticides	USEPA Method 8270D	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	Total Organic Carbon	SM 5310C	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**.

<b>TABLE 5-4 State Criteria for Evaluating Data</b>	
<b>Medium</b>	<b>Criteria for Evaluation</b>
Surficial Soil (0-6" bgs)	Compare data with background concentrations and Appendix 2 Leaching to Groundwater RAGs
Shallow Soil (6"-2' bgs)	Compare data with background concentrations and Appendix 2 Leaching to Groundwater RAGs
Deep Soil Samples (>2')	Compare data with background concentrations and Appendix 2 Leaching to Groundwater RAGs
Lead Paint	Maine Solid Waste Management Rules, Chapter 424
Broken Fluorescent Bulbs	US EPA hazardous material characterization of mercury 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
Surface Water	RAGs for Groundwater by Residential Scenario, Table 3 and MEGs
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 and PCB Building Material	RAG Table 1: Soil Commercial Worker
bgs = below ground surface, TCLP= toxicity characterization leaching procedure , PCB= Polychlorinated biphenyl, ACM= Asbestos Containing Material, US EPA= United States Environmental Protection Agency, OSHA= Occupational Safety and Health Administration, MEDEP = Maine Department of Environmental Protection, RAGs = Remedial Action Guidelines For Soil Contaminated With Hazardous Substances, May 8, 2013, MEGs = Maximum Exposure Guidelines for Drinking Water, Maine Center for Disease Control and Prevention, October 19, 2012.	

CEG reviewed laboratory 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 the laboratory 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

CEG trained personnel shall wear a respirator and National Institute for Occupational Safety and Health (NIOSH) approved cartridge specifically for mercury vapor when setting the air sample canister within Building #85. CEG shall collect air samples within Building #85 for mercury vapor using NIOSH Method 6009. The result of the air sample will determine subsequent personal protective equipment required to conduct any future 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 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-site 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**. The samples will be collected from the former herbicide and pesticide mixing area, the non-vegetated Wullenweber CDAA area, and the floor drain and discharge area associated with Building #156. All samples collected for herbicides and pesticides will also be analyzed for total organic carbon.

## 5.8 Building Material Sampling

The building material samples within building #85 will be collected by trained CEG personnel equipped with a respirator and appropriate NIOSH approved cartridge specifically designed for mercury vapor (color code olive). 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 (100 cm X 100 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 samples shall be representative of caulk material used as a sealant around windows, doors, and/or foundations. Two concrete pads in and adjacent to Building #85 that formerly contained transformers, will also be sampled for PCBs. Three concrete pad samples will be collected from each pad. One representative building material 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 containing materials (ACM) 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 for ACM. These samples have been selected to re-confirm historical data that indicated these areas tested positive for asbestos. Screening for lead paint prior to selecting specific samples to be analyzed will be conducted using the on-site portable XRF or equivalent. Areas indicating the highest lead readings shall be sampled for confirmation.

<b>Table 5-6</b> Surficial and Shallow Soil Sample Collection and Laboratory Analysis							
Samples	Location	Pest	Herb	PCBs	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 overflow 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
BM-20 through BM-26	Concrete associated with transformer pad adjacent to Bldg #85 and within Bldg #85					✓		Determine if original transformer (circa 1960's) impacted the concrete pad with PCBs

### 5.9 Test Pit Sampling

CEG will supervise the excavation of approximately four test pits to investigate the presence and any impacts from the #2 fuel oil UST on the west side of Building #85 and up to two test pits to investigate the non-vegetated Wullenweber CDAA area. 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. If evidence of impacts are evident within the test pits excavated in the Wullenweber CDAA area additional samples will be collected for analysis of the appropriate contaminant of concern which could include VOCs, SVOCs, or metals. 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
TP-5 and TP-6	Random location within the non-vegetated CDAA	✓	✓			✓	If soil screening indicates contaminants beyond pesticides and herbicides as outlined in the surficial soil samples SS-1 and SS-2, additional deeper samples will be collected as well as for analyses of additional contaminants of concern.

### 5.10 Surface Water Sampling

A single surface water sample shall be collected at a point within the drainage swale that is down gradient of the Wullenweber CDAA and as far upgradient of the discharge to the ocean as feasible based on volume and flow of water at the time of the field work being conducted. The method of collecting the surface water sample shall follow CEG's SOP 013 and follow the intent of the dipping method for flowing water. The surface water sample and one duplicate sample shall be analyzed for specific herbicides known or suspected to have been applied at the Subject Property (2, 4-D; Bromocil; and Simazine). The sample shall be split homogeneously for method 8270D for the analyses of Bromocil and Simazine by Pacific Agriculture laboratory of Portland, Oregon and 2,4-D using method 8151A by Phoenix

Laboratories, a subcontractor of Absolute Resource Associates.

### **5.11 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.

**5.11 Sample Collection Summary**

<b>Table 5-9 Sample Collection Summary by Media</b>												
	VOCs	SVOCs	Pesticides	RCRA Metals	Herbicides	EPH	Lead	Mercury	TCLP Mercury	Mercury wipe	Asbestos	PCBs
<b>Project Soil Samples</b>	4	2	5	4	5	4	0	0	0	0	0	0
QC Soil Duplicates	1	1	1	1	1	1	0	0	0	0	0	0
Equipment Blank	1	1	1	1	1	1	0	0	0	0	0	0
Trip Blank	1	0	0	0	0	0	0	0	0	0	0	0
<b>Total Solid samples</b>	<b>7</b>	<b>4</b>	<b>6</b>	<b>6</b>	<b>6</b>	<b>6</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>
<b>Project Building Material (BM) Samples</b>	0	0	0	0	0	0	3	3	1	3	6	12
QC BM Duplicates	0	0	0	0	0	0	1	1	0	1	2	1
Equipment Blank	0	0	0	0	0	0	1	1	0	0	0	1
Trip Blank	0	0	0	0	0	0	0	0	0	0	0	0
<b>Total BM samples</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>5</b>	<b>5</b>	<b>1</b>	<b>4</b>	<b>8</b>	<b>14</b>
<b>Project Air Samples</b>	0	0	0	0	0	0	0	2*	0	0	0	1**
QC Duplicates	0	0	0	0	0	0	0	1*	0	0	0	1**
<b>Total Air Samples</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>3</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>2</b>
*-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 week of mobilization to the Site. Analytical data is anticipated from the laboratories 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 \$34,000.

## **APPENDIX**

Figure 1 Site Locus Map

Figure 2 Proposed Sampling Locations

Absolute Resource Associates Method Reporting Limits Table

SOP #024 Collecting a Wipe Sample

SOP #025 Operating a Mercury Vapor Analyzer

SOP # 026 For Sampling Porous Surfaces for Polychlorinated Biphenyls (PCBs)

Laboratory SOP # 627.8151 Air Analysis for Mercury

Laboratory SOP #6438 Air analysis for PCBs

Laboratory SOP # QA-5160 Soil Analysis for Total Organic Carbon

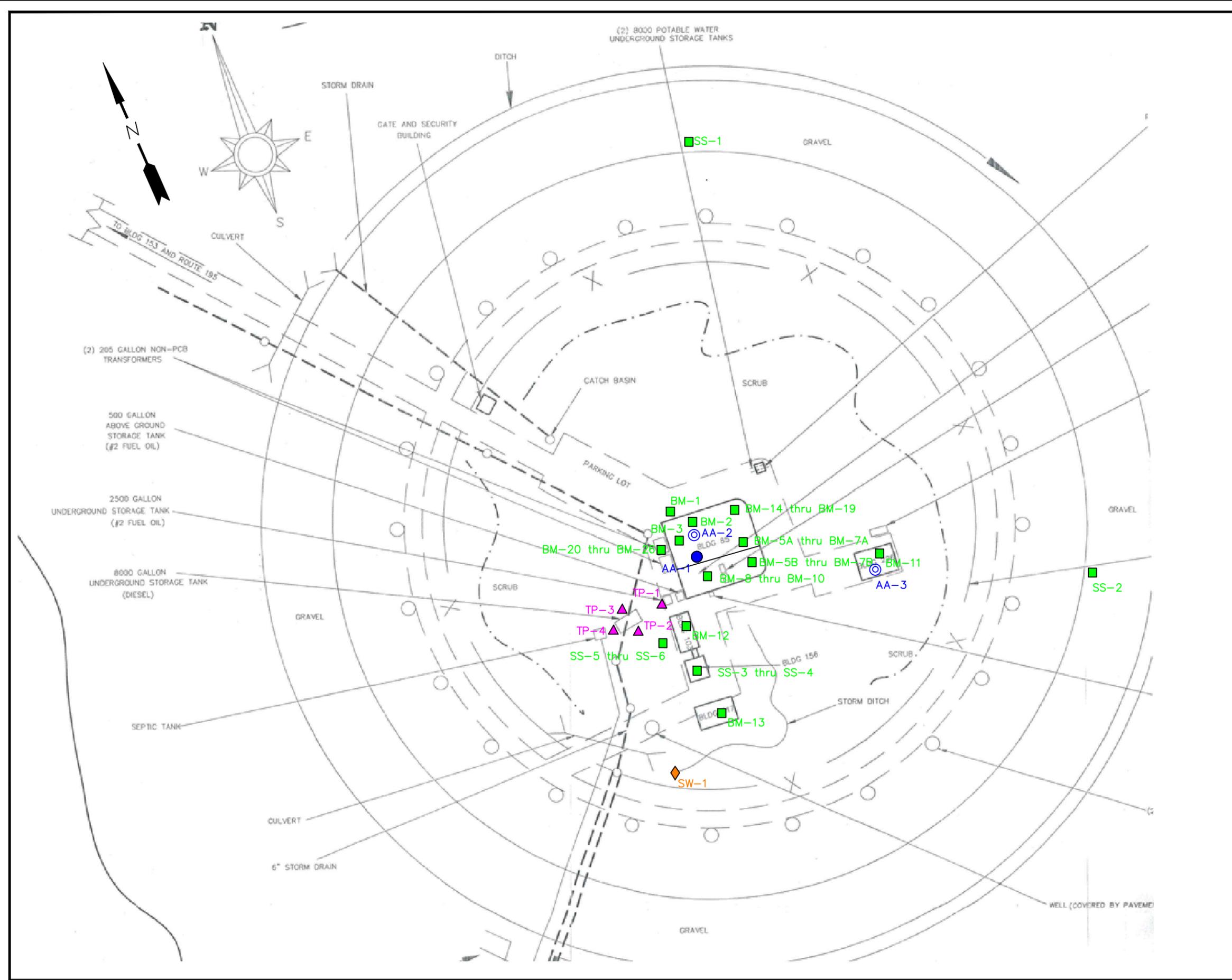
**Figure 1**  
**Locus Map**  
 Naval Security Group Activity Facility  
 Corea Village, Gouldsboro, Maine



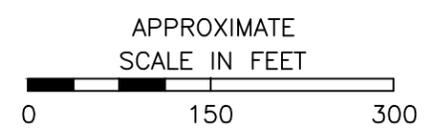
Data use subject to license.  
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MN (16.4° W)

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 0 1/4 1/2 3/4 1 1 1/4 1 1/2  
 Data Zoom 11-7



- LEGEND**
- ▲ TEST PIT
  - AMBIENT AIR SAMPLE FOR MERCURY
  - ⊙ OPTIONAL AIR SAMPLE FOR POLYCHLORINATED BIPHENYLS (PCBs)
  - SHALLOW SOIL SAMPLE OR BUILDING MATERIAL SAMPLE
  - ◆ SURFACE WATER SAMPLE



CLIENT: HANCOCK COUNTY PLANNING COMMISSION		
LOCATION: NAVAL SECURITY GROUP ACTIVITY GOULDSBORO, MAINE		
PM: DW	DETAILED: DW	PROJECT NO.: 1014-257-00
REV. NO.:	DRAWING DATE: 06/30/15	ACAD FILE: 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)
<b>Metals (Analytical Methods 6010/7000)</b>								
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
<b>Volatile Organic Compounds - VOCs (Analytical Method 8260)</b>								
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
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
CHLOROENZENE	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)

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

Analyte	CAS No.	SOIL (mg/kg)		GROUNDWATER (ug/l) 2013 RAGs or 2012 MEGs*	SOIL (mg/kg)		GROUNDWATER (ug/L)	
		2013 MEDEP REMEDIAL ACTION GUIDELINES (RAGs)			REPORTING LIMITS (mg/Kg)	MINIMUM DETECTION LIMITS (mg/Kg)	REPORTING LIMITS (ug/L)	MINIMUM DETECTION LIMITS (ug/L)
		RAG	Exposure Scenario*					
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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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)
		DIETHYLPHTHALATE	84-66-2		10,000	Residential	6,000	0.5
DI-N-BUTYLPHthalate	84-74-2	10,000	Residential	700	0.5	0.04	5	5
DI-N-OCTYLPHthalate	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
<b>Polychlorinated bihenyls - PCBs (Analytical Method 8082)</b>								
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
<b>Pesticides (Analytical Method 8081)</b>								
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
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
<b>Herbicides (Analytical Method 8151A)</b>								
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
<b>Volatile Petroleum Hydrocarbons - VPH (Analytical Method MADEPVPH-04-1.1)</b>								
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)
<b>Extractable Petroleum Hydrocarbons - EPH (Analytical Method MADEPEPH 2004-1.1)</b>								
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
<b>Notes:</b>								
* 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								

## Surface Wipe Sampling

SOP No. 024

CEG Company QAPP

Date: September 2015

Revision 0

Page: 1 of 3

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### INTRODUCTION

Surface wipe sampling is conducted to assess the presence of a contaminant on surfaces in the workplace that may lead to worker exposure. Surfaces contaminated with a hazardous liquid, particles, or dried residue may be contacted by workers, leading either to dermal exposure or transfer to foodstuffs and accidental ingestion. Settled dusts containing toxic material may be disturbed and re-suspended, resulting in inhalation exposure.

In instances where surface contamination is suspected and the employer has not required the use of effective PPE for workers in these areas, wipe sampling may be an effective means of documenting that a skin hazard exists. Wipe sampling can help establish that a significant amount of surface contamination is present in areas in which workers are not effectively protected by PPE. Wipe samples taken inside the sealing surface of "cleaned" respirators can establish the absence of an effective respiratory protection program.

In areas where exposures to toxic metals such as lead (Pb) occur, wipe sampling of settled dust can demonstrate that a reservoir for potential exposure exists; re-suspension of such settled dusts can lead to inhalation exposure. This is particularly true if improper housekeeping techniques are used, such as: dry sweeping; blowing off surfaces with compressed air; or using a shop vac instead of a HEPA-rated vacuum cleaner.

In break areas, the presence of surface contamination can lead to contamination of foodstuffs and hence, accidental ingestion of toxic material. The same is true for contamination on drinking fountains. Contamination found on the clean side of a shower or locker area could suggest the potential for take-home contamination, resulting in additional toxic exposures occurring while away from work. All of these types of wipe sampling results can be used to support violations of the housekeeping requirements found in the expanded health standards in Subpart Z of 29 CFR 1910.

In many instances, several wipe samples taken in an area suspected of being contaminated may be useful. For example, some surfaces which would be expected to be contaminated with chemicals because of airborne deposition of a non-volatile chemical may actually be relatively free of surface contamination because of frequent contact of the surface by workers (i.e., frequently contacted surfaces may be expected to be "clean" because of contaminant removal by frequent worker contact). Wipe

samples of frequently contacted surfaces in conjunction with less frequently contacted surfaces in the same vicinity can be useful to establish the likelihood that skin exposure is occurring in "clean" areas in which PPE is not being used, or is being improperly used.

## **SURFACE WIPE SAMPLING**

The most common surface testing technique is surface wipe sampling. Frequently, the wipe is dipped in distilled water or other suitable solvent prior to wiping the surface of interest. This technique facilitates transfer of the contaminant from the surface to the wipe. It is best to use a minimum of water/solvent on the wipe so that all of the water/solvent will be picked up by the wipe and not left behind on the sampled surface.

The percent recovery of the contaminant of interest from the sampled surface may vary with the characteristics of the surface sampled (e.g., rough or smooth), the solvent used, and the technique of the person collecting the sample. Consequently, surface wipe sampling may be only semi-quantitative. No OSHA standards currently specify acceptable surface limits. Results of surface wipe sampling are used qualitatively to support alleged violations of housekeeping standards and requirements for cleanliness of PPE.

Templates may be used to define a relatively constant surface area for obtaining a wipe sample, but are not always helpful. Templates can only be used on flat surfaces, and they can cause cross contamination if the template is not thoroughly cleaned between each use. Constructing single-use 10-cm x 10-cm templates is recommended (e.g., using cardstock or file folders). The CSHO may want to sample a much larger surface area than the area covered by a template (e.g., the CSHO may want to determine the cleanliness of a lunch table or other large surface area). In all cases, the CSHO should measure the dimensions of the area being sampled and record this value on the OSHA Information System (OIS) sampling worksheet because the mass amount of chemical measured by the laboratory will be used to determine the mass per unit area for the wipe sample.

## **PROCEDURES FOR COLLECTING WIPE SAMPLES**

Preloading a group of vials with sampling filters is a convenient method to carry the sample media to the worksite. Note: Smear tabs should be inserted with the tab end out. Clean disposable gloves should be worn when handling the filters and smear tabs. The gloves should not be powdered.

The following are general recommendations for taking wipe samples. Consult the CSI files for more specific instructions.

- Record each location where a wipe sample was taken. Photographs, sketches, diagrams and other means of noting sampling locations are helpful.

- A new set of clean, disposable, powder-free gloves should be used for each sample to avoid contamination of the filter by previous samples (and the possibility of false positives) and to prevent contact with the substance.
- Withdraw the filter from the vial with your fingers or clean tweezers. If a damp wipe sample is desired, moisten the filter with distilled water or other solvent as recommended. Note: For skin sampling use only distilled water. Other solvents may be appropriate for wiping surfaces depending upon the type of chemical being sampled.
- Depending on the purpose of the sample, it may be useful to determine the concentration of contamination (e.g., in micrograms of agent per area). For these samples, it is necessary to record the area of the surface wiped (e.g., 100 cm<sup>2</sup>).
- Firm pressure should be applied when wiping.
- Using the filter, wipe an area about 100 cm<sup>2</sup>, rubbing the entire area side to side, then up and down. In many cases (such as knobs and levers) it may not be possible to wipe 100 cm<sup>2</sup>. Where precise determination of the contaminant loading (concentration) is desired, prepare single-use 10-cm x 10-cm templates from cardstock or file folders.
- Place the filter in a sample vial, cap and number it, and note the number at the sample location. Include notes which will provide any additional relevant details regarding the nature of the sample (e.g., "Fred Worker's respirator, inside"; "Lunch table").
  - At least one blank filter treated in the same fashion, but without wiping, should be submitted for each sampled area.
  - Some substances (e.g., benzidine, hexavalent chromium, and 4,4'-methylenedianiline) are unstable and may require a solution to be added to the vial as soon as the wipe sample is placed in the vial or may require other special sample handling. If such instability is suspected, check the [CSI](#) file for sample handling instructions or contact the [SLTC](#) for guidance.
  - Submit the samples, each sealed with a Form OSHA-21, and in accord with any special procedures located in OTM Section II Chapter 4 (Sample Shipping and Handling), to the SLTC. Properly document the samples by completing the OIS sampling worksheet.



***JEROME*<sup>®</sup> 431-X<sup>™</sup>**  
**MERCURY VAPOR ANALYZER**  
**OPERATION MANUAL**

March 2005

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700-0046-B1

# JEROME 431-X Mercury Vapor Analyzer Operation Manual



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## 1. FOR THOSE WHO CAN'T READ THE WHOLE MANUAL NOW

This manual contains details that will optimize the results and the life of your instrument. Read and refer to the manual for complete details on operation, maintenance and troubleshooting, special voltage inputs and data output.

The Jerome 431-X is easy to operate and ready for use upon receipt from the factory.

- Remove the instrument from the packing material.

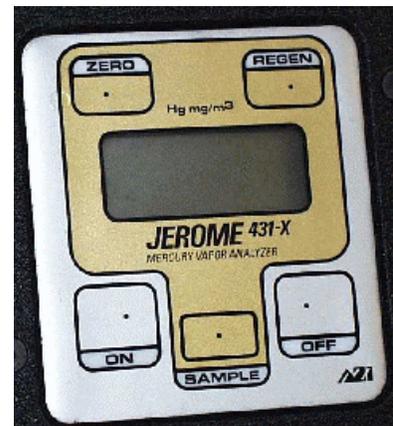
**Retain all packaging materials for any future shipment of the instrument.**

**If the instrument is returned to AZI for any reason, it must be placed in the original packaging materials that have been tested and proven to be effective protection during shipment.**

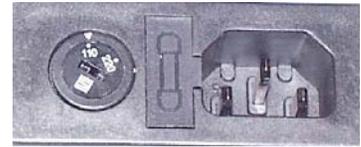
- Call AZI Customer Service at 800-528-7411 or 602-470-1414 for Return Material Authorization (RMA) information prior to returning a unit.
- For all shipments, boxes and packing materials are available from AZI.
- Pack the Jerome instrument only in a Jerome shipping container.

**AZI WILL NOT BE RESPONSIBLE FOR SHIPPING DAMAGE. IF YOU RETURN THE INSTRUMENT IMPROPERLY PACKAGED OR SHIPPED, YOU SHOULD INSURE IT FOR FULL VALUE.**

- Check for any damage and confirm receipt of all parts on your packing list. Contact Arizona Instrument Customer Service at (800) 528-7411 or (602) 470-1414 if you have any questions.
- Press the ON button. The display should read 000 in less than one second.
  - A LO BAT message appears briefly in the upper left corner.
  - If the LO BAT message persists, recharge the battery. See page 17.



- Check the voltage setting (110 or 220 VAC) on the back of the instrument. Ensure that it is set to the correct voltage. If the pointer is not aligned to the local voltage, turn the selector to point to the correct voltage.



- Perform a sensor regeneration by following these steps:
  - Connect the line cord between the connector on the back of the 431-X and an AC power outlet.
  - Press the ON switch and then press the REGEN button.
    - ◆ The instrument will begin a 10 minute regeneration cycle, indicated by .H.H.H flashing on the display. **Do not interrupt this cycle.** For a complete description of this process, see page 12.
    - ◆ If any error message, such as .P.P.P, appears on the display, see the “Troubleshooting” section beginning on page 24.
- When regeneration is complete, zero the sensor by pressing the ZERO button and turning the zero adjust screw, located under the handle, until the display reads 0.
- The instrument is now ready to sample.
- To ensure the input to the instrument contains no Mercury Vapor or mercaptans, use a Zero Air Filter, AZI P/N Z2600 3905. The Zero Air Filter cleans the air sample and should produce sample readings of less than 0.003 mg/m<sup>3</sup>. Therefore, use the filter to:
  - Equilibrate the instrument to temperatures that are higher or lower than the instrument. Sample with filter installed until the reading is below 0.003 mg/m<sup>3</sup>.
  - Identify contamination within the unit.
  - Confirm the presence of Mercury Vapor when readings are elevated. Install filter and verify that the readings go down with filter installed.
- When the instrument measures Mercury Vapor, the zero display will be replaced with a value.

### CAUTION

**Do not adjust the ZERO after the instrument has measured Mercury Vapor or before the next regeneration. (Occasionally the display may drop to .L.L.L (indicating low) between the initial zeroing and the first sample. It is OK to readjust the ZERO if the instrument has not measured Mercury Vapor.)**

- The instrument is designed for ambient air monitoring. **DO NOT allow the probe or the instrument’s intake to be exposed to any liquid.**
- The instrument is not explosion proof.
- Press the SAMPLE button to start a 10 second sampling cycle.
- Perform sensor regeneration after each day’s testing.
- Perform another sensor regeneration and re-zero the instrument before each day’s use.
- Perform sensor regeneration after 30 days of storage or inactivity.

**Call AZI Customer Service, at (800) 528-7411 from the United States and Canada or (602) 470-1414 if you have any questions. If you prefer, you may send e-mail to [support@azic.com](mailto:support@azic.com)**

## 2. INTRODUCTION

The Jerome 431-X Mercury Vapor Analyzer is an ambient air analyzer with a range of 0.001 to .999 milligrams of mercury vapor per cubic meter ( $\text{mg}/\text{m}^3$  Hg).



### CAUTION:

The Jerome 431-X is for vapor use only.  
**DO NOT** allow the probe or the instrument's intake  
to be exposed to any liquid, dust  
or other foreign material.



The 431-X is designed to be easy to operate for quick and accurate analysis of mercury vapor levels. It has few maintenance requirements. However, please take a moment to read this manual before attempting operation. If you have any questions about your application or operation, please call AZI Customer Service at (800) 528-7411 or (602) 470-1414 or e-mail [support@azic.com](mailto:support@azic.com) for assistance.

### 431-X Features

- Automatic sensor regeneration when equipped with the communications option and used with the Jerome Communication Software (JCS) program and the Jerome data logger.
- Regulated film heat voltage during sensor regeneration. This allows the sensor to clean properly with voltages from 100-130 VAC (or 200-260 VAC).
- Survey mode can be locked in.
- DIP switch setting can change the digital meter readings from  $\text{mg}/\text{m}^3$  Hg to nanograms (ng) of Hg (see page 22).
- The Jerome 431-X can be operated from 100-130 or 200-260 VAC. To change the default voltage range, refer to Setting the Input Voltage, page 21.

### Accessories and Maintenance Parts

The Accessories and optional items available to support the 431-X are listed and pictured beginning on page 30.

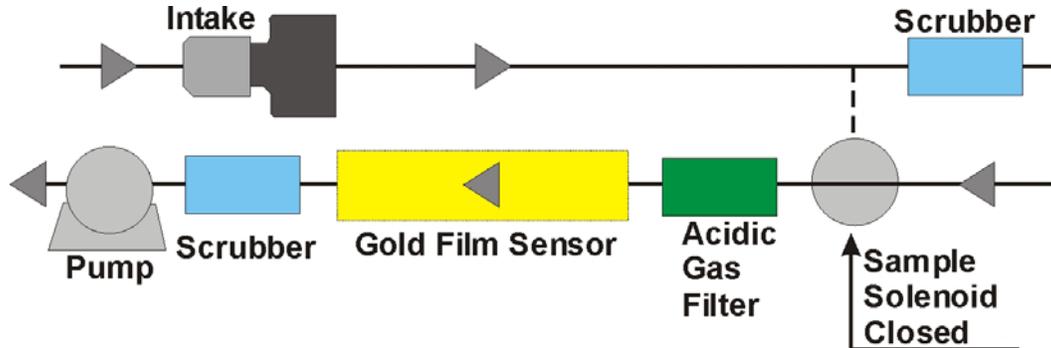
## Applications

- Ambient air analysis
- Odor nuisance monitoring
- Regulatory compliance
- Control room corrosion monitoring
- Quality control
- Scrubber efficiency testing
- Accuracy check for other Mercury Vapor monitors and control systems
- Mercury Vapor source detection
- Leak detection
- The Jerome 431-X can be operated from 100-120 or 200-240 VAC. To change the default voltage range, See “Setting the Input Voltage” on page 21.

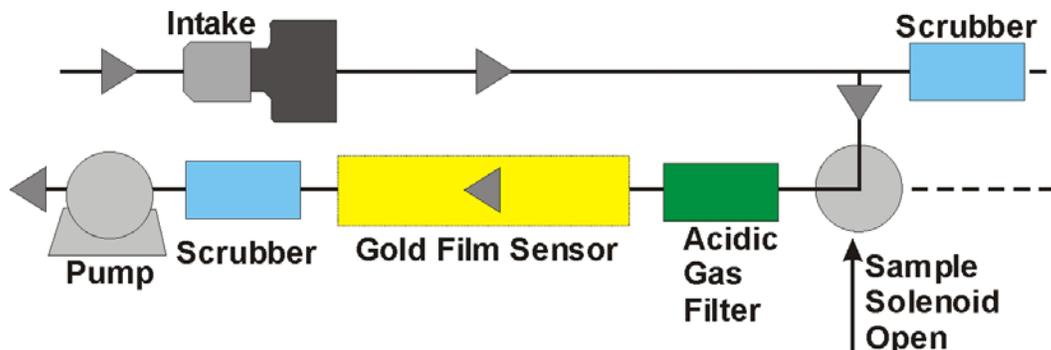
## 3. PRINCIPLE OF OPERATION

A thin gold film, in the presence of Mercury Vapor, undergoes an increase in electrical resistance proportional to the mass of Mercury Vapor in the sample.

When the SAMPLE button is pressed, an internal pump pulls ambient air through a scrubber filter and into the flow system.



After 2 seconds, the sample solenoid bypass opens, closing off the scrubber filter from the flow system.



The sample air passes through a filter (removing any acidic gases which interfere with the sensor's response to mercury) and is drawn over the gold film sensor. The sensor absorbs the Mercury Vapor. Nine seconds after starting, the sample solenoid bypass closes and the remainder of the sample is drawn through the scrubber filter and the flow system. The instrument determines the amount absorbed and displays the measured concentration on the digital meter in milligrams per cubic meter ( $\text{mg}/\text{m}^3$ ) of mercury. An internal DIP switch can be used to change the digital meter display from  $\text{mg}/\text{m}^3$  to nanograms of mercury (see page 22).

The instrument's microprocessor automatically re-zeroes the digital meter at the start of each sample cycle and freezes the meter reading until the next sample cycle is activated, thus eliminating drift between samples.

During the sample mode cycle, bars on the LCD represent the percentage of sensor saturation. Depending on the concentrations, approximately sixty-five samples containing  $0.1 \text{ mg}/\text{m}^3 \text{ Hg}$  may be taken before the sensor reaches saturation. After absorbing approximately 500 nanograms of mercury, the sensor becomes saturated and needs to be cleaned. This is accomplished by a manually activated 10-minute heat cycle, or sensor regeneration that burns the mercury from the sensor. This mercury is absorbed on internal filters to prevent any external contamination. The solenoid bypass closes during the sensor regeneration cycle, causing the air to pass through the scrubber filter, providing clean air for the regeneration process. The flow system's final scrubber prevents contamination to the atmosphere from the desorbed mercury.

The heat generated during the regeneration may cause some low level thermal drift. To ensure maximum sample accuracy, wait 30 minutes after regeneration before zeroing and using the instrument.

## **Zero Air Filter**

The Zero Air Filter removes mercury vapor, mercaptans, and mercury vapor from the air sample. Readings with the filter installed should be near zero.

Because air that is cooler than the instrument will cause low readings and warmer air will cause higher readings, the Zero Air Filter should be used to equilibrate the unit to ambient air. Continuous sampling with clean air will not cause saturation of the gold film sensor but will equalize temperatures faster to allow accurate analysis to begin sooner.

The Zero Air Filter can also be used to identify contamination within the instrument. If the readings do not reduce to near zero with the filter installed, contamination should be suspected. If the readings do drop to near zero with the filter installed but elevate with the filter removed, the presence of Mercury Vapor at the sampled location is confirmed.

For more information on the use of the Zero Air Filter, contact customer service at 1-800-528-7411, 1-602-470-1414, or visit our web site at <http://www.azic.com>.

## 4. INSTRUMENT OPERATION

### LCD Codes

LCD CODE	EXPLANATION
000	Ready to sample
.000	No Mercury Vapor reading
00.0	No Mercury Vapor reading, display in nanograms
.8.8.8	Sensor saturated-regeneration needed (refer to page 12)
.H.H.H	Sensor regeneration in progress (.H.H.H flashes)
.L.L.L	Re-zero needed (refer to page 13)
.P.P.P	Power cord required or low line power, <100 VAC (or 200 VAC) (see pages 16 and 17, Changing the Fuse, if .P.P.P remains on after the cord is connected.)
.H.L.P	High line power, greater then 130 AVC in 110 operation or 260 VAC in 220 operation
.LO BAT	Recharge batteries (refer to page 17)
.E.E.E	Same as LO BAT, automatically shuts off
.HL	Very high concentration has been detected. Refer to your safety policy for additional direction to confirm the concentrations."
<b>DURING SAMPLING</b>	
.-	0-25% sensor saturation
.--	25-50% sensor saturation
.-.-	50-75% sensor saturation
-.---	75-100% sensor saturation
<b>DURING SAMPLING, USING SURVEY MODE</b>	
-	Survey sampling (minus sign flashes continuously)
<b>WHEN ZERO IS DEPRESSED</b>	<b>Adjust to 0 <u>only</u> after sensor regeneration. It is normal for the display to read H after sampling has started.</b>
0	Zero, ready to sample
H	High, turn Zero potentiometer counterclockwise
L	Low, turn Zero potentiometer clockwise

## Daily Operations

Before each day's use of the Jerome 431-X, perform the following steps to verify proper instrument operation:

- Press the power ON button.
  - The digital meter displays 000.
    - ◆ (Disregard the digital meter's initial momentary reading.)
    - ◆ Recharge or replace the battery pack if the LO BAT indicator REMAINS ON. Refer to “Charging Batteries” on page 17 and/or “Replacing the Battery Pack” on page 21.
    - ◆ To ensure the instrument's electronics have stabilized, allow a 1-minute warm up before beginning the next step.
- Use the Zero Air Filter to equilibrate the instrument to ambient air temperature.
  - Install the Zero Air Filter in the instrument's intake.
  - Sample continuously until the readings stabilize.
- Perform sensor regeneration. Refer to page 12 for the procedure.
- Thirty minutes after sensor regeneration is complete, zero the instrument.



**NOTE:** For maximum accuracy, such as when testing with the Functional Test Kit, wait 30 minutes after the sensor regeneration cycle to re-zero the unit. For immediate use, the unit can be re-zeroed immediately after sensor regeneration.

- Press the SAMPLE button.
  - During the sample cycle, the digital meter displays bars (-, --, or ---) to indicate the amount of sensor saturation.
- At the end of the sampling cycle, read the digital meter.
  - The number shown on the digital meter is the Mercury Vapor concentration in mg/m<sup>3</sup>.
  - This value remains on the display until the next sample is taken.
  - The digital meter automatically zeroes at the start of each sample.
- At the end of each day's use, perform sensor regeneration as described in the next section.

**DO NOT ALLOW MERCURY VAPOR TO STAY ON THE GOLD FILM SENSOR OVERNIGHT.**

## Sensor Regeneration

Sensor regeneration is needed to clear the 431-X sensor of any accumulated Mercury Vapor and to prolong the life of the sensor. This simple procedure should be done:

- At the beginning of the day on which the instrument is to be used.
- During the day when the sensor becomes saturated.
- At the end of the day before storing the instrument.
- At a minimum of 30 day intervals while the instrument is in storage.



### CAUTION:

Ensure the voltage selector on the back of the instrument, near the power cord inlet connector, points to the local AC power value. See “Setting the Input Voltage” on page 21.

To clean and protect the sensor, the supplied AC power must be 100 to 120 VAC or 220 to 240 VAC, depending on the available power source.

Once sensor regeneration is initiated, DO NOT interrupt the cycle.



- Attach the power cord to the 431-X and plug it into AC power. AC power is required to thermally regenerate the sensor.
- Press the power ON button.
- Press the REGEN button.
  - The digital meter flashes .H.H.H for the duration of the 10-minute cycle and displays .0.0.0 when the cycle is completed.

### DO NOT INTERRUPT THIS CYCLE.

Wait until the cycle is completed before continuing with the next step.

- A minimum 30-minute wait after the sensor regeneration cycle is complete ensures maximum sample accuracy. However, the unit can be used immediately following the sensor regeneration if necessary. When the sensor regeneration is complete, press ZERO and adjust the ZERO ADJUST pot until 0 appears on the display. Install the zero air filter in the intake and take several samples or lock the instrument into survey mode (see page 15). After approximately one minute, stop sampling and check the ZERO. Adjust to 0. Repeat sampling through the zero air filter until reading remains on 0.

**NOTE:** The digital meter will read .P.P.P after REGEN is activated if the power cord is not plugged in or if the instrument's fuse needs to be replaced. Connect the power cord, or if necessary, replace the fuse. See “Changing the Fuse” on page 22.

## Zero Adjust

- To ensure air entering the instrument is clean, install the zero air filter in the instrument's intake and sample until the readings stabilize.
- While pressing the ZERO button, turn the ZERO ADJUST potentiometer (shown at right) using the trimmer tool until the digital meter reads 0.
  - If the LCD reads H, turn the ZERO ADJUST counter-clockwise;
  - If the LCD reads L, turn the ZERO ADJUST clockwise.



**NOTE:** A minimum 30-minute wait after the sensor regeneration cycle is complete ensures maximum sample accuracy. The unit can be used immediately following the sensor regeneration if necessary. When the sensor regeneration is complete, press ZERO and adjust the ZERO ADJUST pot until 0 appears on the display. Install the zero air filter in the intake and take several samples or lock the instrument into survey mode (see page 15). After approximately one minute, stop sampling and check the ZERO. Adjust to 0 if necessary. Repeat sampling through the zero air filter until sensor remains on 0.

**NOTE:** When ZERO is pressed, and depending upon internal configuration, a number between 00 and 100 may appear on the display instead of H, L, or O. If the instrument is configured with an Option Board, see APPENDIX E - JEROME 431-X OPTION BOARD beginning on page 58.

### CAUTION:

**Do not turn the ZERO ADJUST potentiometer between samples.**

Turn the ZERO ADJUST only after a sensor regeneration cycle otherwise invalid readings will result.

- Press the power OFF button and disconnect the power cord.
- The Jerome 431-X is ready for sampling.

### CAUTION:

**The Jerome 431-X is intended for vapor use only. DO NOT allow the probe or the instrument's intake to be exposed to liquids, dust or other foreign material. Moisture or liquids drawn into the instrument can damage the sensor and flow system.**

## Sample Mode

This mode, used for standard operation, produces optimum accuracy (+/-5% at 0.100 mg/m<sup>3</sup> Hg) with the Jerome 431-X.

- Press the power ON button.
  - The digital meter displays 000. If the unit is set to display in ng, the digital meter displays 00.0. (Disregard the digital meter's initial momentary readings.) Recharge or replace the battery pack if the LO BAT indicator REMAINS ON. Refer to pages 17 and/or 21 for the procedure.
- To ensure the instrument's electronics have stabilized, allow a 1 minute warm up before beginning the next step.
- Press the SAMPLE button.
  - During the sampling cycle, the bar (or bars) shown on the digital display indicate the current percentage of sensor saturation. (Refer to Meter Display Codes, page 10, for code descriptions.)
  - The bar (or bars) flash after 2 seconds and again after an additional 7 seconds. This flashing signals the opening and closing of the solenoid sample bypass. (See the Principles of Operation on page 8 for details.)
- At the end of the 12 second cycle, read the digital meter.
  - The number shown on the digital meter is the mercury concentration in mg/m<sup>3</sup> (or ng). This value remains displayed until the next sample is taken. The digital meter automatically zeroes at the start of each sample.
- When the sensor is completely saturated, the digital meter displays .8.8.8 instead of a value. No further operation is possible until a sensor regeneration is performed. (Refer to page 12 for the Sensor Regeneration procedure.)
- Press the power OFF button when not in use. Install the zero air filter in the instrument intake during storage.

## Sampling Notes

The Jerome 431-X is intended for vapor use only. **DO NOT** allow the probe or the instrument's intake to come in contact with liquids, dust or other foreign material. Moisture or liquids drawn into the instrument can damage the sensor and flow system.

The Jerome 431-X operates a minimum of 6 hours on a fully charged battery.

Use the probe (AZI P/N1400-2002) to locate mercury vapor in hard to reach places. Plug the probe directly into the instrument's intake.

## Survey Mode

The survey mode takes samples every 3 seconds automatically. Use this mode to locate mercury spills or to assess areas of potentially high mercury concentrations. Sampling in the survey mode is not as accurate. Due to the decreased sample volume, the accuracy of the instrument is reduced to +/- 20% @ .100 mg/m<sup>3</sup>.

- Press the power ON button.
  - The digital meter displays 000. If the unit is set to display in ng, the digital meter displays 00.0. (Disregard the digital meter's initial momentary readings.) Recharge or replace the battery pack if the LO BAT indicator REMAINS ON. Refer to pages 17 and/or 21 for the procedure.
  - To ensure the instrument's electronics have stabilized, allow a 1 minute warm up before beginning the next step.
- Press and **hold** the SAMPLE button.
  - The instrument takes a normal 12 second sample, displays the concentration at the end of the cycle and then goes into the survey mode sampling every 3 seconds. The display flashes the measured concentrations at the end of each 3 second sample cycle.
- When you are finished surveying, **release** the SAMPLE button.
  - The final survey value remains displayed until the next sample is taken.

NOTE: Approximately 65 samples at .1 mg/m<sup>3</sup> may be taken before a sensor regeneration is required.

- To lock the instrument in a survey mode:
  - Hold the SAMPLE button down until the sensor status indicator bar(s) "\_ " begins flashing on the display.
  - Press the ZERO button, then release the SAMPLE button.
  - The pump should continue to run and the display should update every 3 seconds.
  - The instrument remains in the survey mode until one of the following occurs:
    - ◆ The sensor is saturated
    - ◆ A LO BAT (low battery) signal is encountered
    - ◆ An HL (high mercury level) is encountered
    - ◆ The instrument is turned OFF.

Press the power OFF button when not in use.

## Operating on AC Power or Generator

- For stationary use, the 431-X may be operated on AC power.
  - Operating the instrument on AC power at all times eliminates the need for the battery pack and its necessary maintenance.
  - The battery may be unplugged or removed completely whenever the instrument is operating on AC power.
- When a generator is used to power the Jerome 431-X, a high quality line conditioner or voltage regulator is required to ensure a pure sine wave and regulated voltage is applied to the instrument. The gold film sensor may be damaged by voltage that varies in amplitude or by surges, spikes, and/or noise on the power line. **This is especially true during the sensor regeneration.**

## Operating on Internal Battery Power

For portable use, the 431-X may be operated on Battery power.

- When you operate the instrument on battery power, please be aware of the following:
  - A fully charged battery pack, AZI P/N Z4000-0907, provides power for a minimum of six (6) hours of operation.
  - For operating more than six (6) hours, an extra fully charged battery pack is needed.
  - Complete battery recharging takes 14 hours. Refer to Charging Batteries on page 17.
  - The 431-X uses a rechargeable Nickel Cadmium (NiCad ) battery. Dispose of worn-out batteries properly when you are replacing the battery pack.

## External battery power

A special version of the Jerome 431-X and a DC Power Kit are available to operate the instrument from a secondary DC source. The source may be a car/truck battery or a storage cell used in conjunction with solar panels.

Call AZI Customer Service at 800-528-7411, 602-470-1414, or e-mail [support@azic.com](mailto:support@azic.com) for additional information.

## Charging Batteries

- Press the power OFF button.
- Connect the AC power cord between the 431-X power receptacle and an AC power source.
  - Complete battery recharging takes 14 hours.
  - The 431-X contains a trickle charger so it may be continually plugged into an AC power source without damaging the battery pack.
- The battery pack may be charged outside the instrument with an optional AZI IDC Battery Charger. (AZI P/N 4000-1011, for 115 VAC, P/N 4000-1012, for 230 VAC)

## Obtaining Maximum Battery Life

There are certain inherent limitations to NiCad batteries. The primary limitation is a memory effect that occurs when the batteries are partially discharged and then recharged, repeatedly. This memory leads to a drastic reduction in the usable battery life. To prevent this memory effect, periodically allow the battery pack to discharge completely, and then recharge the battery pack.

- To obtain maximum battery life, follow these three (3) steps:
  - At least once a month wait until LO BAT appears on the digital meter before recharging the battery pack.
  - Charge the battery pack when the LO BAT indicator comes on. Excessive discharge can damage the battery pack.
  - Before storing the instrument verify the power is OFF.
- When the batteries fail to hold a charge, the battery pack should be replaced.
  - Battery life under normal usage is approximately 1 year, depending on the number of charge and discharge cycles.

## 5. MAINTENANCE

### Preventive Maintenance Calendar

To keep the Jerome 431-X operating at peak performance, follow the maintenance schedule below as a guide. Since maintenance is more a function of application and amount of use rather than time, your requirements may be different from the listed schedule. Call AZI Customer Service at 800-528-7411, 602-470-1414, or e-mail support@azic.com for additional guidance for your environment and operation.

PART/COMPONENT	MAINTENANCE CYCLE	REFER TO PAGE
Charge batteries	At least once per month, after 1 month's storage, or when LO BAT appears	Page 17
Change .25 inch fritware	Weekly or as needed	Page 19
Change internal filters and tubing <sup>1</sup>	After 6 months of use or as needed	Page 20
Replace zero air filter <sup>2</sup>	Annually	
Factory calibration	Annually	Page 23
Calibration check	Monthly or as needed	Appendix A, Page 37
Replace batteries	Annually or as needed. The battery pack contains NiCad batteries. Dispose of properly.	Page 21

**NOTE:** Install the zero air filter into the instrument's intake during storage.

<sup>1</sup> C/M filters contain Mallcosorb™. For safety information see the supplier's Material Safety Data Sheet (MSDS) or call AZI Customer Service at 800-528-7411, 601-470-1414, or e-mail support@azic.com for a copy. Dispose of all filters properly.

<sup>2</sup> Zero air filters and scrubber filters contain Resisorb™. For safety information see the supplier's Material Safety Data Sheet (MSDS) or call AZI Customer Service at 800-528-7411, 601-470-1414, or e-mail support@azic.com for a copy. Dispose of all filters properly.

## Flow System

The Jerome 431-X's flow system is the crucial link between the sensor and the sample. For the instrument to perform correctly, the flow system must be properly maintained. The user maintainable components of this system are the intake filter (.25 inch fritware), a C/M Filter, two scrubber filters and connecting tubing.

Check the Preventive Maintenance Calendar on page 18, for a suggested schedule for changing fritware and scrubber filters. The Tygon™ tubing in the system must be free of crimps for proper flow.

Part	Part Number
Scrubber Filter	Z2600 3930
C/M Filter	Z2600 3928
.25 inch Fritware Filter	2600 3039
Tygon™ Tubing - 1/8" I.D. (1')	2500 3001

### .25 inch Fritware Filter

Replace the .25 inch fritware filter once each week or as needed. In dusty environments, the fritware filter may need to be replaced as often as once a day. Replacement .25-inch fritware filters are available from AZI, Consumable Sales at 800-528-7411 or 602-470-1414.

- Unscrew and remove the intake.
- Push the old fritware filter disc out of the intake with your trimmer tool.
- Avoid touching the new fritware disc with fingers. Use tweezers to insert the new fritware.
- Use the blunt end of the trimmer tool to seat the fritware disc firmly against the inner ledge of the intake.
- Screw the intake back on the Jerome 431-X.



### CAUTION:

**The stem coming from the instrument onto which the outer intake housing is attached must be securely held in place. If loose, the tubing inside the instrument can become twisted when the intake housing is replaced. It may be necessary to open the instrument and tighten the hold-down nuts inside the instrument. Call AZI Customer Service at 800-528-7411, 601-470-1414, or e-mail [support@azic.com](mailto:support@azic.com) if you have any questions**



## Internal Filters

- Replace the internal filters after six (6) months of use, or as needed.
- Press the power OFF button and unplug the power cord.
- Remove the two (2) side screws from the intake end of the instrument and open the case.
- Carefully disconnect the Tygon tubing from both ends of the filters and discard the old filters.

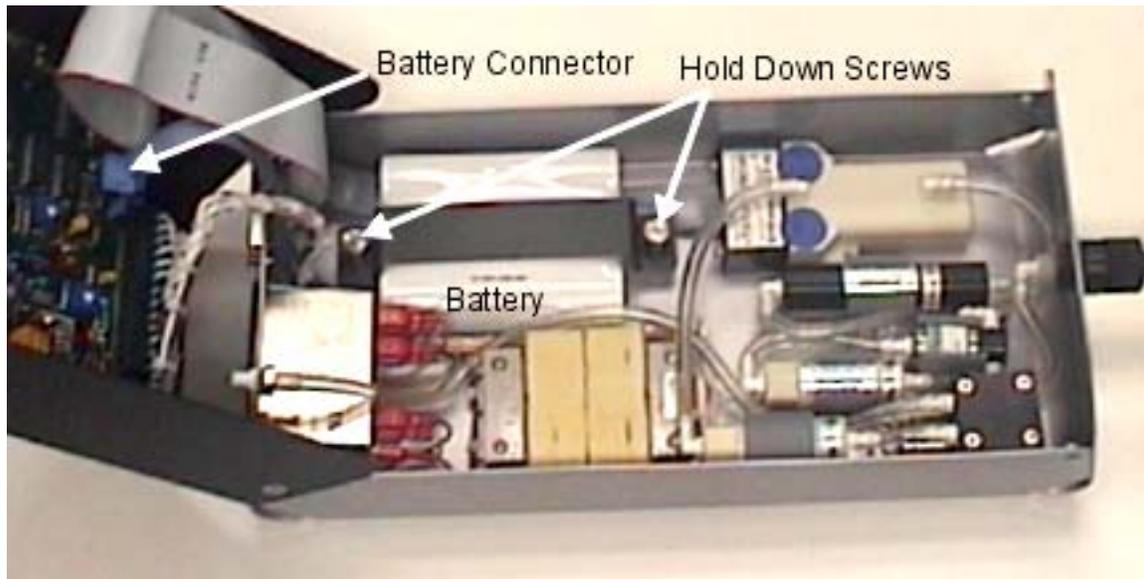


### CAUTION:

**Filters contain either Mallcosorb or Resisorb. Used filters will contain trace amounts of Mercury also. Use proper methods when disposing of used filters. Call AZI Customer Service at 800-528-7411, 601-470-1414, or e-mail [support@azic.com](mailto:support@azic.com) for a copy of the Resisorb MSDS or for other questions.**

- Connect the new filters to the Tygon tubing, ensuring all straight hose barbs point toward the intake/pump corner of the case and elbow hose barbs point toward the sensor housing as shown in the illustration.
  - Push the Tygon as far as it will go onto the filter fittings.
- Push the filters into the mounting clips.
- Remove any crimps or twists in the tubing and ensure that tubing connections are secure. If the tubing is loose, readings may not be accurate. Replace any tubing that has deteriorated due to heat and/or age.
- Close the case and replace the screws.
- Dispose of all filters in accordance with state and federal environmental regulations.

## Replacing the Battery Pack

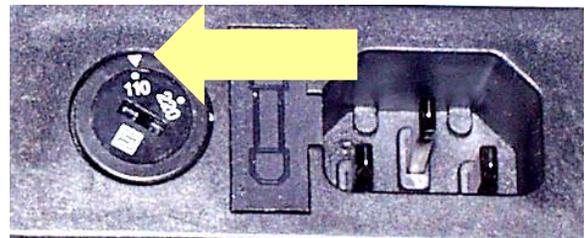


- Press the power OFF button.
- Unplug the power cord.
- Remove the two (2) side screws from the intake end of the instrument and open the case lid.
- Disconnect the battery connector from the board.
- Loosen the two (2) captive screws holding the battery bracket and remove the bracket.
- Remove the old battery pack and replace with a new battery pack.
- Replace the battery bracket and tighten the captive screws.
- Connect the new battery connector to the board.
- Close the case and replace the two (2) side screws.
- Dispose of the old NiCad battery in accordance with state and federal regulations.

## Setting the Input Voltage and Frequency

Instruments are factory set and calibrated to use the power setting requested on the order. However, the voltage setting is easily changed to use either 110 or 220 VAC.

- Ensure the instrument is turned OFF and unplugged.
- Locate the voltage selector on the rear of the instrument.
- Insert a small screwdriver in the voltage selector slot and turn the selector until the arrow points toward your setting choice and a click is heard.



- Remove the two screws toward the front of the instrument and open the lid.
- Locate the DIP Switch SW2 at the top of the main circuit board. This is the red one illustrated at right
- Set SW2 position #1 and #6 as follows.

	60Hz	50Hz
Position #1	OFF	OFF
Position #6	OFF	ON



## Display Nanograms or Milligrams Per Cubic Meter

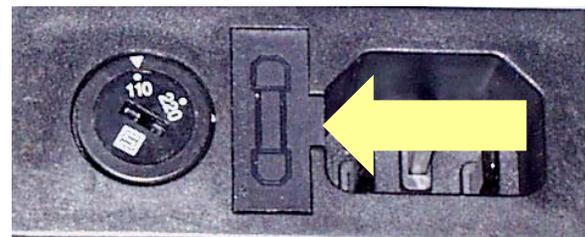
The instrument is factory set to display  $\text{mg}/\text{m}^3$  (milligrams per cubic meter) of mercury (.XXX). For some applications, including dosimeter analysis, the instrument's display can be converted to nanograms.

- Ensure the instrument is turned OFF.
- Remove the two screws near the front of the instrument and open the lid.
- Locate DIP Switch SW2 at the top of the main circuit board.
- Place Position #2 to OFF for nanograms display.
- Return Position #2 to ON for the normal milligram display.

## Changing the Fuse

If the instrument display reads .P.P.P when the instrument is connected to AC power or when REGEN is pressed, or if the battery will not charge, the fuse may need to be replaced. The AC line power could also be less than 100 VAC (220 VAC). Check the fuse with an ohmmeter and the AC line power with a voltage meter.

- Locate the power receptacle on the rear of the instrument.
- Insert a small screwdriver in the slot, located in the power receptacle, and gently slide the fuse compartment out.
- If the fuse in the open-sided clip is open, remove and discard it.
- Replace the discarded fuse with the spare fuse located in the slide-out spare fuse compartment.
- Replace the fuse compartment in the power receptacle.
  - As soon as possible, replace the spare fuse with another 1A, 250V, time delay fuse, AZI P/N 5100 1012).



## 6. CALIBRATION

The Jerome 431-X's gold film sensor is inherently stable and does not require frequent calibration. The interval between calibrations depends upon the application and frequency of use; however, the recommended interval is every 12 months.

The Jerome 431-X has been factory calibrated using laboratory equipment containing NIST traceable permeation tubes. These permeation tubes have a rated accuracy of +/- 2%. In order to calibrate the Jerome 431-X, a sophisticated calibration system is required that ensures stability of the calibration gas source, eliminates any pressure in the calibration gas stream, and controls the temperature of the calibration environment.

We strongly recommend you take advantage of our calibration and maintenance service at Arizona Instrument. Call Customer Service at (800) 528-7411 or (602) 470-1414 to arrange re-calibration. A certificate of calibration is issued from AZI when your instrument is factory calibrated.

### Verification of Calibration and Quality Control

The Functional Test Kit (FTK), AZI P/N Y431 0902, is used to determine if your instrument is within calibration tolerances between recommended annual factory calibrations. It allows you to have complete confidence in the sample results. This test verifies proper instrument operation through the introduction of a known concentration of Mercury Vapor into the Jerome analyzer. **THIS TEST DOES NOT CALIBRATE THE INSTRUMENT.**

If your application requires frequent verification of instrument function, this test demonstrates the unit's operation, calibration, and function. Recording FTK results in an instrument log provides a quality control/quality assurance record of instrument function between regular calibrations. If test results fall within the expected range, you may assume the instrument is functioning correctly.

See APPENDIX A - 431-X FUNCTIONAL TEST KIT on page 37 for more information about the FTK procedures. Complete instructions for use are supplied with the test kit, AZI P/N Y431 0902.

To order the FTK, contact your AZI Sales Representative at (800) 528-7411 or (602) 470-1414.

## 7. 431-X TROUBLESHOOTING

Symptom	Possible Cause	Solution
<b>Power Problems</b>		
Unit does not turn ON. Unit turns on when power cord is plugged in. LCD displays 000 when instrument is operating on AC power.	Discharged battery or  Dead battery.	Recharge battery for a minimum of 14 hours. Refer to page 17.  Replace battery. Refer to page 21.
Unit does not turn on when connected to AC power cord.	Open fuse.  Insufficient power.  Internal component failure.	Replace fuse. Refer to page 22.  Use a voltmeter to verify there is power to the AC outlet.  Call AZI Customer Service for information at 800-528-7411 or 602-470-1414.
<b>Regeneration &amp; Zero Problems</b>		
LCD displays .8.8.8.	Sensor saturated.	Do not attempt to adjust zero pot. The sensor must be regenerated. Refer to page 12 for information.
LCD displays .L.L.L when taking first sample.	Changes in temperature.	Readjust zero pot. See page 13 for information.
LCD displays H at finish of sensor regeneration when zero is pressed.	Internal contamination may redeposit Mercury Vapor from flow system onto gold film sensor.	Remove and replace fritware filter, intake filter disk, scrubber filters and Tygon tubing. Refer to "Flow System" on Page 19.  Check tubing for kinks or crimps. Repeat regeneration cycle. Refer to page 12.
Zero adjust pot cannot be adjusted to 0.	Pot not turned sufficiently.    Sensor may be ruptured or pot may be broken.	1. Turn zero adjust up to 20 times to reach the end. Pot will "click" softly.  2. If no "0", turn pot slowly in opposite direction until display reads "0".  3. If still unchanged, call AZI Customer Service at (800) 528-7411 or 602-470-1414.

## Sampling Problems

Airflow is restricted during the sensor regeneration cycle, causing possible permanent damage.	Kinks and crimps in the Tygon tubing.	Periodically check the Tygon tubing inside the instrument. Refer to page 20.
High erratic results.	Internal Mercury Vapor contamination.	<ol style="list-style-type: none"> <li>1. Install zero air filter in intake and tighten intake nut. Press SAMPLE button. After three samples, if readings are over 0.003 mg/m<sup>3</sup>, replace fritware filter and Tygon tubing. Refer to page 19.</li> <li>2. Perform sensor regeneration with the zero air filter in intake. Refer to page 12. Retest if necessary. Replace scrubber filters and Tygon™ tubing. Refer to page 20.</li> </ol>
High/erratic results	Intake and internal filters may become clogged and need replacement when sampling in a dusty or humid area.	<ol style="list-style-type: none"> <li>1. Open instrument and check for pinched, crimped or disconnected internal tubing.</li> <li>2. In extreme conditions, an additional particle filter may be installed on the intake.</li> </ol>
High/erratic results Readings vary more than 0.05 mg/m <sup>3</sup> when in survey mode.	Loose connections to gold film sensor.	Place a zero air filter into the intake. Place the instrument in survey mode. Move the unit as samples are being taken. Call AZI Customer Service at 800-528-7411 or 602-470-1414 for assistance.
Low response or erratic readings after a long period of non-use.	May need a second regeneration cycle.	<ol style="list-style-type: none"> <li>1. Wait 30 minutes and perform another sensor regeneration.</li> <li>2. Test with FTK. Refer to page 37.</li> <li>3. If still unresponsive, call AZI Customer Service at 800-528-7411 or 602-470-1414 for assistance.</li> </ol>

False readings, may go to .8.8.8 or .L.L.L.	Extremely cold or extremely warm air sampled into unit.	If sampling under these conditions, install zero air filter in intake. Sample until display reads 0.003 mg/m <sup>3</sup> or less. This equilibrates sensor temperature with the temperature of the sample air stream. Remove filter and take samples.
<b>Miscellaneous Problems</b>		
Display reads .P.P.P when regeneration is attempted.	<p>Power cord not attached.</p> <p>Blown fuse.</p> <p>Line voltage less than 100 VAC (or less than 200 VAC for 220 unit).</p> <p>Cycles dipswitch set incorrectly.</p>	<p>Check power cord for connection</p> <p>Replace fuse. Refer to page 22.</p> <p>Check line voltage settings. Refer to page 21.</p> <p>Check input cycle settings. Refer to 55.</p> <p>If fuse and line voltage are OK, it may be circuit board adjustment or component failure. Call AZI Customer Service at 800-528-7411 or 602 470-1414.</p>
Display reads .E.E.E	Very low battery.	<p>Recharge battery. Refer to page 17.</p> <p>Replace battery. Refer to page 21.</p>

## 8. JEROME 431-X TECHNICAL SPECIFICATIONS

Range	0.001 to 0.999 mg/m <sup>3</sup>
Sensitivity	0.003 mg/m <sup>3</sup> Hg
Precision	5% relative standard deviation at 0.100 mg/m <sup>3</sup> Hg
Accuracy	+/-5% at 0.100 mg/m <sup>3</sup> Hg
Response time-sample mode	12 seconds
Response time-survey mode	3 seconds
Flow rate	750cc/min (0.75 liters/min)
Power requirements	100-120 VAC, 50/60 Hz, 1 A, or 220-240 VAC, 50/60 Hz, 1 A
Fuse	F1A 250V, 5mm X 20mm
Internal battery pack	Rechargeable Nickel Cadmium
Operating environment	0° to 40°C, non-condensing, non-explosive
Case construction	Aluminum alloy
Dimensions	15 cm x 33 cm x 10 cm (6" w x 13" l x 4" h)
Weight	3.18 kilos (7 pounds)
Digital meter display	Liquid crystal display (LCD)
Certification	CE mark on 220-240 V~, 431-XE model only.

## Optional Communications Capability

Alarm output	30V DC, 100mA
Dosimeter power output	For dosimeter analysis and regeneration
Data output	<ol style="list-style-type: none"> <li>1. RS-232 Serial, Baud Rate 1200 for use with data logger, and/or Jerome communication program.</li> <li>2. RS-232 Serial data format with 0 &amp; 20mA current logic levels; Baud Rate 1200 (special industrial applications) and Analog 20 mA output.</li> </ol>
<b>"With Option Board" - See APPENDIX E - JEROME 431-X OPTION BOARD on page 58.</b>	
Analog output	0 to 2V or 4 to 20 mA
Auto sample interval	5, 15, 30, or 60 minutes
Auto regeneration interval	6, 24 or 36 hours

## Instrument I/O Interface

The 431-X I/O port (25 pin D-sub) provides the following functions:

- Serial data communication
  - Interface type: RS-232C full duplex, DCE
  - Parameters: 1200 Baud, 1 start bit, 8 data bits, 2 stop bits, no parity
  - Pin assignments:
    - Pin 1 Protective ground
    - Pin 2 Data in
    - Pin 3 Data out
    - Pin 7 Data ground
- Serial current loop
  - Interface type: 20mA current loop, full duplex
  - Parameters - 1200 Baud, 1 start bit, 8 data bits, 2 stop bits, no parity
  - Pin assignments:
    - Pin 1 Protective ground
    - Pin 4 Data out (+)
    - Pin 5 Data in (+)
    - Pin 14 Data out (-)
    - Pin 16 Data in (-)
- Alarm output
  - Maximum voltage 30 VDC
  - Maximum current 100mAmp
  - Pin assignments:
    - Pin 9 Switched battery (+)
    - Pin 10 Alarm output (open collector, active low)
    - Pin 7 Battery ground (-)
    - Pin 23 Battery ground (-)
- Dosimeter Power
  - Pin Assignments:
    - Pin 22 Dosimeter Enable – 24 to 28 Volts AC
    - Pin 23 Battery Ground
    - Pins 12 & 24 Tied – Dosimeter Power
    - Pins 13 & 25 Tied – Dosimeter Power
- Switched battery connection for data logger
  - Pin assignments:
    - Pin 9 Battery (+)
    - Pin 7 Battery ground (-)
    - Pin 23 Battery ground (-)
- Unswitched battery connection for external battery pack pin assignments
  - Pin assignments:
    - Pin 15 Battery (+)
    - Pin 19 Battery (+)
    - Pin 7 Battery ground (-)
    - Pin 23 Battery ground (-)

**NOTE:** Pins 6, 8, 11, 17, 18, 20 and 21 are non-standard and should not be connected.

## Potential Interferences

Potential interferences to the Jerome mercury vapor analyzers are rare and most of these can be eliminated with proper maintenance procedures. However, erroneously high readings can sometimes occur. Here are a few things to be aware of when using the instruments.

The gold film sensors used in the Jerome mercury vapor analyzers do not respond to the following compounds:

- Hydrocarbons
- CO, CO<sub>2</sub>, and SO<sub>2</sub>
- Water vapor (Note that water vapor condensation on the gold film can cause irreparable harm to the sensor and must be avoided.)

The acidic gas filter, contained in the internal filter system, removes the following compounds that cause the gold film sensor to respond:

- Chlorine
- NO<sub>2</sub>
- Hydrogen Sulfide (H<sub>2</sub>S)
- Most mercaptans (organic sulfur compounds or “thiols”)

In areas containing these highly volatile compounds, the filter can become quickly saturated. In such situations, it is recommended that these gases be allowed to dissipate before sampling for the less volatile, more persistent mercury vapor. A special filter designed to remove chlorine gas is available from Arizona Instrument and may be ordered as Chlorine Filter, AZI P/N Z2600-3940. Collection of air samples with Jerome gold coil dosimeters for analysis by the Jerome mercury vapor analyzers will also eliminate interferences.

Ammonia in very high concentrations can cause an offgassing of accumulated acidic fumes from the internal acidic gas filter, resulting in positive readings on the instrument. In these cases, the ammonia odors are very strong. Again, either allow the vapors to dissipate or use the dosimeters. Filter replacement at regular intervals, or when unexpectedly high readings are encountered in areas of these potential interferents, may resolve these problems.

Volatile mercury compounds in general will cause the gold film to respond. Alkyl organic mercuries such as methyl mercury (and other “straight chained” compounds) are typically extremely volatile and change the electrical resistance of the gold film sensor. Any such responses should be considered “qualitative,” **not** quantitative. The instruments are designed and calibrated to elemental mercury vapor only.

Inorganic mercury salts such as mercuric chloride are not very volatile. They may, however, generate some minute level of elemental mercury vapor to which the instruments will respond. This response, again, should be considered a qualitative response only.

## 9. ACCESSORIES & MAINTENANCE PARTS

PART #	ITEM DESCRIPTION
<b>Y431 0901</b>	<b>431 Accessory Kit (See pictures beginning on page 32)</b>
	1400 2002 Probe
	1400 3010 Tubing Adapter, 1/4" to 1/8"
	2300 0001 Trimmer Tool
	2600 3039 .25 Fritware
	6000 4003 Line Cord, 115 VAC - USA and Canada
	Alt. 200-0003 Line Cord, 220-240 VAC - England
	Alt. 200-0008 Line Cord, 220-240 VAC - Europe
	Z2600 3905 Zero Air Filter
<b>Y431 0902</b>	<b>431 Functional Test Kit (See pictures beginning on page 32)</b>
	A2600 0902 Stopper Assembly
	A2600 0903 Syringe Assembly
	A2600 0904 Mercury Vial
	2600 0022 Syringe Needles, 22 Ga. Reusable
	2600 0030 Calibration Vessel, Thermos
	3200 0011 Septa (20)
	Z2600 3914 Septum Holder
<b>Y431 0903 or</b>	<b>431 Maintenance Kit (110 VAC) (See pictures beginning on page 32)</b>
<b>Y431 0904</b>	<b>431 Maintenance Kit (220 VAC) (See pictures beginning on page 32)</b>
	2500 3001 1' of 1/8" Tygon tubing
	2600 3039 .25 inch fritware
	Z2600 3905 Zero Air Filter
	Z2600 3928 C/M Filter
	Z2600 3930 Scrubber Filter
	Z4000 0907 Battery Pack Assembly
<b>Y990-0175 or</b>	<b>Dosimeter Analysis Kit (110 VAC applications)</b>
<b>Y990-0176</b>	<b>Dosimeter Analysis Kit (220 VAC applications)</b>
	990-0177 Pocket Pump, calibrated to 5 ml/min
	990-0159 Converter, 220VAC to 110VAC, 200 WATT (supplied with Y990-0176 for locations using 220 VAC power)
	X412 0901 Personal Mercury Dosimeter (2)
	2500 3001 2' of 1/8" Tygon tubing
	2500 3010 1' of 3/16" Tygon tubing
	2100 6017 Dosimeter Lead Set
	1300 0031 3/16" to 1/8" Reducer
	Z2600 3905 Zero Air Filter

Y6100 0057 **Jerome Data Logger**  
Includes the Jerome Data Logger  
and JCS Software Kit.



Y990-0175 **Personal Dosimeter Kit**  
Includes the Pocket Pump, two  
mercury dosimeters, zero air  
filter, regeneration cable,  
connecting tubing and adaptor.



Y6100 0054 **Jerome Communication  
Software Kit**  
Y990-0170 [TO BE REPLACED BY THE  
JEROME COMMUNICATION  
SOFTWARE (JCS) ON CD  
ROM]



Y411 0904 **Hard Side Carry Case**  
Includes a molded case with die  
cut foam rubber inserts to hold the  
Jerome 431-X and accessories.



1400 0052 **Soft Field Carrying Case**  
Hand/shoulder case with pockets  
for accessories.



**Spare Parts**

X421 0901 Personal Mercury Dosimeter



A2600 0902 Stopper Assembly



Z2600 3914 Septum Holder



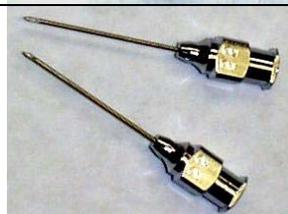
3200 0011 Septa

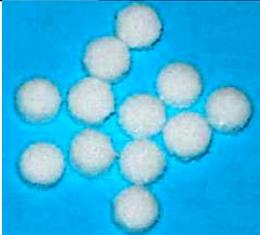


A2600 0903 Syringe Assembly



2600 0022 Syringe Needles, 22 Ga.



1400 2002	Probe	
2300 0001	Trimmer	
1300 0031	1/8 x 3/16 reducer	
Z4000 0907	Battery Pack Assembly	
Z2600 3905	Zero air filter	
Z2600 3928	C/M filter	
Z2600 3930	Scrubber filter	
Z2600 3940	Chlorine Filter	
1400 3010	Tubing adapter	
Y26003945	Intake Kit	 Includes mounting hardware
PS 151	Tube Nut	
2600 3039	.25 inch fritware	

2500 3001 Tygon tubing 1/8" I.D. (1 foot)



2500 3002 Tygon tubing 1/16" I.D. (1 foot)



4000 1011 115 VAC IDC battery charger  
Used to charge an uninstalled battery



4000 1012 230 VAC IDC battery charger  
Used to charge an uninstalled battery



6000 4003 100-120 VAC Line Cord



Alternate – 220-240 VAC Line Cord for  
200-0003 England



Alternate – 220-240 VAC Line Cord for  
200-0008 Europe



Z2600 3911 10 to 1 Dilution Module



5100 1012 Spare Fuse



6000 1055 Jerome Communication Cable



6000 1045 Adaptor, RS-232, 9M/25F



**For current prices and delivery information, call AZI Customer Service at  
(800) 528-7411 or (602) 470-1414.**

## **10. Factory Calibration Service**

Service includes filter replacement, component testing, and instrument calibration to NIST traceable standards.

**For scheduling and shipping authorization, call AZI Customer Service at  
(800) 528-7411 or (602) 470-1414.**

## 11. APPENDIX A - 431-X FUNCTIONAL TEST KIT

If your application requires frequent verification of instrument functionality, this test will benefit you. If the test results fall within the expected range, you may assume the instrument is functioning properly. This test does not calibrate the instrument.

**NOTE: Perform the functional test ONLY after a sensor regeneration.**

The 431-X Functional Test Kit contains all accessories necessary to perform the functional test. See the complete list on page 30 and verify that all the parts to the kit are present.

### **CAUTION:**

**The vial and thermometer contain liquid mercury and are possible sources of mercury contamination. Follow the instructions for handling or transferring the mercury into the Functional Test Kit Vessel carefully.**

**For safety information, see the supplier's Material Safety Data Sheets (MSDS) or call AZI Customer Service at 1-800-528-7411 or 1-602-470-1414 for assistance in obtaining the MSDS.**

## Preparation

- Carefully unpack and inspect the parts of the kit.
- ENSURE that the mercury shipping container and mercury filled thermometer are not broken.
- In a ventilated area, preferably under a fume hood, remove the mercury vial from its shipping container.
- Place the functional test kit vessel and the mercury vial close to each other and open the mercury vial.

### **CAUTION:**

**The edge between the plastic case and the glass inner vessel of the functional test kit vessel are not sealed tight enough to prevent mercury from entering the area between the inner and outer vessels. ENSURE the mercury, transferred in the next step, does not contact the seal where the glass and plastic portions join.**

**NOTE:** The vessel may be disassembled to transfer the mercury and better prevent contamination of the outer portion of the vessel. Instructions to disassemble the vessel can be found on page 38.

## Mercury Transfer

- CAREFULLY pour the mercury into the center of the functional test kit vessel's opening.
  - ENSURE that no mercury residue is on the outside of the vessel. See the supplier's Material Safety Data Sheets (MSDS) or call AZI Customer Service at 1-800-528-7411 or 1-602-470-1414 for clean-up instructions.
- INSTALL the stopper assembly into the functional test kit vessel carefully, to prevent breakage of the thermometer.
  - PRESS the stopper assembly into the vessel to achieve a good seal.
- USE the 431-X instrument to verify that the outside of the vessel is not contaminated and the mercury vapor emission level, if any, is below the OSHA TLV for mercury.
- ALLOW the kit to adjust to room temperature for at least two (2) hours before using.
  - The temperature range for the test is 18-22°C. Avoid temperature fluctuations.



### CAUTION:

Do not use the calibration vessel as a portable container. If the calibration vessel is upset or greatly agitated, mercury droplets will cling to the thermometer stem, the rubber stopper, the mouth of the calibration vessel and the needle guide.



## Vessel Disassembly



### CAUTION:

The inner portion of the vessel is made of glass. Handle the vessel carefully to prevent breakage.



- LOOSEN, BUT DO NOT REMOVE the base of the vessel. The base unscrews from the body.
- SET the vessel on a firm surface.
- HOLD the base stationary and unscrew the body from the base.
- HOLD the base and the inner glass vessel with one hand while removing the body and gasket with the other hand.
- After the mercury is transferred into the glass inner vessel, reassemble in the reverse order.

## Replacing Mercury

An oxide coating will form on the drop of mercury and will cause lower readings in your testing. Gently swirl the vessel to disturb the outer oxidized surface of the droplet. If this does not restore higher readings, it may be necessary to replace the mercury.

- Carefully remove the stopper assembly from the calibration vessel.



**CAUTION:**  
**BE SURE NEEDLE GUIDE IS FREE OF LIQUID MERCURY.**



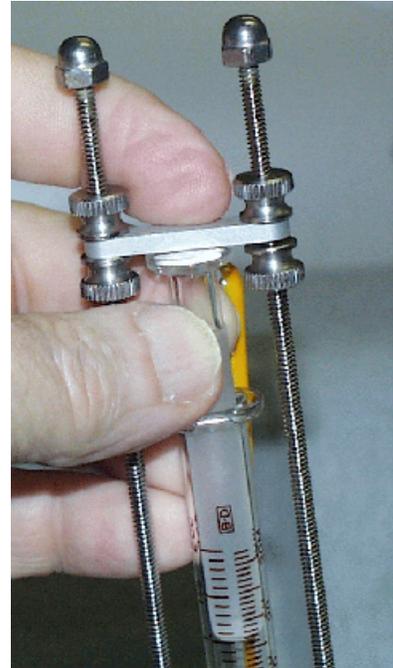
- Carefully pour the mercury into a disposal vessel. Refer to Vessel Disassembly Instructions on page 38.
  - Mercury can become trapped between the plastic calibration vessel and the glass inner-liner.
- Replace the oxidized mercury with approximately ½ cc fresh mercury. (AZI P/N A2600-0904)
  - Do NOT use the syringe for measuring liquid mercury. Dispose of oxidized mercury properly.
- Reassemble the calibration vessel.
- Reinstall the stopper assembly.

## Syringe Technique

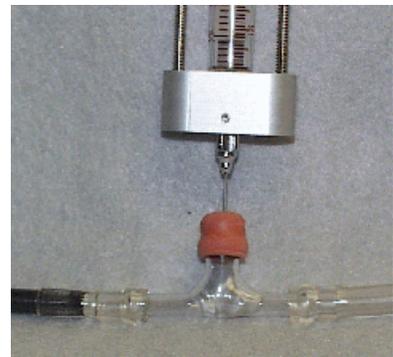
- Pull and hold the syringe plunger against the bar-stop.
- Verify that the black mark on the syringe plunger aligns with the 1cc mark on the syringe barrel.
  - If it does not, the holder assembly must be adjusted. Call AZI customer service at 602-281-1745 or 800-528-7411 for assistance.
- Insert the needle into the needle-guide of the bottle stopper.



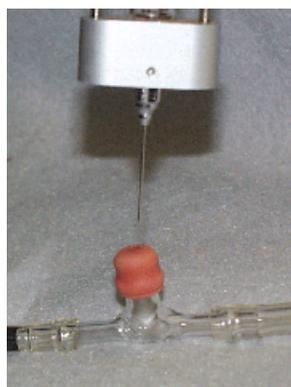
- Operate the plunger two or three times to pump mercury vapor into the syringe. On the final stroke, pull and hold the plunger against the bar-stop.
- Holding the plunger against the bar-stop, remove the syringe from the bottle and move it to the septum attached to the instrument intake.



- Continue to hold the plunger against the bar-stop and insert the syringe needle into the septum.
- Press “SAMPLE” on the instrument.



- When the display flashes, release the plunger and allow gravity to feed the mercury vapor into the airstream. If the plunger stops, gently press it completely closed.
- Remove the syringe needle from the septum.



## Functional Test Procedure

**NOTE:** Perform the functional test **ONLY** after sensor regeneration.

- Allow the calibration vessel to remain stable at room temperature for at least 2 hours.
  - The temperature range for the test is 18° - 22°C.
  - Temperature fluctuations during the test procedure will produce erratic results.
- Replace the .25mm fritware.
  - Refer to page 19 for instructions.
- Replace the septum on the septum holder assembly.
- Plug the tubing adapter end of the septum assembly into the instrument's intake and tighten the intake tube nut.

**NOTE:** To check for a tight seal, gently pull on the septum holder assembly. If it comes out of the intake, it may be necessary to remove the intake tube from the instrument and firmly press the tubing adapter through the intake. Tighten the intake tube firmly to the intake stem.

- Attach a zero air filter to the septum assembly.
- Press power ON.
- Take 3 samples.
  - If the average meter reading is greater than .005, stop here. The instrument may be contaminated. See 431-X TROUBLESHOOTING on page 24.
  - If the average meter reading is less than .005, continue to the next step.
- Note the temperature of the calibration vessel.
- Press the SAMPLE button, wait 2 seconds and **when the display flashes**, inject 1 cc of mercury vapor according to the syringe technique described on page 39. Be sure all mercury vapor has been injected before the solenoid closes (second click and display flash).



### CAUTION:

**Carefully follow these instructions to minimize error.**



- Record the meter reading.
- Repeat the instructions for mercury injection three more times.
  - The readings obtained for the last three 1cc injections should be within +/- 5% of each other.
- Refer to the Temperature Conversion Chart, page 42, for the acceptable range.
  - The average of the last three readings should fall within the range shown on the chart.

**If the average is within range, the JEROME 431-X is functioning properly.**

- If the last three readings are not within +/-5% of each other,
  - Perform sensor regeneration. Press ZERO and turn the ZERO ADJUST (refer to page 12 and 13 for the complete sensor regeneration procedure).
  - Wait 1 hour before proceeding to the next step.
  - Repeat the mercury injection test procedure.
  - If the average of the last three readings is still not within range, refer to the section on Functional Test Troubleshooting below.

**431-X Temperature Conversion Chart**

Temperature in °C	Digital Meter Response
16	.091 to .123
17	.100 to .135
18	.108 to .146
19	.118 to .159
20	.129 to .174
21	.138 to .187
22	.151 to .204
23	.164 to .222
24	.177 to .240

## Functional Test Troubleshooting

If you don't achieve good results with the functional test procedure, check the following:

<b>Results</b>	<b>Solution</b>
Typically too high	Ensure the calibration vessel temperature is stable.
Too Low	Be sure to inject the Hg vapor ONLY after the display flashes (approximately 2 seconds after SAMPLE is pressed).
	Ensure there is no oxidation on the mercury drop in the calibration vessel. Gently swirl the mercury drop in the calibration vessel. Replace if necessary.
	Ensure the instrument's intake is not blocked with foreign matter. Check flow with a flow meter.
	Ensure syringe is calibrated to 1cc. Use a new syringe needle. Straighten or replace crimped or blocked internal tubing.

If you find the above does not solve your problem, please call AZI Customer Service at 800-528-7411 or 602-470-1414.

## 12. APPENDIX B - PERSONAL MERCURY DOSIMETER

The gold coil personal mercury dosimeter is a unique collection device for mercury vapor. The Jerome 431-X Gold Film Mercury Vapor Analyzer and the Personal Mercury Dosimeter determine personal exposure levels and ambient air concentrations, as well as low levels of mercury in natural and stack gases

For personal sample collection, the dosimeter is worn as close to the wearer's breathing zone as possible and is connected by tubing to a pump usually worn on a belt. The dosimeter can also be used for multiple point area monitoring by placing a dosimeter, with pump attached, in various strategic locations.

We recommend a pump flow rate of 5 cc/minute for the most accurate results when sampling in an atmosphere that for eight hours may contain an average of .025 mg/m<sup>3</sup> Hg. If you are considering using any other flow rate, see page 48, Non-Standard Flow Rates and Dilution Modules .

After sample collection is completed, the dosimeter is inserted in the Jerome 431-X's intake. A dosimeter lead set is connected between the dosimeter and the DB-25 connector on the back of specially equipped instruments. The instrument supplies power to volatilize the accumulated mercury from the dosimeter to the gold film sensor. The Jerome 431-X determines the mass of mercury collected by the dosimeter in a 17 second analysis. The dosimeter is ready for immediate re-use after a mercury measurement has been performed.

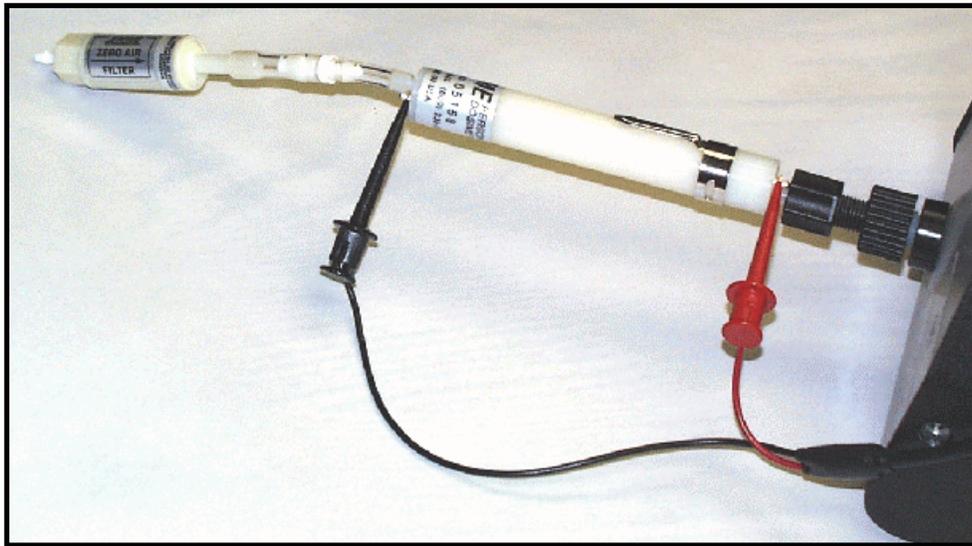
### Dosimeter Technical Specifications

Sensitivity	Less than 0.5 nanograms of mercury
Precision	15% RSD @ 0.100 mg/m <sup>3</sup> Hg
Accuracy	15% @ 0.100 mg/m <sup>3</sup> Hg
Recommended flow rate	5 cc/min (0.005 liters/min) for atmospheres up to 0.025 mg/m <sup>3</sup>
Construction	Nylon and glass housing a gold film coil
Weight	1.5 ounces
Dimensions	0.5" dia. x 4.5"
Capacity	1000 nanograms of mercury
Analysis Time	Less than two 2 minutes

## Before Sampling with the Dosimeter

The personal mercury dosimeter adsorbs mercury vapor over a set period of time. Therefore, before each day's use it is necessary to ensure the dosimeter is mercury free. Perform the following steps to remove any accumulated mercury.

- Connect the system as shown.
  - Insert the dosimeter's large end in the 431-X's intake and gently tighten the intake tube nut to ensure an airtight seal.
  - Connect the Dosimeter Lead Set clips as shown, Short red lead to the rear and long black lead to the far end.



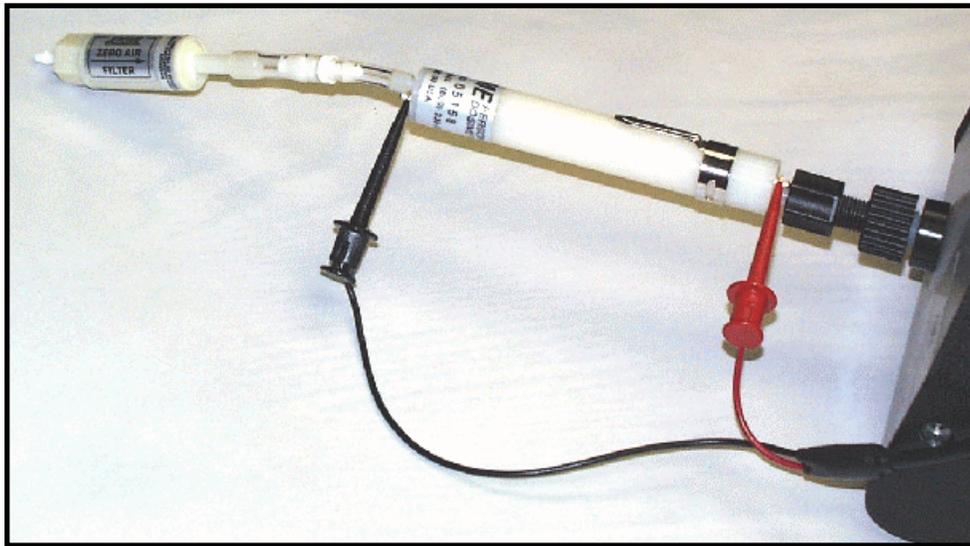
- Attach the power cord to the 431-X and plug it into AC power. AC power is required to heat the dosimeter.
- Attach the Dosimeter Lead Set 25 pin connector to the respective 25 pin communications port.
- Press the instrument's power ON button.
- Press the instrument's SAMPLE button.
  - The digital meter reading will appear in 15 seconds.
- Wait 60 seconds and press the SAMPLE button again.
  - The digital meter should display less than 0.005, verifying all mercury has been removed from the dosimeter coil.
- Wait 2 minutes to cool sensor to prevent false positive response.
- The dosimeter is ready for sample collection.

**NOTE:** For best results, dosimeter analysis should be performed immediately after collection. If analysis cannot take place immediately, place the red end caps on the dosimeter. For accurate results, perform dosimeter analysis no later than five days after sampling.

## Dosimeter Analysis

NOTE: Wait a minimum of 30 minutes after a sensor regeneration before continuing.

- Connect the system as shown.
  - Insert the dosimeter's large end in the 431-X's intake and gently tighten the intake tube nut to ensure an airtight seal.
  - Connect the Dosimeter Lead Set clips as shown, Short red lead to the rear and long black lead to the far end.



- Attach the power cord to the 431-X and plug it into AC power. AC power is required to heat the dosimeter.
- Attach the Dosimeter Lead Set 25 pin connector to the respective 25 pin communications port.
- Press the instrument's power ON button
- Reading the dosimeter (dosimeter desorption)
  - Press the SAMPLE button.
    - ◆ The digital meter reading appears in 15 seconds.
  - Record the digital meter reading (include the decimal point).
  - Wait 30 seconds.
  - Press SAMPLE again and record this digital meter reading. Repeating the heating/reading process ensures complete release of mercury from the dosimeter coil.
- Add the two digital meter readings together. The sum of the two digital meter readings is the figure you will use in your calculations and is referred to as the meter response (MR).

NOTE: A third dosimeter desorption (pressing the SAMPLE button) should give a reading of  $.005 \text{ mg/m}^3$  or less.

Perform the following calculation to obtain the mercury concentration in mg/m<sup>3</sup> based on a time weighted average; **or alternately, DIP switch #2 can be set to OFF** and the digital meter will display nanograms Hg directly (refer to diagram, page 55 ).

### Working Formula and Units of Measure

$$\frac{\text{Meter Response X ConversionFactor}}{\text{Pump FlowRate X Sampling Time}} = \text{Sample Concentration}$$

Where:

Meter Response	=	Total of the two digital meter readings in mg/m <sup>3</sup>
Conversion Factor	=	87.5 ng/mg/m <sup>3</sup> (a constant which changes the meter response to nanograms of Hg)
Pump flow rate	=	5.0cc per minute (calibrated value of the supplied SKC Pocket Pump)
Sampling time	=	Duration of the sample in minutes
Sample concentration	=	In ng/cc mg/m <sup>3</sup>

**EXAMPLE:** To calculate a time weighted average during an 8 hour period using the following values:

Meter response	=	0.600 mg/m <sup>3</sup> (sum of the two meter response readings)
Conversion factor	=	87.5 ng/mg/m <sup>3</sup> (constant)
Pump flow rate	=	5 cc/min
Sampling time	=	8 hours (480 min)

- Convert the meter response (the total of the two digital meter readings) to nanograms of mercury.
  - 0.600 x 87.5 = 52.5 nanograms of Hg
- Determine the total volume of air sampled.
  - 5 cc/min x 60 min/hr x 8 hr = 2400 cc
- Determine the Hg concentration (time weighted average) of the dosimeter.

$$\frac{52.5\text{ng}}{2400\text{cc}} = 0.022 \text{ ng / cc of Hg} = 0.022 \text{ mg/m}^3 \text{ of Hg}$$

Check the sensor status after each dosimeter analysis.

**IMPORTANT:**  
**To prevent the loss of a sample,**  
**perform sensor regeneration as soon as the display shows “- - -” (four bars).**  
**This indicates that the sensor is 75-100 percent saturated.**

- Seal the dosimeter with caps after analysis to prevent mercury contamination during storage.
- If your readings exceed 75 nanograms or more, try the recommendations described next or call AZI Customer Service at 800-528-7411 or 602-470-1414 for assistance.

### **Non-Standard Flow Rates and Dilution Modules**

You may use a pump with a flow rate up to 50 cc/min, but be aware that there are certain limitations. If your pump flow rate exceeds 5 cc/min and your average dosimeter analysis produces nanogram levels of 75 or more, it may be easy to collect enough mercury to saturate the 431-X sensor. You thus risk over ranging your instrument and losing your collection data. Higher flow rates may also impair the capture efficiency of the dosimeter.

We recommend that you drop your flow rate or use a dilution module\* (AZI P/N Z2600-3911). Lowering the flow rate to decrease the sample volume provides the greatest accuracy. Using a dilution module introduces an additional 15% inaccuracy to your analysis. As an alternative to the dilution module, sample for shorter time periods.

#### **Dilution Module Specifications**

Accuracy	+/- 15% of 10:1 ratio	
Input concentration range	Low	0.7 mg/m <sup>3</sup> Hg
	High	5.0 mg/m <sup>3</sup> Hg
Housing	Nylon	
Dimensions	1" w x 2.7" l x 3" h	
Weight	3.3 oz	

The dilution module is factory set to a 10:1 ratio. The mass of mercury entering the dilution module is reduced by 90%, leaving a 10% (X10 dilution) concentration to be introduced into the Jerome 431-X. Since this ratio can change slightly with use, it is important to occasionally determine the current dilution module ratio to ensure accurate results. For normal applications a X8 to X12 ratio is recommended. The 431-X Functional Test Kit contains all accessories necessary to determine the current dilution module ratio.

Call Customer Service at 800-528-7411 or 602-470-1414 if you have questions about flow rates or applications.

\*The dilution module contains Resisorb™, mercury vapor adsorbent. For safety information, see the supplier’s Material Safety Data Sheets (MSDS) or call AZI Customer Service at 1-800-528-7411 or 1-602-470-1414 for assistance in obtaining the MSDS.

## Dilution Module Ratio Check

The 10:1 dilution module was manufactured and calibrated to produce a 10:1 ratio in the sample delivered to the instrument. Over time the ratio may change slightly. To accurately determine the exact dilution offered by the 10:1 dilution module, perform the following tests and calculate the exact dilution ratio. The calculated ratio can then be used in all final calculations where the dilution module is used.

NOTE: Wait a minimum of 30 minutes after sensor regeneration before starting this procedure.

### Direct 431-X Readings:

- Connect the instrument, septum holder assembly and zero air filter, with arrow pointing to the instrument, as shown.
- Press the Jerome 431-X power ON button.
- Inject 1 cc of mercury saturated vapor into the septum, according to the Syringe Technique described on page 37, Appendix A.
- Make 3 additional 1 cc injections sample readings and record the displayed values (include the decimal points).
- Average the results of the last 3 injections.
- Remove the septum assembly and zero air filter from the instrument.
- Connect the 10:1 dilution module to the instrument, connect the septum assembly and zero air filter to the dilution module.
- Inject 1 cc of mercury vapor as above into the septum.
- Make three additional injections and average the three readings.
  - Divide this result into the average from the three direct injections.
- Use the result as the dilution module ratio in your dosimeter analysis.



### Most Accurate Method

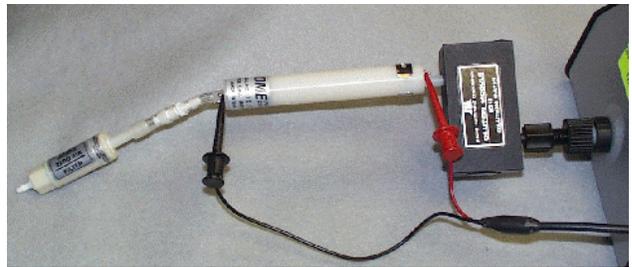
Perform the above test, however attach the dosimeter, septum holder assembly and zero air filter to the sampling pump that will be used. The technique is described in the next section.  
Collection efficiencies should be approximately 100%.

## Loading the Dosimeter

- Connect your pump, dosimeter, septum holder assembly and zero air filter together with 1/8" and 3/16" Tygon™ tubing as shown to the right.
- Turn on the pump.
- Inject 10 cc's of Hg into the septum, 1 cc at a time.
  - A total of ten, 1cc injections, one after another.



- Wait 30 seconds after the last injection then turn off the pump.
- Remove the dosimeter, septum assembly and zero air filter from the pump.
- Connect the instrument, dilution module, dosimeter, zero air filter and dosimeter lead set as shown.
- Attach the power cord to the 431-X and plug it into AC power.
  - AC power is required to heat the dosimeter.
- Press the Jerome 431-X Power ON button and then press the SAMPLE button.
  - The digital meter reading appears in approximately 15 seconds.
- Record the digital meter reading (include decimal point). Wait 60 seconds, then press SAMPLE again and record this reading.
- Repeating the heating process ensures complete release of mercury from the dosimeter coil.
- Add the two digital meter readings together.
  - The sum of the two digital meter readings is the figure you will use in your calculations and is referred to as the meter response (MR).
- Repeat this procedure two more times.
- Average the three meter responses you obtained in this section.



## Dilution Module Ratio Calculations

- Multiply the average obtained for **Direct 431-X Readings** by 10 (the number of 1 cc injections).
  - Divide this result by the average obtained in the section “Loading the Dosimeter” on page 50.
- Use the result as the dilution module ratio in your dosimeter analysis.

### EXAMPLE:

Direct 431-X readings

0.102 mg/m<sup>3</sup>  
0.103 mg/m<sup>3</sup>  
0.104 mg/m<sup>3</sup>  
0.103 mg/m<sup>3</sup> average

Loading the dosimeter

0.120 mg/m<sup>3</sup>  
0.113 mg/m<sup>3</sup>  
0.100 mg/m<sup>3</sup>  
0.111 mg/m<sup>3</sup>

Step 1 (above)

$$0.103 \text{ mg/m}^3 \times 10 = 1.030 \text{ mg/m}^3$$

Step 2 (above)

$$(1.030 \text{ mg/m}^3) / (0.111 \text{ mg/m}^3) = 9.4$$

Dilution module ratio 9.4:1

**NOTE:** For normal applications a X8 to X12 ratio is recommended. If your ratio is not within this range, call Customer Service at 800-528-7411 for assistance.

## Analysis with a Dilution Module

**NOTE:** Wait a minimum of 30 minutes after sensor regeneration before starting this procedure.

- Connect the system as shown in the figure below.
- Attach the power cord to the 431-X and plug it into AC power.



➤ AC power is required to heat the dosimeter.

- Press the Jerome 431-X power ON button and then press SAMPLE button. The digital meter reading appears in 12 seconds.
- Record the digital meter reading (include the decimal point). Wait 30 seconds, then press SAMPLE button again and record this reading.
  - Repeating the heating process ensures complete release of mercury from the dosimeter coil.
- Add the two digital meter readings together.
  - The sum of the two digital meter readings is the figure you will use in your calculations and is referred to as the meter response.
- The following calculations will provide the mercury concentration in  $\text{mg}/\text{m}^3$  based on a time weighted average.

$$(\text{Meter Response converted to nanograms (Ng) of mercury}) \times \frac{\text{Dilution Module Ratio}}{\text{Sample Volume}} = \text{Sample Concentration}$$

- Alternately, DIP switch #2 can be set to OFF and the digital meter will display nanograms Hg directly.

<b>MR</b> (meter response)	total of the two digital meter readings in mg/m <sup>3</sup>
<b>87.5 ng/mg/m<sup>3</sup></b>	conversion factor, a constant which changes the meter response to nanograms of Hg
<b>DM</b> dilution module ratio	the ratio determined on page 51
<b>SV</b> (sample volume)	pump flow rate (in cc/min) multiplied by sample time (in minutes)
<b>Sample concentration</b>	ng/cc = mg/m <sup>3</sup>

**EXAMPLE:**

Assume the following values.

Meter Response (MR)	0.600 mg/m <sup>3</sup> (sum of the two meter response readings)
Conversion Factor	87.5 ng/mg/m <sup>3</sup> (constant)
Dilution Module Rate	9.4
Pump Flow Rate	5 cc/min
Sampling time	8 hours = 480 min
Sample volume	5 cc/min X 480 min = 2400cc

A time weighted average during an 8 hour period is calculated by:

$$\frac{(0.600 \text{ mg/m}^3) \times (87.5 \text{ ng/mg/m}^3)}{2400 \text{ cc}} = 0.021875 \text{ ng/cc}$$

- Convert the meter response (the total of the two digital meter readings) to nanograms of mercury.
  - The MR (0.600) multiplied by the conversion factor (87.5) equals nanograms of mercury
  - 0.600 x 87.5 = 52.5 nanograms of Hg
- Determine the actual mass of Hg collected by the dosimeter.
  - Nanograms of mercury multiplied by the dilution module ratio.
  - 52.5 nanograms x 9.4 = 493.5 nanograms
- Determine the total volume of air sampled.
  - The pump flow rate multiplied by 60 min/hr multiplied by 8 hours.
  - 5 cc/min x 60 min/hr x 8 hr = 2400 cc
- Determine the Hg concentration (time weighted average) of the dosimeter.
  - The mass of Hg collected by the dosimeter divided by the total volume of air sampled.
  - 493.5 nanograms divided by 2400 cc = 0.205625 ng/cc of Hg = 0.205625 mg/m<sup>3</sup> of Hg
- Check the sensor status after each dosimeter analysis.
- Seal the dosimeter with tubing after analysis to prevent excessive mercury contamination during storage.

**IMPORTANT:**  
**Perform a sensor regeneration as soon as the meter display shows “- - -” (four bars) to prevent the loss of sample.**

### Dosimeter Reference Chart 431-X

Expected concentration, related to sample volume and meter response

= Indicates the optimum meter response for that concentration with the corresponding volume

		431-X reading in mg/m <sup>3</sup>									
Estimated concentration in mg/m <sup>3</sup>	0.5	0.429	0.857	HL							
	0.1	0.086	0.171	0.343	0.686	HL	HL	HL	HL	HL	HL
	0.05	0.043	0.086	0.171	0.343	0.686	HL	HL	HL	HL	HL
	0.025	0.021	0.043	0.086	0.171	0.343	0.686	HL	HL	HL	HL
	0.005	0.004	0.009	0.017	0.034	0.069	0.137	0.274	0.549	HL	HL
	0.001	LOW	LOW	0.003	0.007	0.014	0.027	0.055	0.110	0.219	0.439
Volume at 5ml/min	75	150	300	600	1200	2400	4800	9600	19200	38400	
Hours collected	0.25	0.5	1	2	4	8	16	32	64	128	

Use the following formula for calculating the concentration of mercury in air:

$$\frac{(\text{Meter Response}) \times 87.5}{(\text{Flow Rate of Sampling Pump}) \times \text{Time}} = \text{Concentration (mg/m}^3\text{)}$$

The following table shows the relation of flow rate and time to total volume collected.

cc/min	Total volume collected in cc								
100	6000	12000	24000	48000	72000	144000	288000	432000	
60	3600	7200	14400	28800	43200	86400	172800	259200	
20	1200	2400	4800	9600	14400	28800	57600	86400	
10	600	1200	2400	4800	7200	14400	28800	43200	
5	300	600	1200	2400	3600	7200	14400	21600	
	1	2	4	8	12	24	48	72	
	<b>HOURS</b>								

## 13. APPENDIX C - INTERNAL DIP SWITCH SETTINGS

Main circuit board RED DIP switches (SW2)

This is the red DIP switch box located at the top, center of the instrument's main circuit board.



The 431-X provides regulated film heat at both 50 Hz and 60 Hz line frequencies. This also provides two ranges of preset but unregulated film heat (100-120/200-240 volt and 110-130/220-260 volt ranges). The two ranges are available to reduce the effects of chronic low or high line voltage.

**Note:** The ranges are doubled when the AC line selector switch is set to the 220V position. The DIP switch positions 1 and 6 must be properly set.

DIP 1	DIP 6	Function
OFF	OFF	60 Hz regulated film heat (100-130/200-260VAC)
OFF	ON	50 Hz regulated film heat (102-130/205-260 VAC)
ON	OFF	50/60 Hz preset film heat (110-130/220-260 VAC)
ON	ON	50/60 Hz preset film heat (100-120/200-240 VAC)

Regulated film heat should normally be used (DIP 1 OFF) except in the few cases where extremely dirty line voltage conditions may exist. These conditions might be found where large motors are being controlled or other situations may exist where the voltage may vary outside the 100-130 VAC range with regularity. In those cases the two preset heat ranges will allow some degree of satisfactory operation.

Switch Number	Normal Position	Action
2	ON	Nanograms mode
3	ON	Displays relative (not true)voltage during regen (0-255)
4	OFF	Display L-O-H when "zero" button pressed
	ON	Display 00-99 when "zero" button pressed
5	ON	Locks into 0-10mg/m <sup>3</sup> range (survey mode)

## 14. APPENDIX D - JEROME COMMUNICATIONS SOFTWARE

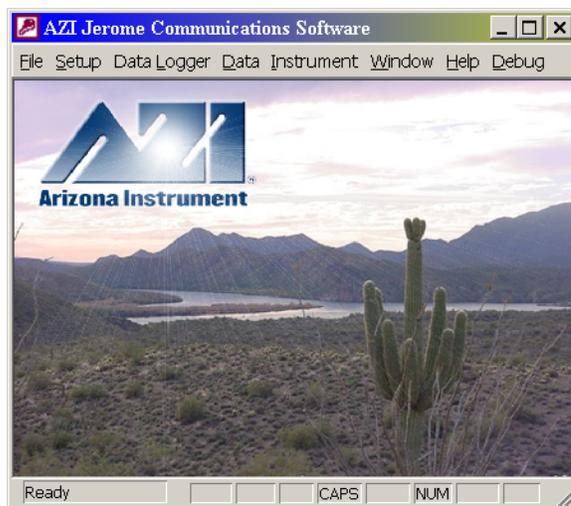
The Jerome Communications Software (JCS) is used with 431-X Mercury Vapor Analyzers that feature the communications configuration option.

- The JCS allows the user to program the instrument for unattended monitoring and to download recorded data stored in the Jerome data logger.
- Automatic sampling can be initiated every one (1) to sixty (60) minutes with programmable audible alarm levels.

The Jerome Communications Software (JCS) operates with the Jerome 431-X Mercury Vapor and Jerome 631-X Hydrogen Sulfide Analyzers that have the “Communications Configuration” option installed. The software can control instrument sampling for unattended continuous operation, collect data, graph this data in real time and perform statistical analysis.

The software can also program the Jerome Data Logger, AZI P/N 6100-0010. This optional accessory enables data storage during manual sampling or portable automatic sampling without being attached to a computer. The data logger initiates automatic sampling, triggers alarms and stores data. The logged data may then be downloaded to the computer when it is convenient.

The JCS is menu-driven and easy to use. Each display screen is designed for clarity with self-explanatory menu options, such as “Operate Instrument” or “Display Stored Data.” Select menu options using either a mouse or a track ball pointing device or a standard keyboard. The user creates records, or files, for computer storage of collected data. Data is easily retrieved for later viewing, graphing, printing or editing with spreadsheet or word processing software, (not provided). Data can be used for ongoing record keeping or for fulfilling local regulatory requirements.



Before using this software, familiarization with the operation of the Jerome Hydrogen Sulfide Analyzer or Mercury Vapor Analyzer is important. Also, prior to installation of this software you should be familiar with the personal computer and operating system you are using. If you have any questions about how to proceed, call AZI Customer Service at (800) 528-7411 or (602) 470-1414 or send an e-mail to [support@azic.com](mailto:support@azic.com) for assistance.

## **JCS Kit Contents**

- One disk containing the Jerome Communication Software
- Jerome Communication Cable, AZI P/N 6000 1055
- Cable Adaptor, SR-232, 9M/25F, AZI P/N 6000 1045
- User's manual

## **System Requirements**

Jerome 431-X with the "Communications Option."  
Windows 98 Second Edition, ME, NT, 2000 or XP  
At least one free serial port  
One free USB port

Optional equipment:

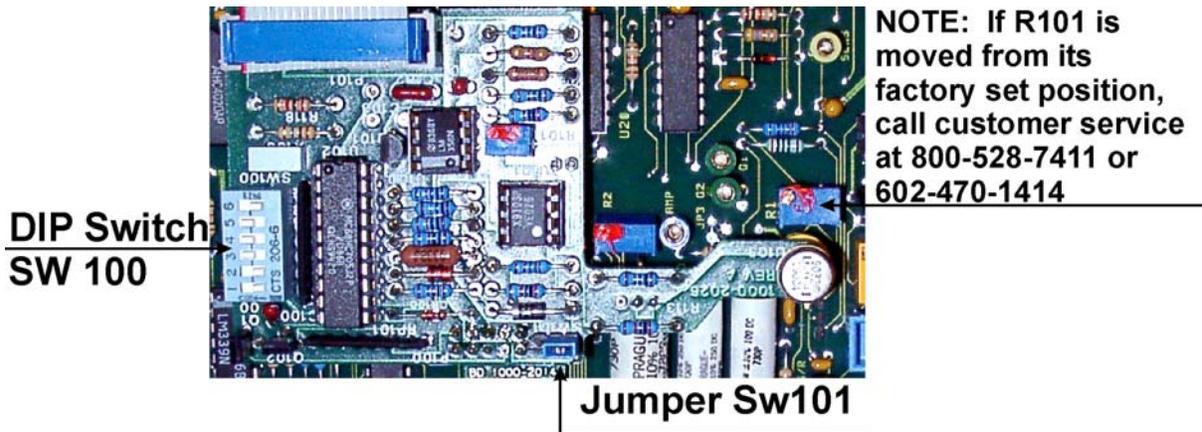
Jerome Data Logger, AZI P/N 6100-0010 (to capture data without a computer nearby)

## **Data Logger Option**

The software can also program the Jerome Data Logger (AZI P/N 6100 0010) used with the Jerome analyzer. The computer programs the data logger that then attaches to the DB-25 connector on the rear of the instrument. The data logger initiates automatic sampling, triggers alarms and stores data. This optional accessory enables portable automatic sampling without a dedicated computer.

## 15. APPENDIX E - JEROME 431-X OPTION BOARD

Proper use of this board requires that the base instrument be fully functional and set correctly for the intended operation.



### Auto-Zero

With the option board installed, the 431-X has a limited auto-zero function. This function cannot be disabled and is transparent to the user. The instrument can be manually zeroed as described in “Zero Adjust” on page 13. However, if the instrument is to be operated by personnel not familiar with the procedure or if it is operated unattended, the auto-zero function should satisfactorily zero the unit after each sensor regeneration.

### Instrument Zeroing

The Jerome 431-X has essentially three ways to zero the sensor reading before samples are taken if the option board is installed.

- The instrument automatically re-zeroes between samples so that each sample is a unique reading. To take a sample, simply press the SAMPLE button.
- The manually adjusted zero, using the switch on the top of the 431-X is used to re-establish a baseline between the reference and sensor gold films **only after a sensor regeneration**. This zero is manually adjusted by pressing the ZERO button and turning the potentiometer on the top of the instrument until the display reads 0. **Adjust only after sensor regeneration;** it is normal for H to be displayed after sampling.
- The 431-X option board provides an auto-zero feature following regeneration that is invisible to the user.

- In some cases, the instrument cannot resume sampling after regeneration. .L.L.L appears on the display when the ZERO button is pressed and the error message “manual bridge adjust needed” is added to the notes column of the JCS text file when the JCS is used. If this problem persists, it may be necessary to re-set the auto-zero.
- When necessary to re-adjust the auto-zero point:
  - Turn the instrument off.
  - Make a note of the original DIP switch settings of SW100 on the option board.
  - On red DIP switch on the control board, SW2, turn DIP switch 4 to ON.
  - Set the switches on the option board’s blue DIP box, SW100, to 1,2,6 OFF; 3,4,5 ON.
  - Turn the instrument ON.
  - Press the Zero button and adjust the potentiometer on top of the instrument until the numbers read between 5 and 7.
  - Switch option board DIP #1 OFF and ON three times, leaving it ON.
  - While pressing the ZERO button, turn the potentiometer on the option board until the numbers read between 5 and 7. Note the display will flicker one digit.
  - Return all switches to their original position.

**NOTE:** The higher the auto-zero number, the lower the sensor capacity and the more sensor regenerations are needed.

## Timed Regeneration

If the unit is to be operated unattended for extended periods, AZI recommends that the sensor be regenerated regularly. Operation under JCS or data logger control automatically regenerates saturated sensors. The option board can control regeneration on a regular basis, every 6, 12 or 24 hours.

The regeneration intervals are set through a combination of switch settings as shown in the following table:

----- SW100----- Switch #1    Switch #2		REGENERATION Interval (Hrs.)
OFF	OFF	OFF
ON	OFF	6
OFF	ON	12
ON	ON	24

## Auto-Sample

If a data logger is either not connected or is operating in the manual sampling mode, the following automatic sampling rates may be selected with option board's SW100 dip switch settings:

Dip switch settings			Sampling frequency
3	4	5	
ON	ON	ON	No automatic sampling
OFF	ON	ON	5 minutes
OFF	OFF	ON	15 minutes
OFF	ON	OFF	30 minutes
OFF	OFF	OFF	1 hour

The switches have no effect if a data logger is connected and operating in automatic sampler mode as programmed through the JCS.

## 4-20 MA Analog Output

The analog output signal at pin 18 of the 25 pin connector can be configured to provide the instrument's native mode 0-2 Volt output or the optional 4-20 mA output by setting the option board jumper (SW101) to the "V" position for voltage, or the "I" position for current. (Pin 23 is the ground pin for the analog output function. Pin 18 is positive with respect to the ground pin).

- The 0-2 Volt output circuit can drive loads of 10 k ohms or higher.
- The 4-20 mA output is a passive transmitter and requires the connected receiver to supply between 10 and 28 volts of excitation potential.

The analog output signal is based on the entire range (0-.999 mg/m<sup>3</sup>).

Note that neither analog output circuit is floating. The negative terminals of both circuits are connected to the instrument's common ground buss.

### SW101 Functions:

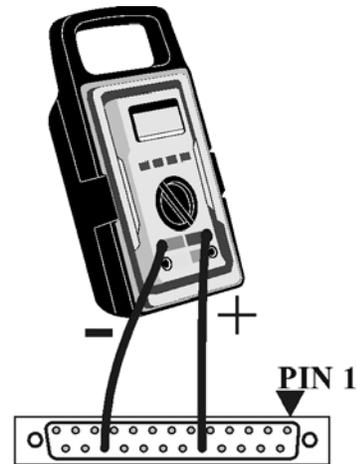
V =	0-2V analog output
I =	4-20 ma analog output

Jerome 431-X instruments shipped after early 1995, are capable of providing 0-2 volts analog output. Instruments shipped before that time can be upgraded by a firmware update and adjustment.

Instruments that are capable of 0-2 volt output can be upgraded to the 4-20 mA output with the addition of an option board upgrade. This must be installed at the factory.

**Connection and Setup:**

- 0-2 volt devices connect as shown in Figure 1. If the instrument includes an option board, be sure its analog jumper (SW101) is set to the “V” position.



**REAR OF CONNECTOR,  
CONNECT PINS 18 AND 23 FOR  
0-2 VOLT OUTPUT.  
JUMPER ON BOARD IS AT “V”**

**Figure 1**

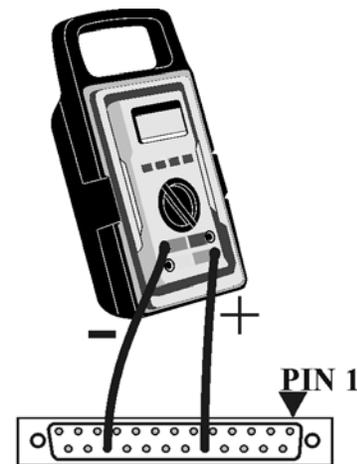
- The 4-20 mA active receivers connect as shown in Figure 2. The active receiver contains a voltage source to power the loop current. The receiver must have an isolated input circuit. That is, it must not be connected to ground or to a voltage source referenced to ground. Be sure that jumper SW101 is set to the “I” position before power is applied.

- 431X reading                      Current  
0.000mg/m<sup>3</sup>                      4mA  
1.000mg/m<sup>3</sup>                      20mA

- The 431X current formula is:  
16mA \* Display Reading + 4mA = (Current)

**Example:**

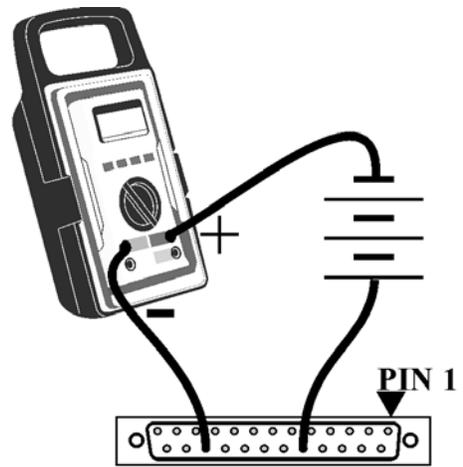
A reading of .100mg/m<sup>3</sup> would produce an output current of,  
16mA \* 0.1 +4mA = 5.6mA



**REAR OF CONNECTOR,  
CONNECT PINS 18 AND 23 FOR  
4-20 mA OUTPUT.  
JUMPER ON BOARD IS AT “I”**

**Figure 2**

- The 4-20 mA passive receivers do not contain a voltage source to power the loop current. They require the addition of a separate isolated power supply. Typically a supply that delivers 15 to 20 volts DC at 50 mA is sufficient. Wire these as in Figure 3. Note that some 12-volt DC wall transformers (as used on portable equipment) may deliver 15 to 20 volts when they are lightly loaded. The Digi-Key T5.9-PIP-ND is a commonly available example of a 12 volt 200mA supply that will deliver around 18 volts nominal when loaded below 20 mA.
- Be sure that both the power supply used and the passive receiver are floating (not connected to earth ground). If either is not floating, the circuit will not work and damage may occur.
- Ensure that jumper SW101 is set to the “I” position before power-up.



**REAR OF CONNECTOR,  
CONNECT PINS 18 AND 23 FOR  
4-20 mA OUTPUT.  
JUMPER ON BOARD IS AT “I”  
Figure 3**

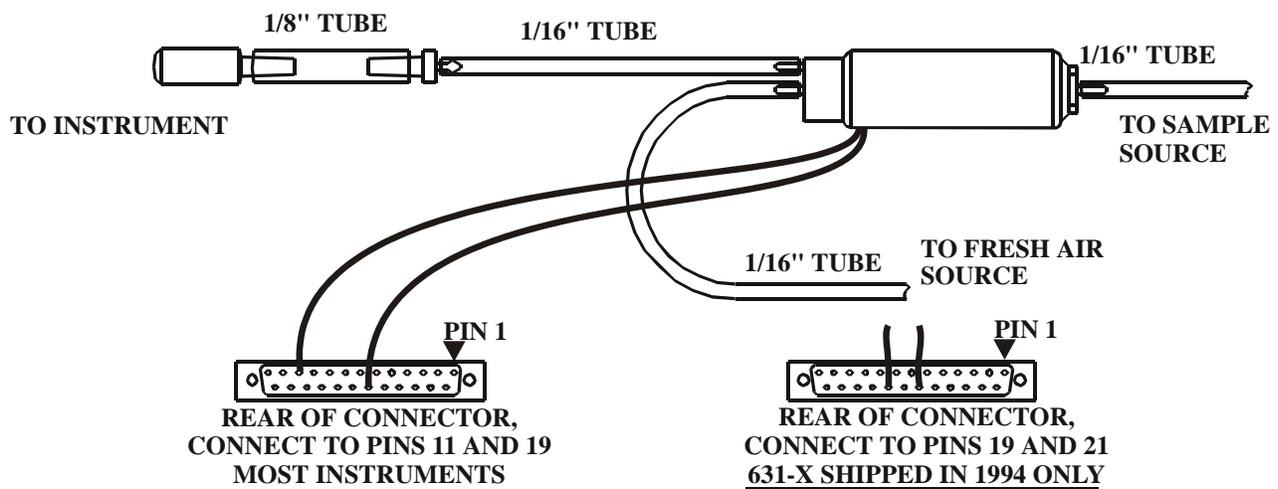
## Fresh Air Solenoid

An external three-way solenoid can be used to provide fresh air or conditioned air during sensor regeneration. This may be necessary if the sample stream lacks molecular oxygen. A low current six volt DC solenoid, connected between pins 19 and 11 of the 25 pin rear panel connector, will be energized during the regeneration cycle if the option board SW100 switch 6 is placed in the OFF position.

If needed, the circuit may be built from the following components and configured as shown in the following diagram. It will only function if the option board is installed in the 431-X instrument.

Required Parts:	Suggested Part	Similar AZI P/N
1 solenoid, 6volt 3way	Angar P/N 407569	1300 1004
1/8" to 1/16" tubing adaptor	Any	1300 0025
1/2" clamp, adhesive mount	Any	6000 0013
1/8" tube to instrument adaptor	Any	1400 3010
3" 1/8" tubing	Tygon Formula R3603	2500 3001
A/R 1/16" tubing	Tygon Formula R3603	2500 3002
1 25 pin male DB-25 connector Solder-cup style	AMP 747912-2	None *
1 connector hood	AMP 749626-2	None *

\* These are types not stocked by AZI, but should be available overnight from many AMP stocking distributors such as Digi-Key Corporation. There are multiple suitable alternatives such as Radio Shack's 276-1547 and 276-1549.



## DC Power Operation

Instruments with the 431-X option board modification can be used with any +12 VDC source for continuous operation, if the AZI Power Inverter Kit, P/N Y031 0902 is installed along with the option board. To preserve the life of the DC power source, usually a car or truck battery, the power inverter will switch on automatically to supply the AC necessary for regeneration only. The external switch on the inverter should always be OFF to preserve battery life during normal sampling.

To work with the power inverter kit, place option board SW100 DIP Switch #6 to the ON position.

When the instrument starts a regeneration with option board SW100 DIP Switch #6 ON, the instrument sends a signal to close the relay on the DC Power Adaptor, AZI P/N 1000 0089, mounted between the data logger and the instrument. This switches the power inverter ON using the inverter's internal switch.

**NOTE:** When this mode is enabled, the instrument does NOT check for 115 VAC for the regeneration. If there is no AC power to the instrument, and a regeneration is initiated, the instrument will flash .H.H.H (rather than .P.P.P), however the sensor will not heat, nor will the sensor be cleaned.

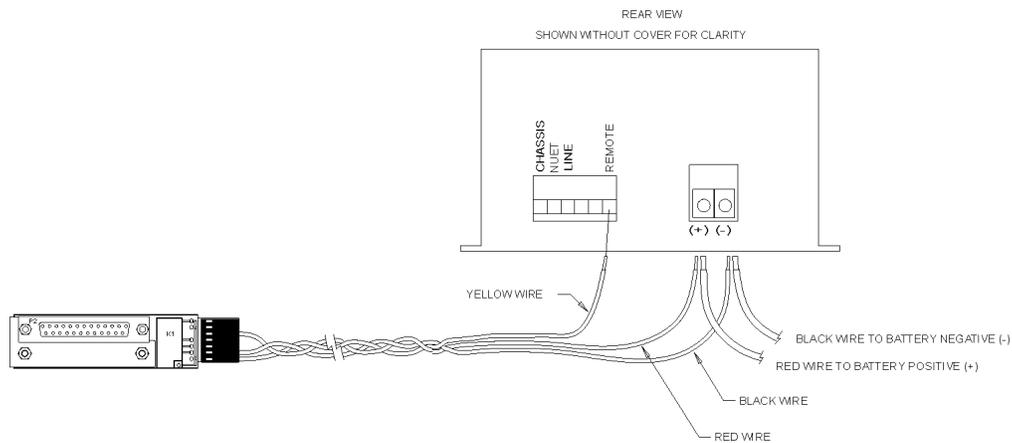
### DC Power Adaptor Kit, AZI P/N Y031 0902

- The DC power adaptor kit consists of:
  - DC Power Adaptor, P/N 1000 0089
  - DC Power Inverter, P/N 4000 1021
  - DC Power Cable Assembly, P/N 6000 1093

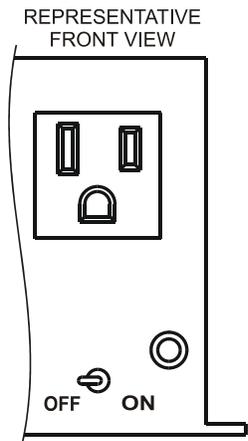
#### Installation

- Ensure that the instrument's option board switches are set correctly for the intended operation with the option board's SW100 DIP Switch #6 set to "ON" for DC operation.
- Mount the interface board to the rear of the instrument. Tighten the mounting screws.
- Place or mount the DC/AC power inverter in a secure position near the instrument.
- Connect the cable from the DC/AC power inverter to the matching connector on the interface board. Note that the connectors are keyed to prevent improper connection.
- Plug the instrument's AC power cord into the power inverter and connect it to the instrument.

Ensure that the inverter's power switch is in the "OFF" position. LEAVE the power switch in the "OFF" position at all times. The interface board will activate the inverter when necessary. If the inverter power switch is placed in the "ON" position, it will cause a continuous drain on the external 12-volt power system.



- Remove the screws from the rear cover of the inverter and remove the cover.
- Place the wires from the external DC source (battery) and the wires from the DC power cable through the holes in the end plate.
- Connect cables from the external 12-volt power source and the DC power cable assembly to the appropriate positive (+) and negative (-) terminals on the back of the inverter and tighten the hold down screws.
- Connect the yellow wire from the DC power cable to the "REMOTE" terminal on the power inverter and tighten the hold down screw.
- Reinstall the cover.
- If the external 12volt lines are not powered, power them now. (Connect them to the battery)
- Connect the instrument's AC power cord between the instrument and the front of the power inverter.
- Turn the instrument "ON."
- Press the "REGEN" switch on the instrument. Inverter operation can be verified in either of two ways:
  - Immediately after pressing "REGEN" the inverter will intermittently "sing." This tone slowly becomes nearly continuous and then ends after 64 seconds.
  - If the area is noisy, use a voltmeter or test lamp to verify that approximately 115 volts is present for about 64 seconds, starting when the "REGEN" switch is pressed.
- Allow the instrument to complete its regeneration before turning it off.
- With the instrument turned off, complete the installation (i.e. connect data logger, communications cables, or other devices and ensure that the DIP switches for the instrument and option board are set correctly.



## 16. APPENDIX F: CALCULATION OF DYNACALIBRATOR MERCURY CONCENTRATION

### UNIT DEFINITIONS

Constants	Value	Description
R (L*atm/mol/°C)	0.082057	Universal gas constant
MV <sub>NTP</sub> (L/mol)	24.453	Molar volume at NTP
MW <sub>HG</sub> (g/mol)	200.59	Molar weight
K <sub>HG</sub> (L/g)	0.122	Molar constant at site
P <sub>SITE</sub> (mm Hg)	730	Atmospheric pressure
T <sub>SITE</sub> (°K)	298	Air temperature in rotometer
F <sub>CarrierTopNTP</sub> (SLM)	0.173	Carrier flow at NTP, top float
F <sub>CarrierBotNTP</sub> (SLM)	0.173	Carrier flow at NTP, bottom float
PermRate (ng/min)	745	Permeation rate at 100°C (+/- 2%)
FDilutionTop <sub>CAL</sub> (SLM)		Calibrated dilution flow at NTP, top float
FDilutionBot <sub>CAL</sub> (SLM)		Calibrated dilution flow at NTP, bottom float
FDilutionTop <sub>NTP</sub> (SLM)		Corrected dilution flow at NTP, top float
FDilutionBot <sub>NTP</sub> (SLM)		Corrected dilution flow at NTP, bottom float
FTotalTop <sub>NTP</sub> (SLM)		Corrected total flow at NTP, top float
FTotalBot <sub>NTP</sub> (SLM)		Corrected total flow at NTP, bottom float
ConcTop <sub>NTP</sub> (ng/m <sup>3</sup> )		Concentration at NTP, top float
ConcBot <sub>NTP</sub> (ng/m <sup>3</sup> )		Concentration at NTP, bottom float
Normal Temperature and Pressure (NTP) is at 25°C and 760 mm Hg		

### CALCULATIONS

Equations	
Perfect gas law	
	$P * V = n * R * T$
	$V / n = (R * T) / P$
	$MV = (R * T) / P$
Correct dilution flow rotometer to NTP	
	$FDilution_{NTP} = FDilution * \text{sqrt}(P_{NTP} / 760) * \text{sqrt}(298 / T_{NTP})$
Correction for carrier flow is not required because flow is not set by rotometer	
Total mass flow at NTP	
	$FTotal_{NTP} = FCarrier_{NTP} + FDilution_{NTP}$
Concentration at NTP	
	$Conc_{NTP} = PermRate / FTotal_{NTP}$

Flowmeter Calibration Data At Site		Total Mass Flow At NTP		Concentration At NTP	
FDilutionTop <sub>NTP</sub> (SLM)	FDilutionBot <sub>NTP</sub> (SLM)	FTotalTop <sub>NTP</sub> (SLM)	FTotalBot <sub>NTP</sub> (SLM)	ConcTop <sub>NTP</sub> (mg/m <sup>3</sup> )	ConcBot <sub>NTP</sub> (mg/m <sup>3</sup> )
0.000	0.000	0.173	0.173	4.306	4.306
0.308	0.247	0.481	0.420	1.550	1.772
0.615	0.495	0.788	0.668	0.945	1.115
1.004	1.098	1.177	1.271	0.633	0.586
1.392	1.701	1.565	1.874	0.476	0.397
1.777	2.348	1.950	2.521	0.382	0.295
2.162	2.995	2.335	3.168	0.319	0.235
2.555	3.610	2.728	3.783	0.273	0.197
2.947	4.225	3.120	4.398	0.239	0.169
3.315	4.869	3.488	5.042	0.214	0.148
3.683	5.513	3.856	5.686	0.193	0.131
4.054	6.156	4.227	6.329	0.176	0.118
4.425	6.799	4.598	6.972	0.162	0.107
4.781	7.461	4.954	7.634	0.150	0.098
5.137	8.123	5.310	8.296	0.140	0.090
5.501	8.799	5.674	8.972	0.131	0.083
5.865	9.474	6.038	9.647	0.123	0.077
6.219	10.172	6.392	10.345	0.117	0.072
6.572	10.869	6.745	11.042	0.110	0.067
6.931	11.566	7.104	11.739	0.105	0.063
7.289	12.264	7.462	12.437	0.100	0.060
7.642	13.005	7.815	13.178	0.095	0.057
7.994	13.746	8.167	13.919	0.091	0.054
8.351	14.527	8.524	14.700	0.087	0.051
8.707	15.308	8.880	15.481	0.084	0.048
9.037	16.069	9.210	16.242	0.081	0.046
9.367	16.830	9.540	17.003	0.078	0.044
9.707	17.635	9.880	17.808	0.075	0.042
10.047	18.441	10.220	18.614	0.073	0.040
10.405	19.300	10.578	19.473	0.070	0.038
10.763	20.159	10.936	20.332	0.068	0.037

## **17. WARRANTY**

Arizona Instrument LLC (seller) warrants to buyer that Jerome products delivered pursuant to this agreement shall, at the time of delivery, and for a period of one (1) year. Thereafter (the Internal Battery Pack, where applicable, is warranted for a period of ninety [90] days only), to be free from defects in material or workmanship and shall conform to seller's specifications or such other specifications as seller has agreed to in writing. Seller's obligations with respect to claims under this warranty shall be limited, at seller's option, either to the replacement of defective or non-conforming product or to an appropriate credit for the purchase price thereof subject to the provisions of seller's Warranty Policy as amended from time to time, said Policy being incorporated herein by reference.

Returned products under warranty claims will be shipped to seller's plant by buyer at buyer's expense and shall be accompanied by a statement of the reason for the return and an approved Return Material Authorization Number issued by seller. Buyer remains responsible for payment for products not accepted for warranty adjustment, handling costs, and freight costs associated therewith.

Notwithstanding the foregoing, no warranty shall be enforceable in the event that product has been subjected to environmental or stress testing by buyer or any third party without written approval of seller prior to such testing. Further, no warranty shall be enforceable if the alleged defect is found to have occurred because of misuse, neglect, improper installation, repair, alteration, accident, or improper return handling procedure by buyer.

Discontinued product is warranted only for a credit or replacement at seller's option.

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**Arizona Instrument LLC**

**Jerome 431-X Mercury Vapor Analyzer Operation Manual**

**Part Number 700-0037**

**March 2005**

If you have any questions regarding the operation of this instrument, please call our toll free number (800) 528-7411. Internationally, call (602) 470-1414 or fax (602) 470-1888.

## **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 ( $\leq 2$  centimeters and  $\leq 3$  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 determine 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 required 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 ( $\pm 2^\circ\text{C}$ ) 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.

#### Sample Collection

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 pan (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

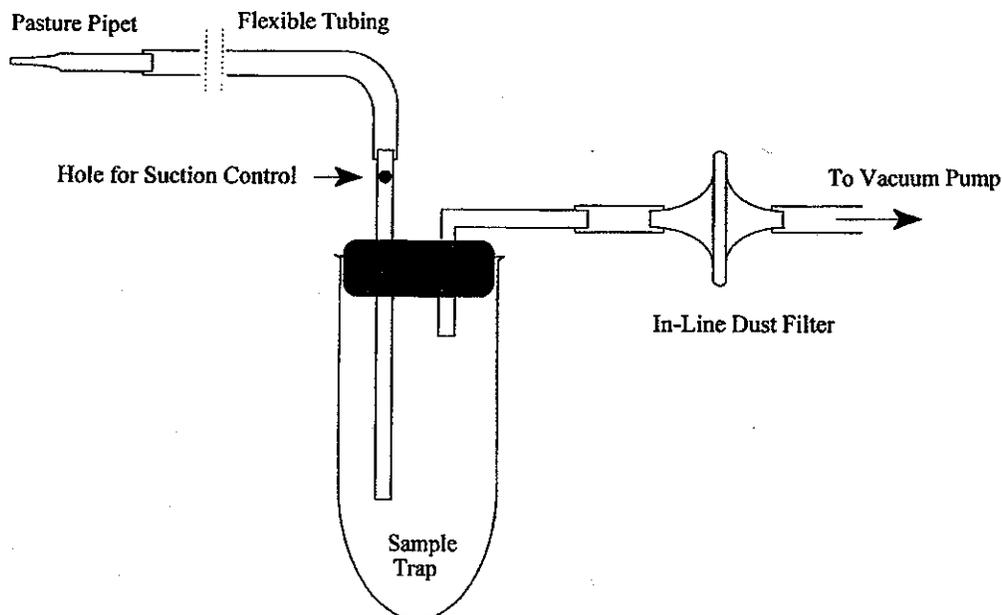
The trap presented in Figure 1 is a convenient and thorough way for collecting and removing concrete powder from drilled holes. The trap system is designed to allow for control of the suction from the vacuum pump and easy trap clean-out between samples. Note, by placing a hole in the inlet tube (see 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

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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  $\geq$  practical quantitation limit (PQL), then the control limit for RPD is  $\leq 50\%$ ,
- 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  $\geq$  PQL, then the control limit for RPD is  $\leq 25\%$ ,
- 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

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

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.

## 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

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## 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  $\text{HgCl}_2$ . Mercury is then reduced with stannous chloride from  $\text{HgCl}_2$  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

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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.

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**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<sub>3</sub>).** 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<sub>2</sub>•2H<sub>2</sub>O).** Fisher T142-500, or equivalent. (*Analytical reagent*)
- 8.5 Stannous Chloride Reduction Solution (2%).** Add 20 g of SnCl<sub>2</sub>•2H<sub>2</sub>O 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*)

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**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  $\geq 0.995$ . 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

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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

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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  $\leq 20\%$  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)

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- 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<sub>3</sub> 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.

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- 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 ( $\mu\text{g/L}$ )
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  $\text{SnCl}_2$  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.

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- 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:

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

Where:

A = Digestion final volume in mL, typically 50 mL  
B = Concentration of sample from instrument read-out in  $\mu\text{g/L}$   
DF = Dilution Factor  
V = Volume in  $\text{M}^3$

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

- 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.

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## 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, January 1999. 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:

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- *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 <sub>12</sub> H <sub>9</sub> Cl	27323-18-8
Dichlorobiphenyl	C <sub>12</sub> H <sub>8</sub> Cl <sub>2</sub>	25512-42-9
Trichlorobiphenyl	C <sub>12</sub> H <sub>7</sub> Cl <sub>3</sub>	25323-68-6
Tetrachlorobiphenyl	C <sub>12</sub> H <sub>6</sub> Cl <sub>4</sub>	26914-33-0
Pentachlorobiphenyl	C <sub>12</sub> H <sub>5</sub> Cl <sub>5</sub>	25429-29-2
Hexachlorobiphenyl	C <sub>12</sub> H <sub>4</sub> Cl <sub>6</sub>	26601-64-9
Heptachlorobiphenyl	C <sub>12</sub> H <sub>3</sub> Cl <sub>7</sub>	28655-71-2
Octachlorobiphenyl	C <sub>12</sub> H <sub>2</sub> Cl <sub>8</sub>	31472-83-0
Nonachlorobiphenyl	C <sub>12</sub> H <sub>1</sub> Cl <sub>9</sub>	53742-07-7
Decachlorobiphenyl	C <sub>12</sub> Cl <sub>10</sub>	2051-24-3

Pesticides	Formula	CAS #
4,4'-DDD	C <sub>14</sub> H <sub>10</sub> Cl <sub>4</sub>	72-54-8

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4,4'-DDE	C <sub>14</sub> H <sub>8</sub> Cl <sub>4</sub>	72-55-9
4,4'-DDT	C <sub>14</sub> H <sub>9</sub> Cl <sub>5</sub>	50-29-3
Aldrin	C <sub>12</sub> H <sub>8</sub> Cl <sub>6</sub>	309-00-2
Alpha-BHC	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	319-84-6
Alpha-Chlordane	C <sub>10</sub> H <sub>6</sub> Cl <sub>8</sub>	5103-71-9
Beta-BHC	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	319-85-7
Delta-BHC	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	319-86-8
Dieldrin	C <sub>12</sub> H <sub>8</sub> Cl <sub>6</sub> O	60-57-1
Endosulfan I	C <sub>9</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>3</sub> S	959-98-8
Endosulfan II	C <sub>8</sub> H <sub>6</sub> Cl <sub>6</sub> O <sub>3</sub> S	33213-65-9
Endosulfan Sulfate	C <sub>9</sub> H <sub>4</sub> Cl <sub>6</sub> O <sub>4</sub> S	1031-07-8
Endrin	C <sub>12</sub> H <sub>8</sub> Cl <sub>6</sub> O	72-20-8
Endrin Aldehyde	C <sub>12</sub> H <sub>8</sub> Cl <sub>6</sub> O	7421-93-4
Endrin Ketone	C <sub>12</sub> H <sub>8</sub> Cl <sub>6</sub> O	53494-70-5
Gamma-BHC (Lindane)	C <sub>6</sub> H <sub>6</sub> Cl <sub>6</sub>	58-89-9
Gamma Chlordane	C <sub>10</sub> H <sub>6</sub> Cl <sub>8</sub>	5103-74-2
Heptachlor	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub>	76-44-8
Heptachlor Epoxide	C <sub>10</sub> H <sub>5</sub> Cl <sub>7</sub> O	1024-57-3
Methoxychlor	C <sub>16</sub> H <sub>15</sub> Cl <sub>3</sub> O <sub>2</sub>	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 than those specified in Method 680.

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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 C12 – C18 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

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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

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**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 ID 0.25 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  $-10^{\circ}$  –  $-20^{\circ}$ C 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 Chlorpyrifos 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	
<b>136Congeners/209Congeners</b>			
BZ 1 (1 <sup>st</sup> 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 (1 <sup>st</sup> 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
<b>Add'l Ret. Window PCB</b>	<b>Isomer Group</b>	<b>Stock Conc. ng/mL</b>	
<b>136Congeners/209Congeners</b>			
BZ 3 (last Mono)	Cl1	400	1000 ng/mL
BZ 10 (1 <sup>st</sup> Di)	Cl2	400	1000 ng/mL
BZ 15 (last Di)	Cl2	400	1000 ng/mL
BZ 19 (1 <sup>st</sup> Tri)	Cl3	400	1000 ng/mL
BZ 37 (last Tri)	Cl3	400	1000 ng/mL
BZ 54 (1 <sup>st</sup> Tetra)	Cl4	400	1000 ng/mL
BZ 77 (last Tetra)	Cl4	400	1000 ng/mL
BZ 104 (1 <sup>st</sup> Penta)	Cl5	400	1000 ng/mL
BZ 126 (last Penta)	Cl5	400	1000 ng/mL
BZ 155 (1 <sup>st</sup> 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 (1 <sup>st</sup> Octa)	Cl8	400	1000 ng/mL
BZ 205 (last Octa)	Cl8	400	1000 ng/mL
BZ 208 (1 <sup>st</sup> Nona)	Cl9	400	1000 ng/mL
<b>Surrogates</b>			
DBOB	N/A	5000 ng/mL	
BZ 198	Cl8	5000 ng/mL	
<b>Carbon-labeled Surrogates</b>			
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**

<u>Calibration Level</u>	<u>136 Cong. /Surr Stock</u> 400ng/mL	<u>Calibration L6</u> (200ug/L)	<u>Final Volume</u>
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**

<u>Calibration Level</u>	<u>Surr. Stock</u> 5000ng/mL	<u>209 Cong. Stock</u> 1000ng/mL	<u>Calibration L6</u> (200ug/L)	<u>Final Volume</u>
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 than 10x the blank concentration for the

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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 exceedances.

## 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 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.

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

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**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.

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

#### **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

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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:

$$\% \text{ Recovery} = \text{Found Amount} / \text{True Value} \times 100$$

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.

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- 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 observed, 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  $\pm 10$  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  $\pm 50$  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  $\pm 50$  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<sub>s</sub>) at each concentration level, for each PCB congener. Average the RRF<sub>s</sub> from the initial multi-point calibration, to generate mean RRF<sub>s</sub>, for quantification of each PCB congener. Follow the same calculations for each surrogate compound. The RRF<sub>i</sub> for the quantification of each Homolog group is the RRF<sub>i</sub> of the PCB calibration congener assigned to that Homolog group (*i.e.*, Trichlorobiphenyls are quantified using the RRF<sub>i</sub> of the PCB calibration congener BZ29, which is associated with the Trichlorobiphenyl group). The RRF<sub>s</sub> are based on the internal standard compounds, and are calculated using the formula below. (The relative response factors for the continuing calibration verifications (RRF<sub>Cs</sub>) 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.

$$\text{RSD} = \text{SD} / \text{mean RRF}_1 \times 100$$

where:

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

$$\text{RRF}_1 = (\text{A}_c \times \text{C}_{\text{IS}}) / (\text{A}_{\text{IS}} \times \text{C}_c)$$

where:

A<sub>c</sub> = Area of the characteristic ion for the standard compound to be measured.

A<sub>IS</sub> = Area of the characteristic ion for the representative internal standard compound.

C<sub>IS</sub> = Concentration of the representative internal standard compound (ng/mL).

C<sub>c</sub> = 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<sub>IS</sub>, calculate the **Sample Extract Amount** for each Pesticide, PCB congener or Homolog group and surrogate in the sample extracts using the following formula:

$$Q_e = (\text{A}_a \times Q_{\text{IS}}) / (\text{A}_{\text{IS}} \times \text{RRF}_1)$$

where:

Q<sub>e</sub> = Sample extract concentration (ng/mL) of target analyte, from quantitation report.

A<sub>a</sub> = Area of the characteristic ion for the target analyte.

A<sub>IS</sub> = Area of the characteristic ion for the representative internal standard compound.

Q<sub>IS</sub> = Concentration (ng/mL) of representative internal standard compound, from quantitation report.

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

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

where:

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

V<sub>s</sub> = 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<sub>is</sub> = 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.

$$\text{Percent Difference (\%D)} = [(RRF_1 - RRF_C) / RRF_1] \times 100$$

where:

RRF<sub>1</sub> = Mean response factor from initial calibration.

RRF<sub>C</sub> = 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:

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

## 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	<u>Surrogate Compounds</u>	
Dichlorobiphenyls	B	A	DBOB	A
Trichlorobiphenyls	B	A	BZ 198	B
Tetrachlorobiphenyls	B	A	<u>Carbon-labeled Surrogates</u>	
Pentachlorobiphenyls	B	A	BZ 19	A
Hexachlorobiphenyls	B	A	BZ 202	B
Heptachlorobiphenyls	B	B		
Octachlorobiphenyls	B	B	<u>Carbon-labeled ISs</u>	
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 <sup>o</sup> Ion	2 <sup>o</sup> 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

*Printouts of this document may be out of date and should be considered uncontrolled. To accomplish work, the published version of the document should be viewed online.*

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:

<u>Analyte</u>	<u>CAS Number</u>
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:

	<b>Absolute #</b>	<b>Conc. ug/ml</b>
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:

	<b>Volume (ul) of Stock Solution</b>	<b>Final Volume (hexane)</b>
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:

	<b>Conc. ug/ml</b>
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		Final Vol.	<u>μL Internal Standard</u>
	<u>Working Std</u>	<u>μL DCAA</u>		
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  $\leq 20\%$  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  $\pm 15\%$ . 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  $\pm 15\%$  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  $\pm 15\%$  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 Pentafluorobenzoylation Derivatization", SW-846, Revision 1, December 1996.



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**Title: Analysis of Total Organic Carbon by SM5310C, E415.1, and E9060A**

QA Officer: *Jung Guenette* Date 4-10-14  
Laboratory Director: *Jim Loh* Date 4-10-14  
Author: *JL Miller* Date 4/10/14  
Analyst: \_\_\_\_\_ Date \_\_\_\_\_

By signing this SOP, the analyst has read and understood the latest version of the test method; has read and understood this SOP; acknowledges receipt of this SOP; and is responsible for the implementation of this SOP.

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Revision History:

Revision	Changes	Date
1	Updated SM revision to 2000& 2011 and qc criteria	1/13
2	pH check clarification, LOD/Q & MDL locations	03/14

Title: **Analysis of Total Organic Carbon by SM5310C, E415.1, and E9060A**

## **1.0 Purpose and Applicability**

- 1.1 This method is applicable to groundwater, drinking water, wastewater, and other aqueous samples.
- 1.2 This SOP covers both Drinking water and Wastewater approved SM5310C revisions and associated QC. The procedures are accurately represented in this SOP, and cover both versions. The QC from the most stringent version will be followed. See Section 3, Applicable References, for the method revision dates applicable to this SOP.
- 1.3 This method is for use only by or under the supervision of analysts experienced in the use of the instrumentation and in the interpretation of the resulting data. Each analyst must demonstrate the ability to generate acceptable results with this method.

## **2.0 Definitions**

- 2.1 Initial Calibration Verification (Instrument Performance Check) (ICV): A standard at a mid-point in the calibration curve to verify the integrity of the curve. The ICV is made from a second source standard. All analytes must have a different lot number than the one that made the calibration curve.
- 2.2 Continuing Calibration Verification (CCV): A CCV is analyzed at the beginning of each analytical batch, when verifying the previous calibration. The CCV is analyzed at a mid-point of the initial calibration curve. The concentration of the CCV may be varied in order to reveal any concentration specific biases.
- 2.3 Calibration Standard (ICal): Standard solutions prepared from the stock standard at levels corresponding to the calibration curve. These are used to calibrate the instrument response with respect to analyte concentration.
- 2.4 Laboratory Control Sample (Laboratory Fortified Blank) (LCS): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly as a sample, its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 2.5 Laboratory Control Sample Duplicate (Laboratory Fortified Blank Duplicate) (LCSD): See definition above.
- 2.6 Prep Blank (Laboratory Reagent Blank) (Method Blank) (PB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample, including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus at levels which may interfere with the analysis. The concentrations found in the PB must be below the reporting limit for each analyte.
- 2.7 Matrix Spike: An aliquot of a field sample spiked with a known amount of a

Title: **Analysis of Total Organic Carbon by SM5310C, E415.1, and E9060A**

- standard. These are analyzed at the customer's request.
- 2.8 Matrix Duplicate: A duplicate analysis of a sample in which the percent difference is calculated. The duplicate can serve as demonstrating the precision of the method. These are analyzed at the customer's request.
  - 2.9 Replicate Analysis: All calibration standards, QC, and samples are analyzed in duplicate. This is done by the instrument during analysis from one vial. The average of the two results is used for the final reported value. The relative percent difference between the two results must be <10% or the result is qualified accordingly.
  - 2.10 Carry-over: when a sample is contaminated by analytes left in the purging device, trap or syringe by a previous sample analysis with high levels of analytes.
  - 2.11 Interferences: Occurrences affecting results on a sample analysis. Examples include: Impurities in the purge gas, solvent vapors in the lab, diffusion of volatile organic compounds through the septum seal into the sample, carry-over from samples with high levels of analytes.
  - 2.12 Trip blank (field reagent blank): a VOC vial filled with reagent water is carried through sampling and handling protocol, and then analyzed along with the other project samples to check for cross contamination.

**3.0 Applicable Documents/References**

- 3.1 Standard Methods for the Examination of Water and Wastewater, SM 5310C-2000, SM5310C-2011.
- 3.2 EPA Method 415.1
- 3.3 SW846 Method 9060A
- 3.4 ARA SOP QA-400 Sample Receiving and Identification
- 3.5 ARA QA Manual

**4.0 Materials and Apparatus**

- 4.1 Equipment:
  - 4.1.1 Syringes: Gastight 10uL, 25uL, 50uL, 250uL, 500uL, & 1mL
  - 4.1.2 Various pipettes
  - 4.1.3 Volumetric flasks: Grade A, 50mL & 100mL
  - 4.1.4 VOA vials: 43mL glass with Teflon lined silicon septum
  - 4.1.5 pH paper
  - 4.1.6 OI Analytical Aurora 1030 Wet Oxidation TOC Analyzer
  - 4.1.7 OI Analytical 1088 Rotary Autosampler
- 4.2 Reagents/Standards
  - 4.2.1 Purified/deionized water (reagent grade) Source: Made In-House
  - 4.2.2 UHP Nitrogen

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- 4.2.3 Hydrochloric Acid (HCl) Trace metal grade.
- 4.2.4 Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>) Trace metal grade.
- 4.2.5 Total Organic Carbon Standard Solution at 1000mg/L: Purchased pre-made from a reputable supplier. Directions for manual preparation can be seen in 4.2.6.
- 4.2.6 Total Organic Carbon Standard Solution at 1000mg/L: Dissolve 2.13g of reagent grade potassium hydrogen phthalate in a 1L class A volumetric flask containing 500mLs of reagent grade water. Add 1mL of concentrated sulfuric acid and dilute to a final volume of 1L.
- 4.2.7 Sodium Persulfate Solution (10%): The solution is prepared by adding 100g of sodium persulfate to water and bringing to a final volume of 1L.
- 4.2.8 Phosphoric Acid (5%): The solution is prepared by adding 59mLs of 85% phosphoric acid to water and bringing to a final volume of 1L.

**5.0 Method/Calibration/Interferences**

- 5.1 Method Summary: The organic compounds present in an aqueous sample are converted to carbon dioxide through ultraviolet promoted persulfate oxidation. The product of this oxidation is carried to the non-dispersive infrared detector for quantitation.
  - 5.1.1 Samples are collected in duplicate in sulfuric acid pre-preserved 40mL VOA vials.
  - 5.1.2 Preserved samples must be analyzed within 28 days of sampling. Unpreserved samples must be analyzed within 7 days of sampling.
  - 5.1.3 Shipment of samples must follow the requirements outlined in the sample login SOP.

**5.2 Calibration Procedure**

- 5.2.1 The calibration standard from section 4.2.5 is used to construct a calibration curve with a minimum of 5 points. The typical points can be seen in the table below:

Calibration Level (mg/L)	Calibration Standard Volume (mL)	Final Volume (mL)
1	0.1	100
2	0.2	100
5	0.25	50
10	0.5	50
50	2.5	50

- 5.2.2 The analyst's discretion is used in selecting the curve fit and weighting which best represents the data, paying particular attention to the low end of

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the calibration range. Usually, the inverse of concentration is most appropriate weighting. If linear fit is to be used to create the curve then the calibration coefficient needs to be 0.995 or greater. If average of response factors is used to create the curve, then the RSD of the response factors must not exceed 20%.

5.2.3 The typical reporting limit for TOC is 1mg/L.

5.3 Interferences

5.3.1 Carbonate and bicarbonate carbon will cause interferences and must be removed prior to the final calculation. This is accomplished by the acidification of the sample vial and purging with nitrogen.

**6.0 Procedure**

6.1 Instrument Setup

6.1.1 When idle, the instrument is in “gas saver” mode. To begin using the instrument, it must be removed from gas saver mode.

6.1.2 Depending on how long the instrument has been idle, fresh standards should be prepared and the lines and syringes should be purged.

6.2 Setting up a sequence: The sequence, which contains the QC and sample information, is entered in the Aurora software. The Aurora software is located on the computer used for data acquisition. To create a sequence, “Editor” is selected from the Aurora software, then “Sequence”. Old information is deleted or changed, and new sample information is added, including method, auto-sampler position, sample name and volume. The sequence should always begin with a “clean-up” analysis to rinse the lines and sparge tube. After the clean-up has run, a reagent blank should be analyzed. This analysis is used by the instrument to determine the TOC content present in that day’s reagents. The sequence is saved using the date the sequence starts. Analysis may now begin.

6.3 Preservation Check: After the sequence is complete, the remaining sample in the VOA vials is checked for adequate preservation using pH paper. The result is recorded in the instrument run log. Any sample with a pH above 2 is flagged in the final report.

6.4 Data Analysis: After a sample analysis is complete, the data is evaluated by someone experienced with the OI software.

**7.0 Quality Control Requirements**

7.1 Initial Demonstration of Capability: This is used to characterize instrument performance (linear calibration ranges and analysis of the ICV) and laboratory performance (MDLs) prior to analysis by this method.

7.2 Initial Calibration Verification: The ICV must be from a second source or a different lot number than the standard used for calibration and be within  $\pm 15\%$  of

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the expected concentration.

- 7.3 Method Detection Limit: The MDLs are established for all analytes. Reagent water is fortified at a concentration of two to three times the estimated method detection limit. MDLs should be determined initially or whenever there is a significant change in the instrumental configuration.

7.3.1 To Determine an MDL, a minimum of seven replicates of the fortified reagent water are processed through entire analytical system. Calculate the MDL for each analyte as follows:

$$\text{MDL} = (t) * (s)$$

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. For seven replicates t = 3.14.

s = standard deviation of the replicate analyses.

The MDL data is stored electronically in the QA/MDL file folder.

7.4 Laboratory Performance Quality Control and Corrective Actions

7.4.1 Continuing Calibration Verification: The CCV must be within  $\pm 10\%$  of the expected concentration. If the CCV performed at the beginning of a run is unacceptable, the CCV should be remade and re-analyzed, if acceptable the batch can continue, otherwise a new initial calibration is required.

7.4.2 Continuing Calibration Blank: The CCB is analyzed after the CCV. Contamination in the CCB must be lower than the Reporting Limit and should be less than  $\frac{1}{2}$  the reporting limit. If the sample results show presence of the contaminant, the reported result must be flagged as possible lab contamination. Blank results are not subtracted from sample results.

7.4.3 Prep Blank (PB): The PB is analyzed once every batch of 20 samples or 24 hours, whichever is reached first. The PB undergoes the same sample preparation process. The PB must be below the reporting limit of the analyte and should be less than  $\frac{1}{2}$  the reporting limit, if not refer to the QAM for corrective action.

7.4.4 Laboratory Control Sample (LCS): The LCS is analyzed once every batch of 20 samples or 24 hours, whichever is reached first. The percent recovery is calculated and must be within  $\pm 15\%$  of the true value. LCS failures indicate method problems and must be corrected. Samples

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associated with failed LCSs are re-analyzed, or if no further sample is available, are flagged in the final report.

- 7.4.5 Laboratory Control Sample Duplicate (LCSD): The LCSD is analyzed once every batch of 20 samples or 24 hours, whichever is reached first. See LCS definition above. The RPD between the LCS and LCSD must be +/-20%.
- 7.4.6 Matrix Spike: A matrix spike is analyzed when adequate sample exists or upon customer request. A known amount of analyte is added to an aliquot of a randomly chosen sample. The calculation of the percent recovery is as follows:

$$R = (C_s - C) / s * 100$$

R = percent recovery

C<sub>s</sub> = Fortified sample concentration

C = Sample background concentration

s = Concentration equivalent of analyte added to the

sample

7.4.6.1 The percent recovery for the matrix spike is calculated and must be within +/-25% of the true value. If the matrix spike fails this criterion, the LCS/D results are reviewed to determine whether the failure is matrix specific or an analytical problem. If found to be matrix specific, the report for the spiked sample is noted with this failure.

- 7.4.7 Matrix Duplicate: A duplicate sample is analyzed when adequate sample exists or upon customer request. The relative percent difference (RPD) between the two results should be <20%. The equation for calculating relative percent difference is as follows:

$$RPD = (D1 - D2) / ((D1 + D2) / 2) * 100$$

D1 = The initial result for TOC

D2 = The duplicate result for TOC

- 7.4.8 LOD/LOQ: Refer to the QAM Analytical Procedures section for specific LOD/LOQ requirements. LOD/LOQ data is stored electronically in the QA/LOD&LOQ file folder.

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## **8. Retention Times**

8.1 The retention times in the analytical system should be stable such that peaks can be accurately identified. The retention times are confirmed daily by the evaluation of calibration check standards. The CCV is checked to confirm that the previously identified retention time windows incorporate the applicable analytes for the method defined range. If the peaks in the CCV are not at the expected retention times, the method is updated to account for the time shift. This updated method is used to analyze all subsequent samples and QC. The stability of the system is verified throughout the run with the continued analysis of CCVs and LCSs. The experience of the analyst should weigh heavily in the qualitative interpretation of chromatograms.

## **9. Responsibilities**

9.1 The analyst uses the Quality Control Criteria outlined in this SOP to evaluate and validate the data. The analyst or technician's responsibility is to follow this SOP and record all the data in the lab notebook. Any irregularities within the test or samples should be recorded in the lab notebook and reported to the lab manager who is responsible for reviewing the data.

## **10. Health and Safety**

- 10.1 Refer to the Chemical Hygiene Plan (SOP QA604) and applicable MSDS.
- 10.2 Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation.
- 10.3 Every sample in the lab should be handled as if it is hazardous waste.
- 10.4 All technicians shall be familiar with the Chemical Hygiene Plan.

## **11. Pollution Prevention and Waste Management**

- 11.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Every effort is made to make only enough standard as will be used prior to expiration.
- 11.2 The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Waste management is regulated by the Hazardous Waste Manager/Coordinator. Sample waste is neutralized and is disposed into the municipal waste system.