## CLEANUP CONSTRUCTION MANAGEMENT PLAN Caustic Plume/Cell Building Interim Action GP West Site, Bellingham, Washington

Prepared for: Port of Bellingham

Project No. 070188-001-15A • January 7, 2013 Final





earth <del>+</del> water

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## **1** Introduction

This Cleanup Construction Management Plan (CCMP) addresses compliance monitoring, construction management, and reporting requirements associated with the planned interim action for the Caustic Plume/Cell Building area at the Georgia-Pacific (GP) West Site (Site) in Bellingham, Washington (Figure 1). The interim action is to be conducted by the Port of Bellingham (Port) in accordance with the Interim Action Work Plan (Aspect, 2011b), which is Exhibit C to the 2011 Amendment to Agreed Order No. 6834 between the Port and Washington State Department of Ecology (Ecology). The Caustic Plume/Cell Building interim action includes permanent removal of soils and building materials containing high concentrations of mercury, and is the second of two phases of interim action cleanup to be conducted under the Agreed Order Amendment<sup>1</sup> (refer to Aspect, 2011b).

The Port's Construction Plans and Specifications (Specifications) provide instructions for the Port's selected construction contractor (Contractor) to complete the Caustic Plume/Cell Building interim action so as to meet the goals of the Interim Action Work Plan. This CCMP references those Specifications and focuses on construction-related activities which are the responsibility of Aspect Consulting, LLC (Aspect) as the Port's authorized on-site representative, referred to in the Specifications as "the Engineer."

## 2 Interim Action Goal

The goal of the Caustic Plume/Cell Building interim action is to achieve permanent control of a substantial mercury contaminant source to groundwater and air through removal and off-site disposal of mercury-contaminated soil and building materials. Source control to reduce or eliminate contaminant migration and exposure pathways is the first and most important step for cleanup.

Within the Caustic Plume source area, two localized occurrences of visible elemental mercury have been encountered in subsurface soils: at the former Caustic Filter House (CFH) where mercury was filtered from the caustic produced in the chlor-alkali process, and at the former Mercury Recovery Unit (MRU) where mercury was recovered from the brine used (recycled) in the electrolysis process. The high soil mercury concentrations in these two locations represent substantial sources of mercury to soil vapor and groundwater within the Caustic Plume subarea (Aspect, 2011a).

Some of the remaining structural building materials within the Mercury Cell Building contain elevated total mercury concentrations, and a small percentage of the structural concrete contains mercury leachable in the toxicity characteristic leaching procedure

<sup>&</sup>lt;sup>1</sup> The first phase of the interim action was removal of petroleum-contaminated soils from the Bunker C Tank area, the results of which are reported in Aspect (2012).

(TCLP) above the toxicity characteristic criterion for hazardous waste (waste code D009). Portions of the building materials also contain high levels of lead in paint. The building also contains regulated building materials including asbestos-containing material, fluorescent bulbs, etc.

This interim action is not intended as final cleanup for mercury soil contamination throughout the Caustic Plume subarea, as defined for the Remedial Investigation/ Feasibility Study (RI/FS), but it is intended to permanently remove known sources of mercury, including the highest mercury soil concentrations detected on Site. The mercury source control will be achieved by removal of contaminated soil to meet interim action soil remediation levels outlined in Section 2.1, and complete removal of the Cell Building. There are no remediation levels applicable to the building materials being removed at the Mercury Cell Building. The Cell Building structure and its appurtenances will be completely removed, with disposal of the demolition debris waste in accordance with applicable laws and regulations, as defined in the Specifications.

### 2.1 Soil Remediation Levels

For the purposes of the interim action, there are three applicable soil remediation levels for mercury: elemental mercury visible on excavation sidewalls or bottom, plus two different soil mercury concentrations defining lateral and vertical extent of soil removal. The Specifications define minimum extents of soil to be excavated from the MRU and CFH locations, subdivided into excavation cells MRU-A, MRU-B, CFH-A, and CFH-B, each of which will be excavated to a depth of 12 feet. The minimum excavation extents will be expanded laterally and vertically as necessary to remove visible elemental mercury and achieve the following lateral and vertical soil remediation levels (concentrations) as determined from excavation verification soil sampling and analysis:

- Lateral Remediation Level = 2,100 mg/kg. The interim action excavations will be expanded laterally beyond the minimum extents as necessary to remove soil with total mercury concentrations exceeding a lateral remediation level of 2,100 mg/kg, as determined from excavation <u>sidewall</u> verification soil sampling and analysis. This concentration is anticipated to remove visible liquid elemental mercury and be protective of both groundwater (via dissolved-phase leachability) and indoor air (via soil vapor intrusion); and
- Vertical Remediation Level = 24 mg/kg. To help ensure against the need for additional (deeper) excavation within the interim action excavation locations (during final Site cleanup), the vertical soil profile within each excavation area will be excavated to a maximum depth of 15 feet<sup>2</sup> to achieve a vertical remediation level of 24 mg/kg total mercury, as determined from excavation <u>bottom</u> verification soil sampling and analysis. This concentration is anticipated to be protective of all exposure pathways, including direct contact exposure, under unrestricted land use.

 $<sup>^{2}</sup>$  The depth for compliance with soil cleanup levels based on direct contact exposure is 15 feet (WAC 173-340-740(6)(d)). In accordance with the Interim Action Work Plan and Specifications, an interim action excavation will not extend deeper than 1 foot into the underlying Tidal Flat Aquitard (refer to Aspect, 2012).

Final soil cleanup levels will be defined as part of the final Cleanup Action Plan (CAP), issued by Ecology, to address each exposure pathway defined in the RI/FS (e.g., direct contact, leaching to groundwater, generation of NAPL, vapor generation, etc.), and are not addressed in this interim action.

## **3** Construction Preparation

### **3.1 Remedial Action Management Plan**

The Contractor has been given flexibility in planning and executing the interim action to meet performance standards defined in the Specifications. The Specifications are largely "performance-based," in that they specify required outcomes but rely on the Contractor to propose the most efficient means and methods (within specified constraints) of achieving those outcomes. Granting the Contractor flexibility in their means and methods for the interim action will take advantage of the Contractor's previous experience with similar projects, and will place the contractor in more of an "ownership" role with respect to the interim action outcome.

As described in the Specifications, prior to mobilization, the Contractor will prepare and submit for approval a Remedial Action Management Plan (RAMP) that proposes detailed construction means and methods for the interim action. The RAMP will consist of the following, specific to the interim action:

- A Best Management Practices (BMP) plan addressing the details of the Contractor's means and methods for erosion, sedimentation, and stormwater controls;
- A Contingency Plan addressing the details of the Contractor's means and methods for environmental protection (e.g., controlling and preventing spills of hazardous materials);
- An Excavation and Backfilling Plan addressing the details of the Contractor's means and methods for soil excavation and backfilling, including recovery of liquid mercury accumulations if encountered, chemically amending backfill material for the CFH excavation, and decontamination of excavation equipment prior to demobilization;
- A Dewatering and Water Management Plan addressing the details of the Contractor's means and methods for dewatering excavations, depressurizing the Lower Sand beneath the excavations, capturing runoff from, and run-on to, the contaminated areas, and treatment and disposal of the water generated during the project;
- A Soil Stabilization Plan addressing the details of the Contractor's means and methods for conducting soil stabilization including soil physical screening, weighing and monitoring of required reagent quantities, mixing of the reagents,

packaging and curing of the amended soil, and decontamination of the equipment used prior to demobilization;

- A Building Demolition Plan addressing the details of the Contractor's means and methods for demolition and disposal of the Mercury Cell Building, including abatement of identified regulated materials; and
- A Waste Management Plan addressing the details of the Contractor's means and methods to load, transport, and dispose of waste materials.

Aspect will review and provide comments on the RAMP. The RAMP will also be provided for Ecology review. No work at the Project Site, with the exception of site inspections and surveys, shall be performed until the RAMP is approved.

### 3.2 Monitoring Well Decommissioning

Prior to the start of interim action construction, Aspect will oversee the decommissioning of existing monitoring well CP-MW15, located within the CFH excavation area (Figure 2), in accordance with the requirements of Chapter173-160 WAC.

## 4 Construction Oversight and Monitoring

During the Caustic Plume/Cell Building interim action, Aspect will oversee and monitor the following Contractor's construction activities to ensure compliance with the Specifications:

- Mobilization and site preparation, including oversight of temporary removal of utilities as needed and establishment of temporary erosion and sediment controls;
- Mobilization and setup of the water treatment system;
- Drilling and construction of deep wells for depressurization of the Lower Sand;
- Excavation (Fill Unit) dewatering, Lower Sand depressurization, and water treatment and conveyance to the Port's aerated stabilization basin (ASB);
- Excavation and handling of soil and debris, including removal and handling of visible mercury to the extent practical, and designating the excavated material for disposal;
- Chemical stabilization of soil requiring it, including a two-step test run of the stabilization process, compliance monitoring for the stabilized soil, and designating the stabilized soil for off-site disposal;
- Loading and off-site disposal of contaminated soils and debris and compilation of certificates of disposal as documentation;
- Excavation backfill, including chemical amendment where specified, compaction, and repaying of excavation areas; and

• Decontamination of equipment and demobilization.

Aspect will also conduct soil sampling and analysis within the excavation to verify that soil remediation levels are achieved (i.e., interim action goal for soil excavations achieved).

An AHERA-accredited building inspector from Argus Pacific, under subcontract to Aspect, will oversee and document the abatement of regulated building materials prior to demolition of the Mercury Cell Building. Aspect will then oversee Cell Building demolition, including segregation, loading, and disposal of hazardous and non-hazardous structural building materials.

These activities are described in the subsections below. Refer to the Specifications for additional detail on the required construction activities.

### 4.1 Mobilization and Site Preparation

Aspect will monitor the following Contractor mobilization and site preparation activities for compliance with the Specifications:

- Mobilizing construction equipment, materials, utilities, and appurtenances for utility removal, concrete demolition, soil excavation, excavation dewatering and Lower Sand depressurization, soil stabilization, and excavation backfill;
- Temporarily or permanently removing and, if needed, replacing water utilities in or adjacent to excavation areas, as required;
- Constructing a bermed and lined soil stabilization area within the Mercury Cell Building, including means to weigh and mix the input reagents with contaminated soil, to collect water and convey it to the water treatment system, and to enclose the stabilization equipment to facilitate capture and treatment of mercury air emissions;
- Constructing erosion and sedimentation controls to prevent construction site sediment runoff to surface water or to existing stormwater conveyance, including the installation of rigid, impermeable inserts into existing catch basins;
- Mobilizing and installing air treatment system(s) to treat air emissions from the vacuum excavator and soil stabilization system; and
- Mobilizing and installing a water treatment system for water generated from surface runoff within the excavation and material handling areas, excavation dewatering, Lower Sand depressurization, and accumulation in the soil stockpile and stabilization areas. The water treatment system includes water retention tanks for removing settleable solids, an oil-water separator, conveyance piping between system components and to the ASB pump station, and a flow meter to measure the system discharge.

The Contractor will propose in the RAMP where to locate various activities such as soil stockpiling and water storage/treatment.

### 4.2 Soil Excavation and Performance Monitoring

#### 4.2.1 Soil Excavation

Figure 2 shows the minimum soil excavation areas for the CFH and MRU excavation areas, based on the current understanding of subsurface conditions. Excavation sidewalls will extend laterally beyond the excavation bottoms only as far as needed to maintain a stable excavation and remove the required contaminated soil.

Each excavation area has been subdivided into Excavation Cells: MRU-A, MRU-B, CFH-A and CFH-B, as shown on Figure 2. Each Excavation Cell is further subdivided vertically into Excavation Blocks (Block 1 through Block 11), as depicted in cross section on Figure 3. The subdivision into Blocks is based on designation of the excavated soil into the following three waste categories, in accordance with the federal Resource Conservation and Recovery Act (RCRA) as implemented under the state Dangerous Waste Regulations (Chapter 173-303 WAC):

- Non-Hazardous. Soil containing toxicity characteristic leaching procedure (TCLP) mercury concentrations below 0.2 mg/L and total mercury concentrations below 1,000 mg/kg. Once excavated, these soils will be properly disposed of in a permitted Subtitle D Landfill.
- WT02. State-only toxic dangerous waste (WT02) since TCLP mercury concentrations are below 0.2 mg/L but total mercury concentrations are above 1,000 mg/kg<sup>3</sup>. Once excavated, these soils will be properly disposed of in a permitted Subtitle C Landfill.
- **Stabilization-Required.** Soil containing TCLP mercury concentrations at or above 0.2 mg/L (exhibiting the toxicity characteristic under RCRA); these soils also contain total mercury concentrations above 1,000 mg/kg. These soils will be stabilized on site to achieve the alternative land disposal restriction (LDR) treatment standards for mercury-contaminated soils in accordance with 40 CFR 268.49<sup>4</sup>, so that the stabilized soil can be land disposed at a Subtitle C landfill.

The soil within the Stabilization-Required Blocks will be stabilized prior to disposal. In addition, to achieve a greater degree of protectiveness in the interim action, soil containing visible mercury within the Non-Hazardous and WT02 Blocks will also be segregated and stabilized on site prior to off-site disposal at a Subtitle C landfill. Soil within the Non-Hazardous and WT02 Blocks not containing visible mercury will not be stabilized prior to disposal.

Aspect will direct the excavation activities, with sequential excavation of the Blocks in the order numbered on Figure 3, and handling excavated material in accordance with

<sup>&</sup>lt;sup>3</sup> Refer to derivation of the 1,000 mg/kg total mercury threshold concentration for WT02 provided to Ecology on June 22, 2011.

<sup>&</sup>lt;sup>4</sup> Reduce TCLP mercury concentrations by at least 90% <u>or</u> to 10 times the universal treatment standard (UTS) in 40 CFR 268.48, whichever is less stringent. For mercury, 10 x 0.025 mg/L TCLP mercury (UTS under 40 CFR 268.48) = 0.25 mg/L TCLP mercury as the LDR treatment standard for this project.

Section 2-01-2(1) of the Specifications. Each excavation cell will be advanced in 2-foot lifts according to the numbered sequence.

Soil in Blocks designated as Stabilization-Required and soil containing visible mercury in any Block will be removed using vacuum excavation ("vactor"). The Contractor can use a conventional excavator to remove soil in Blocks designated Non-Hazardous or WT02, and can use the excavator to loosen soil requiring stabilization to facilitate its vactor removal. When excavating other than Stabilization-Required Blocks, Aspect will determine presence/absence of visible mercury by visually observing the excavated material in the excavator bucket. If there are recoverable accumulations of liquid mercury present during excavation, Aspect will direct Contractor to recover the mercury to the extent practicable, and properly containerize and dispose of it in accordance with the requirements of Section 2-01.3(5) in the Specifications.

If excavation side walls need to be extended laterally from a Block in order to maintain sidewall stability or remove contaminated materials to meet remediation levels, Aspect will direct the Contractor to handle the excavated materials identically to the soil in the Block whose sidewalls are being laterally extended.

#### 4.2.2 Debris Management

Aspect will direct Contactor to handle all excavated debris, including the current asphalt pavement, as contaminated materials, and to dispose of it in an appropriate off-site landfill; none of the debris will be recycled or reused. Excavated debris will be handled as needed, including removal of visible mercury to the extent practicable within a defined debris-handling area established, with Aspect's oversight, in accordance with Section 2-02.3(2) of the Specifications.

Aspect will direct Contractor to handle and dispose of debris removed from excavations in accordance with the designation of the Block from which the debris is removed, unless elemental mercury is visible on the debris. That is, the Contractor will be directed to manage debris removed from Blocks designated Non-Hazardous that does not contain visible mercury as part of the Non-Hazardous Waste stream, and to manage debris not containing visible mercury and removed from Blocks designated WT02 as part of the WT02 waste stream.

If elemental mercury is visible on debris removed from Blocks designated Non-Hazardous or WT02, Aspect will direct the Contractor to remove the visible mercury to the extent practicable, and to collect and properly containerize and dispose of it in accordance with the requirements of Section 2-01.3(5) in the Specifications. Once the visible mercury has been removed from the debris from Non-Hazardous or WT02 Blocks to the extent practicable, as determined by Aspect, Aspect will direct the Contractor to dispose of the debris in the WT02 waste stream (Subtitle C landfill).

Within Blocks designated as Stabilization-Required, debris that the Contractor determines is of large enough size to interfere with the chemical stabilization process (described below) will be transported to the Waste Management's Chemical Waste Management (CWM) Subtitle C Landfill in Arlington, Oregon, where it will be treated using macroencapsulation to meet LDRs for debris prior to Subtitle C disposal as federal hazardous waste (D009). The macroencapsulation will use CWM's Macro Secure<sup>TM</sup>

macroencapsulation system to meet requirements of 40 CFR 268.45 and CWM's Debris Treatment Plan (Appendix D to the Specifications) which has been approved by Oregon Department of Environmental Quality.

#### 4.2.3 Performance Monitoring and Over-Excavation

During the excavation of each Block, Aspect will monitor performance of the excavation by observing for visible mercury and by sampling and submitting for chemical analysis, soil from the excavation sidewalls and bottoms. Samples will be collected from the bucket of the Contractor's excavator.

To document that the 2,100 mg/kg mercury lateral remediation level is met, Aspect will collect excavation sidewall verification soil samples at a horizontal spacing of 10 feet and at 2-foot depth intervals (e.g., 0 to 2 feet, 2 to 4 feet, 4 to 6 feet, etc.), across the full depth of each excavation cell sidewall. A minimum of two verification samples will be collected from each excavation cell sidewall (if less than 20 feet in length) within each 2-foot depth interval.

Once a 10-foot length of excavation sidewall, all depth intervals, is confirmed to be below remediation levels, the associated excavation bottom soil sample will be collected (using the excavator bucket) on a systematic 10-foot grid (one sample per 10-foot by 10foot square), with a minimum of two bottom samples per excavation cell, to document that the 24 mg/kg mercury vertical remediation level is met at depth.

Where visible mercury is present in an excavation cell sidewall, or where verification soil sample exceeds the lateral soil remediation levels, Aspect will direct the Contractor to extend the length of sidewall represented by the sample 1 to 2 feet laterally. If visible mercury is absent in the new sidewall, a new sidewall verification soil sample will be collected at that location. Where an excavation bottom verification soil sample exceeds a remediation level, Aspect will direct the Contractor to deepen the excavation in that area by approximately 1 foot, if practicable. If visible mercury is absent in the new excavation bottom, a new bottom verification soil sample will be collected at that location.

If an excavation is advanced deeper than the 12-foot minimum depth to remove additional contaminated soil, Aspect will direct the Contractor to handle and dispose of the deeper materials in the same manner as the material in the Block at the 12-foot depth (subject to visible mercury presence).

Aspect will monitor excavation to observe that, under no circumstances, will the Contractor excavate more than 1 foot into the underlying Aquitard Unit, which, based on existing information, is at a depth ranging from 15 to 19 feet below Existing Grade.

#### Laboratory Analyses

Aspect will subcontract a mobile laboratory operated by Frontier Global Sciences to be onsite while excavation is taking place. Frontier will analyze the verification soil samples from the excavation for total mercury by EPA Method 7473. Frontier's mobile lab is accredited by Ecology for the analysis.

In the unlikely event that soil samples are collected when the mobile laboratory is not on the site, Aspect will submit the samples for total mercury analysis to ALS Environmental laboratory (formerly Columbia Analytical Services) of Kelso, Washington – the laboratory conducting chemical analyses for the Site RI/FS in accordance with the RI/FS Work Plan (Aspect, 2009).

### 4.3 Off-Site Disposal of Soil Not Requiring Stabilization

Excavated soil designating as Non-Hazardous and WT02 and not containing visible mercury requires no treatment prior to off-site disposal, and can therefore be direct loaded for disposal at an appropriate off-site landfill, as follows:

- WT02 soil will be transported under manifest to Waste Management's Chemical Waste Management Subtitle C Landfill in Arlington, Oregon, for disposal in accordance with applicable laws, regulations, and permits.
- Non-Hazardous soil will be transported for disposal at a permitted Subtitle D landfill, in accordance with applicable laws, regulations, and permits.

### 4.4 Monitoring and Disposal of Stabilized Soil

Based on bench-scale stabilization testing of soil representative of the soil that is to be stabilized from the interim action area, chemical stabilization of mercury-contaminated soil will be conducted using a cement + sulfur reagent mixture. Treating the soil with this amendment promotes conversion of elemental and ionic mercury forms in soil to mercury sulfide, with the goal of reducing mercury leachability to below the alternative LDR treatment standard (0.25 mg/L TCLP mercury) and reducing mercury vapor emissions (Anchor QEA, 2012).

The soil stabilization will be conducted inside the Cell Building, within a defined soil stabilization area established in accordance with Section 2-02.3(3) of the Specifications, and verified by Aspect. The soils to be stabilized will be mixed with reagents according to the following specified weight-based proportions (units):

- 100 units of contaminated soil;
- 45 units of Portland cement;
- 15 units of water from the on-site water supply; and
- 5 units of granular sulfur.

Aspect will verify that Contractor weighs each component using a calibrated on-site scale, and maintains each component quantity within +/- 10 percent of the specified component weight. The contaminated soil will be mixed with the stabilization reagents using pug mill(s), or equivalent approved by Aspect during review of Contractor's RAMP.

The first step of the stabilization process will involve physical screening of the soil to remove oversize debris that the Contractor determines would interfere with pug mill operation. Segregated oversize debris will be managed as described in the Debris Management section above. Throughout physical screening and soil amendment/mixing for the stabilization process, Aspect will direct the Contractor to recover liquid mercury

liberated from the soil or debris to the extent practicable, and properly containerize and dispose of it in accordance with the requirements of Section 2-01.3(5) in the Specifications.

Following the physical screening, Aspect will monitor the Contractor's two-stage addition and mixing of the treatment reagents with contaminated soil: (1) the granular sulfur will be thoroughly mixed with the soil, and then (2) the sulfur-amended soil will be thoroughly mixed with the cement and water. The final amended soil will be discharged into Super Sacks® or equivalent portable container of approximately 1 cubic yard capacity.

Aspect will verify the adequacy of physical mixing by visually inspecting the mixing during each mixing stage, and by visual inspecting the amended soil discharged to each Super Sack® immediately upon filling, prior to its curing to semi-solid state (expected within 2 hours). If the QC testing indicates that the stabilized material entering a specific Super Sack® is not adequately mixed, Aspect will direct Contractor to immediately attempt further mixing that material in the second pug mill, without more reagent added.

The contaminated soil will be stabilized in treatment batches, each with a maximum total weight of 34,000 pounds (17 tons), as measured by on-site scale and verified by Aspect. To maintain this weight limit, each treatment batch will be sized at approximately 9 cubic yard (cy), divided into ten Super Sacks® holding approximately 0.9 cy of amended soil. The Contractor will label each Super Sack® using a batch-specific unique identifier defined by Aspect and then stage them together (as a batch), covered by polyethylene sheeting, to cure for a period to be determined by Aspect (e.g., up to 14 days) for completion of the chemical sequestration reactions effecting mercury stabilization.

#### Stabilization Test Runs Prior to Full Scale Stabilization Operation

Prior to beginning full-scale stabilization of contaminated soil, Aspect will verify quality control in the Contractor's stabilization process by overseeing a test run of the process involving the following two steps:

- The first step of the test run will include stabilizing a minimum of two batches (minimum 3 tons each) of contaminated material from a Non-Hazardous Block to verify physical mixing of reagents in both mixing stages (sulfur, then cement). Each test batch of amended soil will be placed into Super Sacks<sup>®</sup>. Aspect will verify satisfactory physical mixing satisfactory prior to the Contractor proceeding to the second step of the test run. Following a curing period approved by Aspect, the stabilized first-step test batches will be loaded and transported for disposal at a Subtitle D landfill.
- 2. The second step is a full-scale test run that will include stabilizing two treatment batches of soil from Blocks designated as Stabilization-Required or soil from other Blocks that contain visible mercury, as directed by Aspect. Aspect will sample for TCLP mercury analysis each of the twenty Super Sacks® of amended soil from the two test-run treatment batches and, based on the TCLP data, will direct the Contractor to manage each Super Sack®, as outlined below.

The stabilization process will be temporarily shut down following completion of the twostep test run, until directed by Aspect to initiate full-scale stabilization – after receipt and interpretation of the TCLP results.

### 4.4.1 Compliance Monitoring for Stabilized Soil

During full-scale stabilization, Aspect will conduct compliance monitoring for each batch of stabilized soil to assess treatment compliance with the 0.25 mg/L alternative LDR treatment standard and the 0.2 mg/L toxicity characteristic criterion (i.e., TCLP mercury concentration below 0.2 mg/L meets both standards).

Aspect will collect one representative composite sample for each approximately 9-cubic yard treatment batch (ten Super Sack®) for TCLP mercury analysis. However, sampling and analysis of each Super Sack® within a treatment batch will be conducted based on results of the composite sampling for that batch, as described below.

To obtain a compliance sample representative of the batch, Aspect will collect an approximately 125-gram aliquot (1/4 cup volume), plus three extra aliquots for potential replicate analyses if warranted, from within each Super Sack® immediately after filling. The samples will be collected using a plastic measuring cup, which will be properly disposed of after use. Each sample aliquot of stabilized soil will be placed in a uniquely-labeled plastic bag, in which it will cure for 10 to 14 days with the corresponding treatment batch.

One cured aliquot from each of the ten Super Sacks® in a treatment batch will be combined to constitute the compliance sample for that batch, which will be submitted to ALS Environmental laboratory for laboratory analysis. The cured aliquots will be cementitious so will be crushed together at the analytical laboratory to produce a particle size less than 1 cm, but not pulverized, in accordance with EPA's TCLP method (EPA Method 1311). The homogenized crushed sample material will be analyzed for TCLP mercury (EPA Methods 1311 and 7470).

### 4.4.2 Waste Designation and Disposal of Stabilized Soil

If a composite sample for a stabilized treatment batch contains a TCLP mercury concentration greater than one-half the alternative LDR treatment standard (i.e., greater than 0.125 mg/L), Aspect will submit for TCLP mercury analysis a replicate sample aliquot from each of the ten Super Sacks® comprising the batch (ten individual analyses for the batch). Each individual Super Sack® then will be designated as waste for disposal based on its individual TCLP result.

Using the compliance monitoring data, Aspect will designate the stabilized soil waste and direct the Contractor to manage it as follows:

- A treatment batch or individual Super Sack® with detected TCLP mercury below 0.2 mg/L will designate as state-only dangerous waste (WT02), and will be loaded, transported under manifest, and disposed of at the CWM Subtitle C Landfill in Arlington, Oregon;
- A treatment batch or individual Super Sack® with detected TCLP mercury between 0.20 and 0.25 mg/L will designate as federal characteristic hazardous

waste (D009) meeting LDR treatment standards, and will be loaded, transported under manifest, and disposed of at the CWM Subtitle C; and

• A treatment batch or individual Super Sack® with detected TCLP mercury at or above 0.25 mg/L will designate as characteristic hazardous waste not meeting LDR treatment standards, and will be loaded and transported under manifest to Waste Management's Mercury Waste Solutions facility in Wisconsin for retort (LDR treatment standard under 40 CFR 268.40) and disposal.

Figure 4 is a flowchart illustrating the decision process outlined above. Aspect, as the Port's agent, will sign the manifest for each shipment of dangerous/hazardous waste transported from the Site.

### 4.5 Air Emissions Control and Monitoring

Emissions of mercury in air (both vapor and particulate phases) will be monitored and controlled during soil excavation and soil stabilization conducted as part of the interim action. The interim action soil remediation project will be relatively brief in duration, and highly dynamic in terms of work activities, locations, and conditions. The air monitoring data will be used to assess Contractor compliance with air quality performance standards (action levels) established in the Specifications. By meeting action levels within the work zone throughout the brief project duration, protection of on-site workers and off-site receptors will be achieved.

Frontier Global Sciences (Frontier), under subcontract to Aspect, will conduct the on-site air monitoring for mercury within the work areas and at locations around the Project Site perimeter to assess fugitive airborne mercury emissions from the Project Site. Frontier will monitor for mercury in vapor and particulate phases using both stationary 8-hr composite (sorbent trap) and real-time monitoring (Lumex hand-held instrument at approximately 2-hour intervals) methods. More frequent monitoring can be conducted at specific locations if exceedances of action levels are detected.

The data from the composite monitoring are more robust than that provided by the realtime monitoring, both in terms of analytical method and representativeness (timeaveraged) at the Project Site perimeter; as such, the composite monitoring data provide the most reliable documentation of air quality around the perimeter of the Project Site throughout the interim action. However, because of the Contractor's requirement to respond to an exceedance of action levels while the work is underway (in real time), compliance with action levels will be evaluated primarily using the Lumex real-time monitoring data by necessity. Frontier will also use the Lumex to monitor mercury concentrations within the breathing zone of workers, in accordance with Aspect's sitespecific health and safety plan.

The 8-hr composite samples and real-time measurements will be taken at a height between 5 and 6 feet above ground level. Starting three days prior to Site earthwork, monitoring will be conducted once per day to establish baseline (background) mercury air concentrations, and it will continue past the end of stabilization until baseline concentrations are reached. Appendix A provides a detailed Air Monitoring Plan for the project, including sampling and analytical methods. A brief overview of the air monitoring and emissions control activities is provided below.

#### 4.5.1 Perimeter Monitoring

The soil remediation work (excavation and chemical stabilization) is currently expected to occur predominantly during March and April 2013. Data collected at Northwest Clean Air Agency's (NWCAA) Bellingham monitoring station<sup>5</sup> during March and April of the previous two years (2011 and 2012) indicate the prevailing wind direction in that season is southeasterly (i.e., toward mill north<sup>6</sup>). The NWCAA data for Bellingham are consistent with site-specific wind data collected on the GP West Site during the same season of 2010, collected as part of a wind power feasibility study. For that study, Convivium Renewable Energy (2011) collected a full year of wind direction (and speed) data at a temporary meteorological tower constructed a few hundred feet north-northwest of the Project Site. Wind direction was recorded at 10-minute intervals between January 9, 2010, and January 8, 2011, at heights of 30 and 45 meters above grade<sup>7</sup>. The prevailing wind direction measured at the station during March and April 2010 was southeasterly (average of 166 degrees azimuth, standard deviation of 75 degrees azimuth).

Although the prevailing wind direction is toward the northwest (mill north), four perimeter monitoring stations (A1, A2, A3, A4) will surround the Project Site perimeter to account for variability in wind direction throughout the work day and throughout the multi-week project. A wind sock will also be set up at the Project Site so wind direction can be tracked visually. Figure 1 depicts the approximate locations of the perimeter monitoring stations.

If air mercury concentrations at the property perimeter are above the  $0.025 \text{ mg/m}^3$  perimeter action level<sup>8</sup> during excavation, Aspect will direct the Contractor to suppress mercury vapors within the excavation, using more rigorous capture and treatment of the vapors and/or application of vapor suppressants on the exposed or stockpiled soil.

#### 4.5.2 During Soil Excavation

During vacuum excavation of soil, exhaust from operation of the vacuum excavator (if used) and operation of the portable mercury recovery vacuum will be filtered and treated with sulfur-impregnated activated carbon to achieve mercury concentrations at or below the 0.1 mg/m<sup>3</sup> permissible exposure limit (PEL), which is an action level for the interim action, within 10 feet downwind of the vacuum exhaust discharge point. Frontier will monitor for airborne mercury using 8-hour composite samples and real-time measurements within this zone (station C1), which is allowed to provide flexibility for monitoring the high velocity air stream without damaging the sampling equipment.

<sup>&</sup>lt;sup>5</sup> Data reported on Puget Sound Clean Air Agency's web site

<sup>(</sup>http://www.pscleanair.org/airq/windrose/)

<sup>&</sup>lt;sup>6</sup> Consistent with other Site documents, the directional reference of "mill north" is approximately 45 degrees west of true north (see true north/mill north arrows on figures).

<sup>&</sup>lt;sup>7</sup> During March and April 2010, only the 45-meter station was recording.

<sup>&</sup>lt;sup>8</sup> The American Conference of Governmental Industrial Hygienists (ACGIH) threshold limit value (TLV) of 0.025 mg/m<sup>3</sup> mercury is a time-weighted average for a normal 8-hour workday, 40-hour work week, over a working lifetime.

If mercury concentrations measured within the 10-foot zone exceed the  $0.1 \text{ mg/m}^3$  action level, Aspect will direct the Contractor to terminate vacuum operations and reconfigure the exhaust treatment system so as to achieve the action level.

### 4.5.3 During Soil Stabilization

To manage mercury vapor generation during soil stabilization, the Contractor will construct a temporary enclosure over the pug mill(s) assembly within the Cell Building. A large-capacity blower on one end of the enclosure will draw air from beneath it through treatment canister(s) filled with sulfur-impregnated activated carbon so as to achieve mercury concentrations at or below the 0.1 mg/m<sup>3</sup> action level within 10 feet downwind of the vacuum exhaust discharge point. Frontier will monitor for airborne mercury using 8-hour composite samples and real-time measurements within this zone (station C1 if the treatment system is same as for vacuum excavator). If mercury concentrations measured within the 10-foot zone exceed the 0.1 mg/m<sup>3</sup> action level, Aspect will direct the Contractor to terminate stabilization operations and reconfigure the stabilization and/or air treatment processes so as to achieve the action level.

In addition, Frontier will establish two monitoring stations (B1 and B2) around the inside perimeter of the Cell Building, where 8-hour composite samples and real-time measurements will be collected throughout the stabilization process. If mercury concentrations measured at these interior stations exceed the 0.1 mg/m<sup>3</sup> action level, Aspect will direct the Contractor to assess the source of mercury vapors within the building, and suppress them using more rigorous capture and treatment of the vapors and/or application of vapor suppressants on exposed or stockpiled soil.

### 4.6 Water Management

Water accumulating on the Project Site - from stockpiles of contaminated soils, run-on to the Project Site from surrounding areas, excavation dewatering, and Lower Sand depressurization, will be collected and conveyed to the Contractor's on-site water treatment system, and conveyed from there to the Port's ASB pump station. Aspect's oversight and monitoring of the Contractor's water management activities is described below.

### 4.6.1 Excavation Dewatering

Aspect will oversee the Contractor's dewatering of the excavations, whether using wellpoints outside the excavation and/or sumps within it. Aspect will direct the Contractor that excavation will only take place when standing water has been pumped from the excavations and when the soils to be excavated are not water-saturated. The water pumped from the excavations will be conveyed to the Contractor's water treatment system and from there to the Port's ASB, as per Section 2-03 of the Specifications.

### 4.6.2 Lower Sand Depressurization

Prior to construction, Aspect will review and approve of the Contractor's Dewatering and Water Management Work Plan<sup>9</sup>, which outlines means and methods to depressurize the

<sup>&</sup>lt;sup>9</sup> Part of the RAMP, and prepared by licensed hydrogeologist or registered professional engineer.

Lower Sand confined aquifer and monitor achievement of the depressurization performance standard (maintain the Lower Sand's artesian water level beneath each excavation area at a minimum depth of 20 feet below existing grade throughout the excavation and backfilling process for each excavation area), Aspect will then oversee the Contractor's construction of one or more wells for the depressurization of the Lower Sand, and then oversee the depressurization. The requirements for the depressurization well construction, including sealing off the Fill Unit before penetrating the Lower Sand, are detailed in Section 2-03.2(2) of the Specifications.

Throughout the entire excavation and backfilling process for each excavation area, Aspect will oversee Lower Sand depressurization by periodically checking the water levels in the Contractor's chosen Lower Sand monitoring wells near the excavations. If the monitoring indicates that the water level beneath an excavation is less than 20 feet below existing grade, Aspect may direct the Contractor to stop all other activity until the depressurization performance standard is met.

The water pumped from the Lower Sand will be conveyed to the Contractor's water treatment system and from there to the Port's ASB, as per Section 2-03 of the Specifications.

#### 4.6.3 Stormwater Collection

Aspect will oversee the Contractor's collection of stormwater generated within the Project Site, including collection of water that may accumulate in stockpile and soil stabilization areas. If Aspect observes that Contractor's stormwater collection system is allowing stormwater to discharge to the subsurface or to surface water, Aspect will direct the Contractor to stop other activities and eliminate the discharge. The Contractor will be required to address the cause of the discharge prior to resuming Site activities. The water pumped from the Lower Sand will be conveyed to the Contractor's water treatment system and from there to the Port's ASB, as per Section 2-03 of the Specifications.

### 4.6.4 Water Treatment and Disposal

The Contractor is responsible for providing and operating an on-site treatment system for removal of settleable solids and, if present, separate-phase oil. System requirements are described in Paragraph 2-03.2(4) of the Specifications. Aspect will oversee the Contractor's operation of their water treatment system in accordance with the Specifications, including monitoring the system for leaks and for compliance with Project water quality performance standards for discharge to the ASB (total settleable solids below 100 ml/L and no visible separate-phase oil).

In compliance with Ecology's letter approving the discharge of non-routine wastewater from this project to the ASB (Ecology, 2012), Aspect will collect for total mercury analysis one sample per week of water from the Contractor's water management system prior to it being discharged to the ASB pump station. The water sampling results will be provided to Ecology and included in the Interim Action Report. Aspect will also receive from the Contractor the daily totals of flow discharged from the water treatment system, and will include the information as part of the Interim Action Report.

### 4.7 Off-Site Disposal of Excavated Material

Aspect will oversee the Contractor properly disposing of the five different classifications of waste generated from the soil cleanup as part of this interim action: Non-Hazardous Waste, State-Only Dangerous Waste (WT02), Federal Hazardous Waste (D009) Meeting Treatment Standards, Federal Hazardous Waste (D009) Not Meeting Treatment Standards, and Macroencapsulated Hazardous Waste Debris (D009). Aspect has submitted waste profiling paperwork to off-site disposal facilities and received preapprovals for disposal of the various waste streams. The Contractor is responsible for selecting the permitted off-site soil disposal facility, and obtaining the disposal permits and hazardous waste manifest forms.

The Contractor will provide Aspect with copies of the certificates of disposal for material disposed of off site, and Aspect will include them in the Interim Action Report.

### 4.8 Excavation Backfill, Compaction, and Paving

Aspect will oversee the Contractor's backfilling the excavations with gravel borrow (and, depending upon the condition of the subgrade material, quarry spalls), compaction, and paving of the excavated areas to restore pre-construction grade (Existing Grade in the Specifications). Aspect will review and record Contractor-submitted documentation that the backfill material was imported from a known source of uncontaminated fill, and will review and document Proctor tests for the imported gravel borrow.

Aspect will also oversee Contractor backfilling the CFH Excavation Cells using imported gravel borrow that has been mixed with 3 percent by weight powdered siderite, with the intent to help neutralize high pH groundwater at that location. Aspect will monitor Contractor uniformly mixing the powdered siderite with gravel borrow prior to placing it in the excavation, in compliance with Section 2-01.3(8) of the Specifications.

Materials Testing and Consulting Inc., under subcontract to Aspect, will verify, using a nuclear density gage, the required backfill compaction to minimum 90 percent of maximum dry density, in compliance with Section 2-01.3(9) of the Specifications.

Following backfill and paving, Aspect will install one monitoring well (CP-MW16) in the Fill Unit (not excavation backfill) on the mill west (downgradient) edge of the completed CFH excavation. The new monitoring well will be used to monitor changes in Fill Unit groundwater chemistry over time in response to the source removal and sideriteamended backfill, providing useful field-scale treatability data while the Site feasibility study and subsequent remedial design for final action proceed.

## 4.9 Cell Building Demolition

Prior to demolition of the Cell Building, Argus Pacific Inc., under subcontract to Aspect, will oversee and document the Contractor's abatement of regulated building materials in the Cell Building. The regulated materials include asbestos-containing materials, lead-containing paints, polychlorinated biphenyl (PCBs) in light ballasts and transformers, and mercury-containing fluorescent and high-intensity discharge (HID) light bulbs.

Following abatement, Aspect will oversee Cell Building demolition, including the segregation, loading, and disposal of hazardous and non-hazardous structural building materials, in accordance with Section 2-04.2 of the Specifications. Aspect will monitor the demolition and segregation of the approximately 10 tons of concrete that is characterized as Hazardous Waste, and oversee its loading for transport to CWM Subtitle C Landfill in Arlington, Oregon, where it will be treated using macroencapsulation to meet LDRs for debris prior to Subtitle C disposal as federal hazardous waste (D009). Aspect will also monitor demolition of the non-hazardous building material and its loading for Subtitle D landfill disposal.

When the Cell Building floor slab is demolished and removed, Aspect will observe the exposed underlying soils for visible mercury. If visible mercury is observed, Aspect will contact the Port and Ecology, and initiate plans for its removal as part of the interim action.

### 4.10 Equipment Decontamination and Demobilization

Prior to Contractor demobilization, Aspect will monitor the Contractor's decontamination and demobilization of equipment used to excavate, handle, or stabilize mercurycontaminated material in the interim action. Water generated by the decontamination process will be conveyed to the Contractor's water treatment system and then the ASB pump station. The final step of Contractor demobilization will be decontamination and demobilization of the water management equipment. The settled solids generated during decontamination of earthwork and water management equipment will be temporarily stockpiled, chemically tested to profile it for proper landfill disposal, and accordingly disposed of offsite.

## **5** Reporting of the Interim Action

In accordance with the Interim Action Work Plan, an Interim Action Report will be prepared after completion of the Caustic Plume-Cell Building interim action to document the interim action activities for Ecology review. At a minimum, the report will include the following:

- Description of cleanup activities conducted, including deviations from the Specifications;
- Maps illustrating the as-built excavation areas and other pertinent information;
- Detailed performance monitoring information, including sample location, analytical methods, data quality review, and results;
- If interim action soil remediation levels were not achieved (e.g., due to soil inaccessibility), discussion of why they were not, and documentation of inferred areas and depths of residual soil exceeding the remediation levels;

- Documentation of water management activities and monitoring during construction;
- Documentation of excavation backfill quantities by source;
- Documentation of installation of new monitoring well CP-MW16;
- Documentation of Cell Building hazardous materials abatement, demolition, and demolition debris handling; and
- Documentation of contaminated soil and debris disposal, including quantities removed and disposed, and copies of hazardous waste manifests and landfill certificates of disposal.

The Caustic Plume Area Interim Action Report will be submitted to Ecology as a draft for review. Ecology comments will be incorporated and a final Report prepared. The results of the interim action will also then be incorporated into the draft feasibility study for the Site.

### 6 References

- Anchor QEA, 2012, GP West Site High-Mercury Soil Stabilization Bench-Scale Test Results, February 10, 2012.
- Argus Pacific, 2011, Regulated Building Materials Assessment Report, Mercury Cell Building, Port of Bellingham, October 2, 2011
- Aspect, 2009, RI/FS Work Plan, Georgia-Pacific West Site, Bellingham, Washington, September 10, 2009.
- Aspect, 2011a, Interim Action Pre-Design Investigation Report, Georgia-Pacific West Site, Bellingham, Washington, April 28, 2011.
- Aspect, 2011b, Interim Action Work Plan, Georgia-Pacific West Site, Bellingham, Washington, August 22, 2011, Final.
- Aspect, 2012, Bunker C Tank Interim Action Report, Georgia-Pacific West Site, Bellingham, Washington, February 24, 2012, Final.

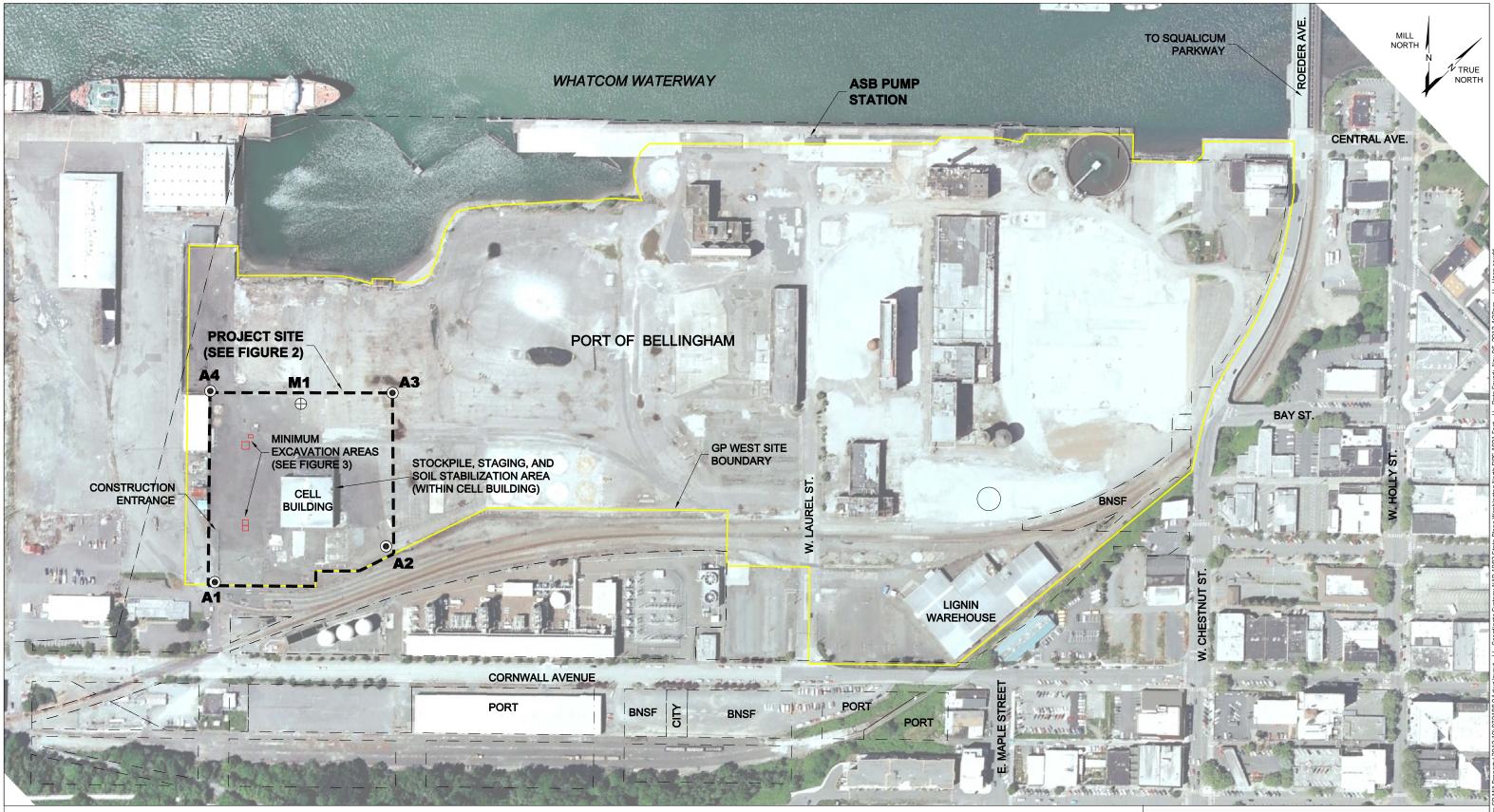
Convivium Renewable Energy, 2011, Port of Bellingham Met Tower Final Data Report.

Ecology, 2012, Letter from Mark Henderson, Ecology Bellingham Office, to Brian Gouran, Port of Bellingham, regarding Approval of discharge of non-routine wastewater to the ASB, August 23, 2012.

## 7 Limitations

Work for this project was performed and this report prepared in accordance with generally accepted professional practices for the nature and conditions of work completed in the same or similar localities, at the time the work was performed. This report does not represent a legal opinion. No other warranty, expressed or implied, is made.

All reports prepared by Aspect Consulting are intended solely for the Client and apply only to the services described in the Agreement with Client. Any use or reuse by Client for purposes outside of the scope of Client's Agreement is at the sole risk of Client and without liability to Aspect Consulting. Aspect Consulting shall not be liable for any third parties' use of the deliverables provided by Aspect Consulting. Aspect Consulting's original files/reports shall govern in the event of any dispute regarding the content of electronic documents furnished to others.



A1 Air Sample Location

 $M1 \oplus$  Meteorological Station and Wind Sock

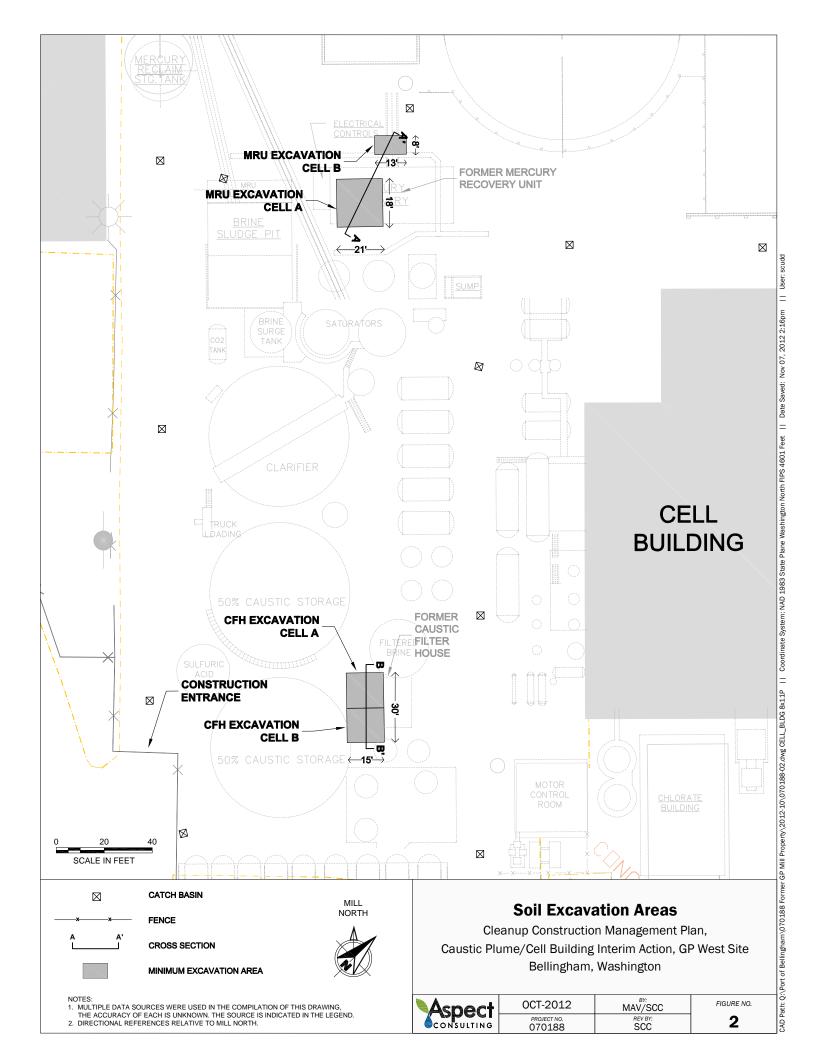
0 240 480

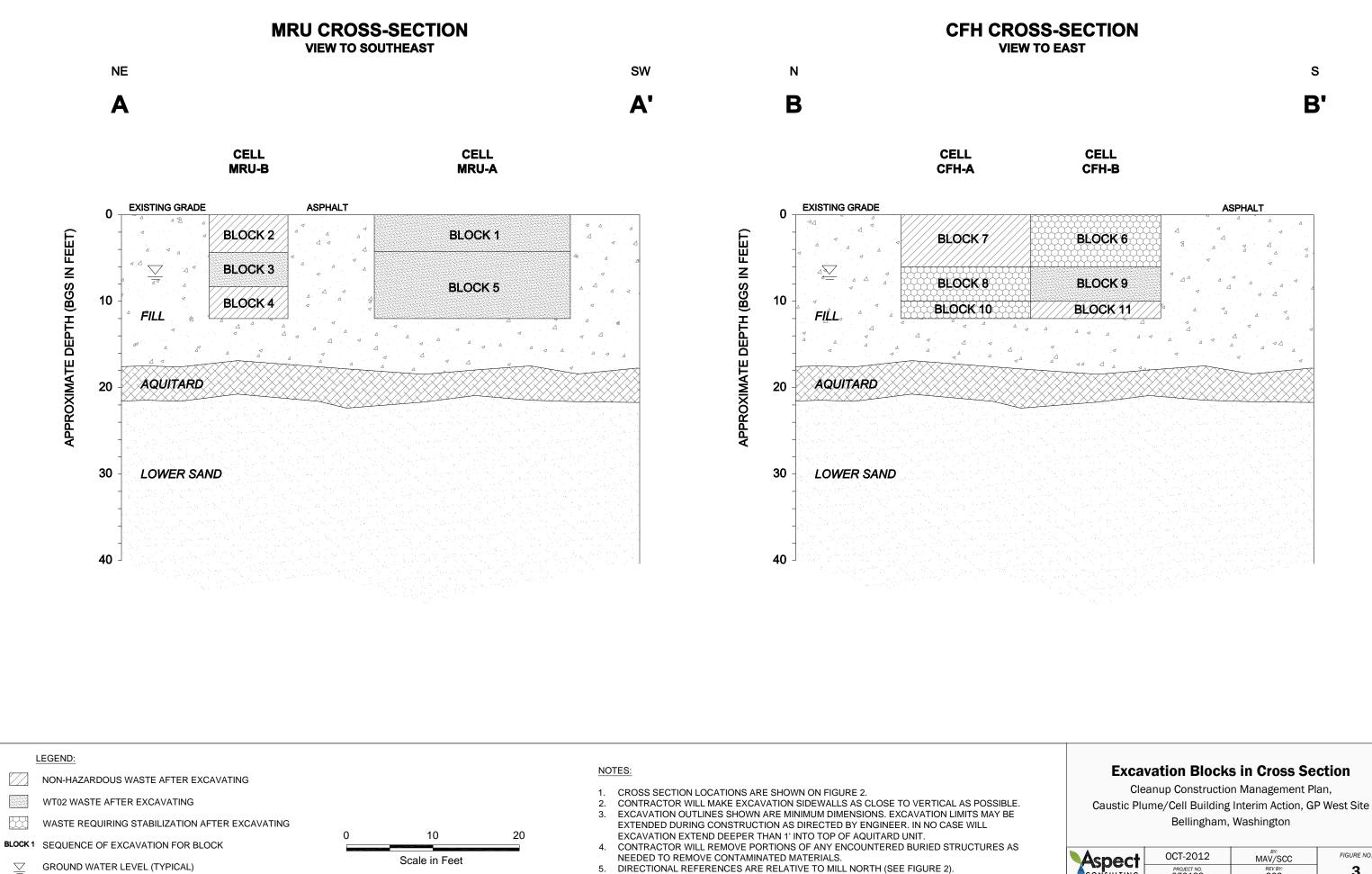
Scale in Feet

#### Site Plan

Cleanup Construction Management Plan, Caustic Plume/Cell Building Interim Action, GP West Site Bellingham, Washington

	NOV-2012	MAV/SCC	FIGURE NO.	
CONSULTING	PROJECT NO. 070188	REV BY: SCC	1	





- 1				
	Aspect	OCT-2012	MAV/SCC	FIGURE NO.
	CONSULTING	PROJECT NO. 070188	REV BY: SCC	3

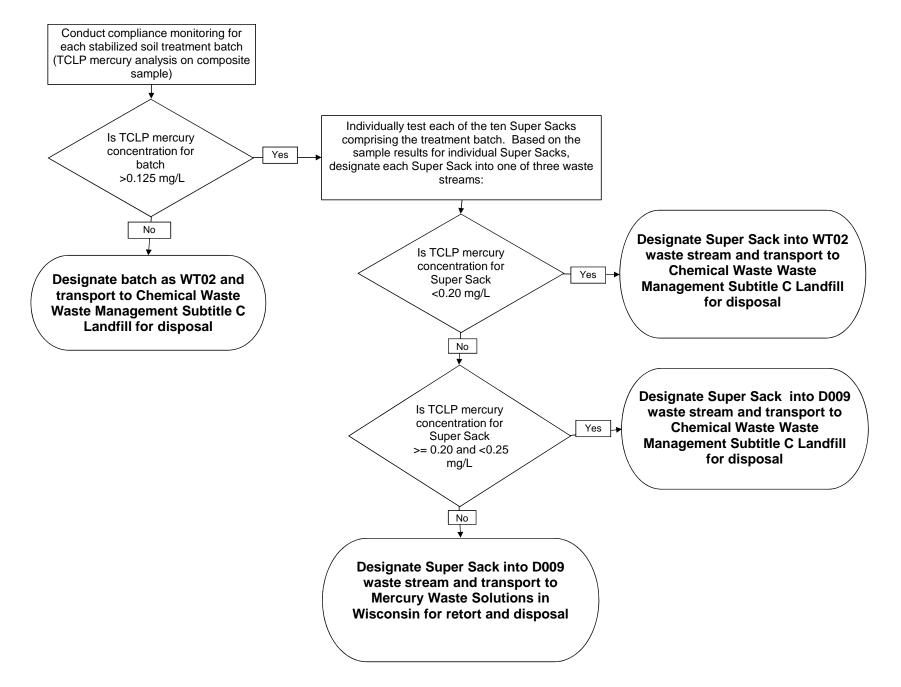


Figure 4 Flowchart for Management of Stabilized Soil

## **APPENDIX A**

Air Monitoring Plan (prepared by Woodard and Curran)



# Air Monitoring Plan

Georgia-Pacific West Site Bellingham, Washington

Prepared for: Frontier Global Sciences, Inc.

225986.00 September 14, 2012



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**APPENDIX C: LABORATORY ANALYTICAL SOPS** 

### ACRONYMS

ACGIH BNSF	American Conference of Governmental Industrial Hygienists BNSF Railway Company
COC	Contaminant of Concern
CVAFS	Cold Vapor Atomic Fluorescence Spectrophotometry
FSTM	Frontier Sorbent Total Mercury
MTCA	Model Toxics Control Act
ng/L	nanograms per liter
ng/m³	nanograms per cubic meter
nm	nanometer
PSCAA	Puget Sound Clean Air Agency
SOP	Standard Operating Procedure(s)
TLV	Threshold Limit Value
ug/L	micrograms per liter
ug/m <sup>3</sup>	micrograms per cubic meter
UŠEPA	United States Environmental Protection Agency
W&C	Woodard & Curran

### 1. INTRODUCTION

#### 1.1 SITE SETTING

The Georgia-Pacific West Site (Site), located at 300 W. Laurel Street, Bellingham, Washington was used to manufacture paper products from 1926 through 2007. The Site encompasses approximately 64 acres located on the Bellingham waterfront, bordered on the north by the Whatcom Waterway, to the east and south by the BNSF Railway Company (BNSF), and on the west by the Bellingham Shipping Terminal. The Site is relatively flat and currently covered by pavement and several remaining mill buildings. The Caustic Plume interim action, for which this Air Monitoring Plan applies, will remove mercury-contaminated soil and debris within the Caustic Plume Area at the southwest end of the Site.

#### 1.2 PURPOSE OF MONITORING

This plan establishes an ambient air monitoring and sampling program such that Model Toxics Control Act (MTCA) requirements for protection monitoring are met during soil remediation activities, with anticipated duration on the order of 3 to 6 weeks. Protection monitoring will confirm that human health and the environment are adequately protected during the interim action activities.

Mercury is the contaminant of concern (COC) for this interim action. This air monitoring plan includes air monitoring within the core of the work activities (at excavation, at chemical stabilization system, etc.) as well as around the Project Site perimeter to promptly detect a mercury concentration exceedance of 0.025 mg/m<sup>3</sup>, which is a defined action level approved by the Washington State Department of Ecology (Ecology) for this interim action and is based on the American Conference of Governmental Industrial Hygienists (ACGIH) threshold limit value (TLV) as a time-weighted average for a normal 8-hour workday and a 40-hour work week.

#### 1.3 APPROACH

This plan calls for both stationary 8-hour composite air sampling and real-time air monitoring. Stationary air monitoring stations will be established to collect 8-hour composite samples at four fixed monitoring points located around the project site perimeter. Two fixed air monitoring stations will also be established inside the Mercury Cell Building to collect 8-hr composite samples when treating (chemically stabilizing) highly contaminated soils and another fixed monitoring station will be used to monitor discharges from the vacuum excavator and the soil stabilization system near the Cell Building.

A portable instrument capable of taking real-time measurements of mercury in the ambient air will be used to assess short term mercury releases and to monitor at fixed locations and locations other than the fixed locations depending on the nature of work being conducted. Fixed and portable methods and instruments will have detection limits low enough to determine exceedances of defined air action levels.

The interim action soil remediation project will be relatively brief in duration, and highly dynamic in terms of work activities, locations, and conditions. The air monitoring data will be used to assess Contractor compliance with air quality performance standards (action levels) established in the Technical Specifications. Because of the Contractor's requirement to respond to an exceedance of action levels while the work is underway (real-time), the determination of Contractor compliance with performance standards will be conducted primarily using the real-time monitoring data by necessity. By meeting action levels within the work zone throughout the brief project duration, protection of on-site workers and off-site receptors will be achieved. The data from the composite monitoring are more robust than that provided by the real-time monitoring, both in terms of analytical method and representativeness (time-averaged); as such, the composite monitoring data provide a rigorous and accurate documentation of air quality around the perimeter of the Project Site during the interim action.

#### 1.4 METHODOLOGY

#### 1.4.1 Real-time Monitoring

Real time air monitoring will be performed using a portable, hand-held Lumex RA-915 Light mercury vapor analyzer. The Lumex operation is based on differential Zeeman atomic absorption spectrometry using high frequency modulation of light polarization. A radiation source (mercury lamp) is placed in a permanent magnetic field. The mercury resonance line (wavelength = 254 nanometers [nm]) is split into three polarized Zeeman components (p, s- and s+, respectively). When radiation propagates along the direction of the magnetic field, a photodetector detects only the radiation of the s- components, one of those falling within the absorption line profile and another one lying outside. When mercury vapor is absent in the analytical cell, the radiation intensities of both s- components are equal. When absorbing atoms appear in the cell, the difference between the intensities of the s- components increase as the mercury vapor concentration grows. The s- components are separated by the polarization modulator. The spectral shift of the s-components is significantly smaller than the widths of molecular absorption bands and scattering spectra, hence the background absorption by interfering components does not affect analyzer's readings. A multipath cell with an effective length of about 10 meters is used to enhance the sensitivity of analysis.

The Lumex has a reported detection range of 0.1 to 100 micrograms per cubic meter or 0.0001 to 0.1 mg/m<sup>3</sup>. The Lumex is portable and will be used to provide real-time measurements of mercury in the ambient air at each of the fixed project perimeter monitoring stations, at each cell building monitoring station and the air treatment system exhaust at least four times throughout the workday depending on the schedule of remediation activities being performed. In addition, the Lumex will also be used to monitor worker breathing zones and can be used to monitor selected locations of interest during the remediation activities. The locations of interest will vary depending on conditions at the Site including wind direction and types of remediation tasks being conducted.

#### 1.4.2 Composite Sampling

Four fixed monitoring stations around the Project Site perimeter, two fixed monitoring stations inside the Cell Building and one monitoring the exhaust of treatment systems will be equipped with sampling pumps, critical orifice, vacuum gauge, sample traps, and associated equipment. 8-hour composite samples of total mercury in the ambient air will be collected and analyzed according to the Frontier Sorbent Total Mercury (FSTM) or functional equivalent. Mercury samples will be collected on specially prepared iodated carbon sorbent traps supplied by the laboratory. These traps combined with a sensitive analytical method (cold vapor fluorescence) enable routine quantification of ambient air mercury with a 0.5 ng/m<sup>3</sup> (5.0E-7 mg/m<sup>3</sup>) detection limit (24 hr sample at an estimated 4 slpm flow rate. The critical components of this method are described below:

(1) FSTM Low-Level Hg Sample Media – A critical component of the FSTM method is the standardized, low level mercury blank of the sample media. Further the proprietary material used to chemically impregnate the special carbons is highly efficient at capturing mercury and has been shown to be 99.9% efficient in mercury capture. The trap has two sections, one designed as the sample trap and the second used to measure (quality assure) potential mercury breakthrough.

(2) Analytical Method (functional equivalent of United States Environmental Protection Agency (USEPA) 1631 CVAFS) – The cold vapor atomic fluorescence spectrophotometry (CVAFS) method is capable of measuring mercury at levels down to 0.08 ng/L or 8.0E-5 mg/m3. This low-level method enables the measurement of very low concentrations and of mercury and is a critical component of the FSTM method.

(3) Frontier Sampling Equipment – Frontier has designed and manufactured sampling equipment that enables the quantification of the sample volume down to a tenth of a liter of air. Special Mass Flow Meter and Mass Flow Totalizers are utilized that enable precise quantification of sample volumes.

(4) Basic Clean Sample Handling Techniques – The FSTM method is a very easy technique to deploy affectively in the field which is why this method is desirable compared to the excessively complicated USEPA Compendium

Method I-05. By following basic clean sample handling techniques (wearing gloves, bagging sample traps etc), the sample traps are easily protected and shipped to the lab, and have a one-year storage life after sampling.

#### 1.5 ACTION LEVELS

Two air quality action levels are defined in the Technical Specifications for the interim action:

- 0.025 mg/m<sup>3</sup> within the perimeter of the Cell Building and at the Project Site perimeter (American Conference of Governmental Industrial Hygienists threshold limit value); and
- 0.1 mg/m<sup>3</sup> within 10 feet of the discharge from air treatment system(s) for the vacuum excavator and the soil stabilization system within the Cell Building (OSHA permissible exposure limit).

The results of the Project Site perimeter and the Mercury Cell Building real-time sample readings will be compared to the 0.025 mg/m<sup>3</sup> mercury action level. The results of the air treatment system(s) exhaust real time sample readings will be compared to the 0.1 mg/m<sup>3</sup> mercury action level, with the recognition that the Project Site perimeter monitoring will include emissions from the treatment system(s). If any exceedance of the action levels is measured by the Engineer, the Contractor must adjust work practices so as reduce mercury air emissions and achieve the action level, as described in Section 1.6.

#### 1.6 CORRECTIVE MEASURES

If any Lumex real-time reading at a monitoring station exceeds an action level, an immediate second measurement will be taken. If the second reading exceeds the action level, the Engineer (Aspect Consulting) will be notified and the current work practices assessed. Aspect Consulting will assess the meteorological conditions, the current remediation activities and whether they will continue, and air controls implemented, and make recommendations to the Contractor for immediate changes to work practices and/or air controls, as appropriate, to avoid creating the working conditions that caused the exceedance of the action level. The frequency of real-time monitoring at the station showing the exceedance will be increased to approximately hourly. The Contractor is solely responsible for adjusting work practices as needed to meet the action levels.

### 2. SAMPLE COLLECTION AND ANALYSIS

#### 2.1 SAMPLING EQUIPMENT

The sampling equipment for portable ambient air quality measurements consists of a Lumex brand 915+ mercury vapor analyzer.

The sampling equipment for fixed location 24-hr composite samples consists of the following:

- Sample pump;
- Mass flow meter and totalizer;
- Iodated carbon sample traps;
- Flow control systems; and
- Sample box containing all equipment.

#### 2.2 SAMPLING LOCATIONS

The soil remediation work (excavation and chemical stabilization) is expected to occur predominantly between mid-September and mid-November 2012. Based on Puget Sound Clean Air Agency's (PSCAA) wind speed and wind direction data collected at PSCAA's Bellingham monitoring station from mid-September through mid-November 2011 (one year prior), the prevailing winds in Bellingham at that time of year range from southeasterly to southsoutheasterly. The PSCAA data for Bellingham are consistent with site-specific wind data collected on the GP West Site during the same season of 2010, as part of a feasibility study for development of wind power as an element of the future site redevelopment. For that study, Convivium Renewable Energy (2011) collected a full year of wind direction (and speed) data at a temporary meteorological tower constructed immediately northwest of the Project Site. Wind direction was recorded at 10-minute intervals between January 9, 2010, and January 8, 2011, at heights of 30 and 45 meters above grade. The prevailing wind directions at the two heights were virtually identical (average and standard deviation within 1 degree azimuth) for the period mid-September through mid-November 2010: average of 157 degrees azimuth (south-southeasterly) and standard deviation of 81 degrees azimuth.

Although the prevailing wind direction during the soil remediation project timeframe is predicted to be from the Project Site toward Bellingham Bay, the stationary monitoring stations will surround the Project Site to account for variability in wind direction throughout the work day and throughout the multi-week project.

Figure 2-1 depicts locations for the four fixed perimeter monitoring stations (A1-A4) surrounding the Project Site boundary, the two fixed monitoring stations within the Cell Building (B1 and B2) and the fixed monitoring station at the outlet of the treatment system exhaust (C1). Precise locations for the fixed stations may be adjusted in the field based on access or other considerations. Locations for the two fixed monitoring station system set up. Treatment system discharge monitoring locations will follow the treatment systems – presumably fixed for the stabilization system and transient for the vacuum excavator. Breathing zone measurements will be taken in worker breathing zones.

In addition to fixed monitoring stations, Figure 2-1 also depicts the location for a meteorological station and windsock. The meteorological station will collect data (wind speed, direction, temperature, relative humidity) throughout the duration of the project. It has been located in an area such that representative wind flow data will be collected unaffected by building downwash and cavity effects. It has also been located in an area that should be visible to Contractors during excavation activities such that they may quickly asses the wind direction and relative strength visually by viewing the windsock.

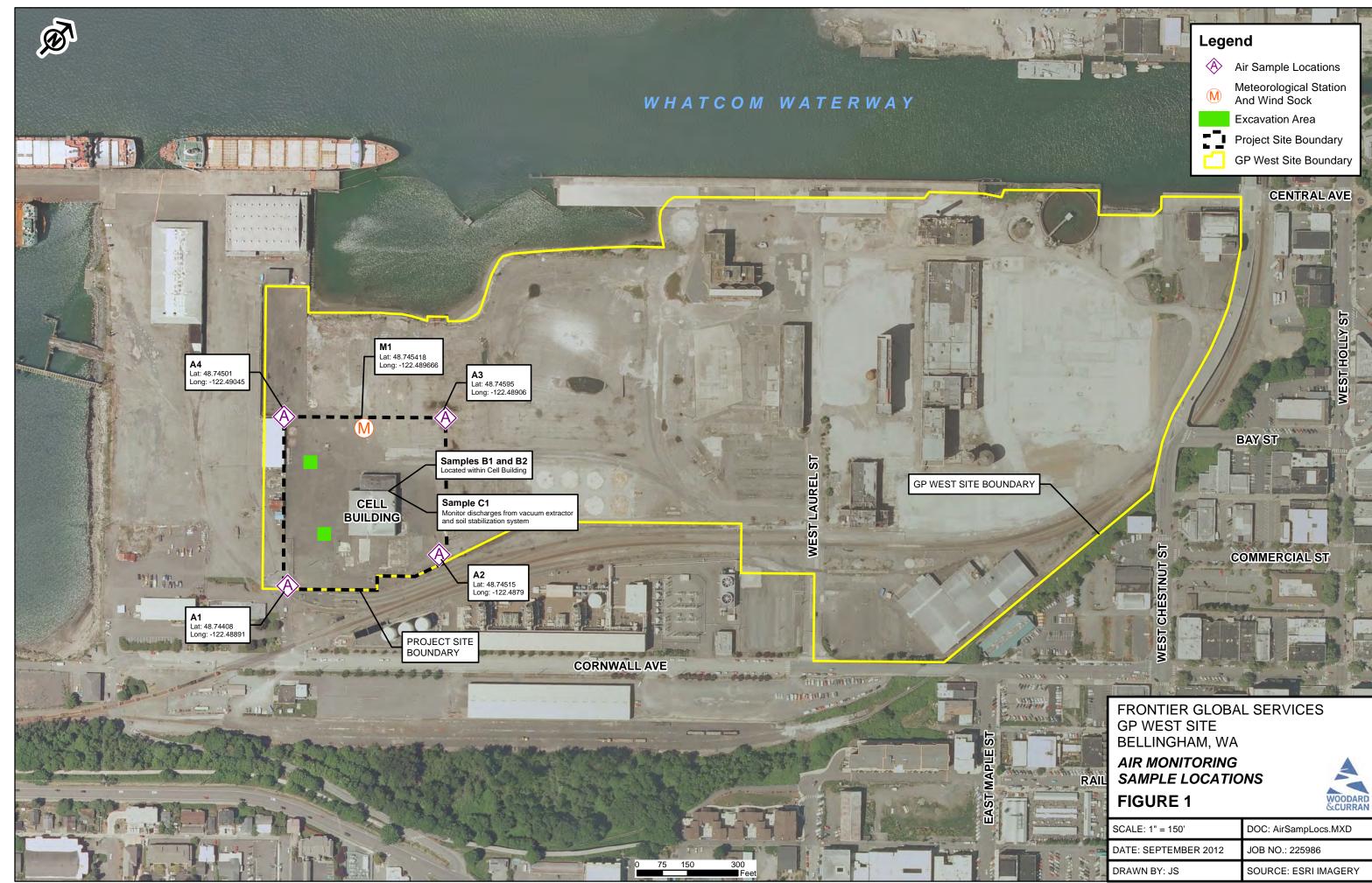
Monitoring Location	Description	Geographic Coordinates <sup>1</sup>	
Looution		[Lat]	[Long]
A1	Southern corner of project boundary	48.74408	122.48891
A2	Eastern corner of project boundary	48.74515	-122.4879
A3	Northern corner of project boundary	48.74595	-122.48906
A4	Western corner of project boundary	48.74501	-122.49045
B1	Inside cell building	TBD	TBD
B2	Inside cell building	TBD	TBD
C1	Exhaust of on-site mercury control system	aust of on-site mercury control system Within 10 ft ra	
M1	North-West project boundary line	48.74542	-122.48967

#### Table 2-1: Air Monitoring Sampling Locations

1 Geographic Coordinate System WGS 1984.

Coordinates are in decimal degrees.8-hrcomposite samples and real-time sampling and analysis using the Lumex analyzer will be taken at a height between 5 feet and 6 feet above ground level in accordance with USEPA guidance (Air-Superfund National Technical Guidance Study Series, April 2003.)

When conducting Lumex real-time monitoring at fixed monitoring locations, the Lumex measurements should be taken as close to the inlet to the stationary sample collection device as is practicable.







SCALE: 1" = 150'	DOC: AirSampLocs.MXD
DATE: SEPTEMBER 2012	JOB NO.: 225986
DRAWN BY: JS	SOURCE: ESRI IMAGERY

#### 2.3 SAMPLING FREQUENCY

Starting 3 days prior to remediation activities, a Project Site perimeter survey and Mercury Cell Building survey will be performed once per day by obtaining both 8-hour composite samples and real-time Lumex analyzer readings taken four times per day (approximately two hours apart) at the fixed monitoring location to establish pre-construction baseline mercury concentrations.

Once soil remediation earthwork has commenced, real-time Lumex analyzer readings will be taken at each station at least four times throughout the workday at approximately 2-hour intervals depending on the schedule of remediation activities being performed (e.g., not monitoring stabilization area when stabilization is not occurring, not monitoring vacuum excavator exhaust when it is not in operation, etc.). The time of day for real-time measurements will be coordinated with remediation work to assess activities that might generate the most emissions. Additional monitoring will be performed if exceedances of the action levels are recorded as described in Section 2.6. While moving between fixed sample locations, the Lumex will remain in detection mode to provide an additional assessment of air quality at the project site. Readings in transit are for informational purposes only and do not have to be recorded.

8-hour composite air samples will be collected daily from the fixed perimeter, Cell Building, and treatment system exhaust sampling locations during soil remediation activities. It is expected that the composite samples will be collected over an 8-hour period, starting first thing each work day morning and ending when the workday stops. If the duration of the workday is expected to be longer than 8 hours, the Engineer will determine if a second 8-hour composite sample should be initiated following collection of the first 8-hour composite sample. This determination will be made based on the amount of time past 8 hours that workers are expected to be on site, the nature of the work that workers will be performing, and the results of real-time Lumex readings taken throughout the day.

The air monitoring program may be modified if results indicate additional or less frequent monitoring is warranted or appropriate. As the project progresses, the frequency of perimeter surveys and Mercury Cell Building surveys may be modified based on the results of previous surveys and the specific or remaining remediation activities planned. For example, if previous monitoring results have shown no exceedances and future work has less potential for mercury vapor releases, a reduction in perimeter monitoring frequency may be satisfactory.

Perimeter monitoring will continue for one week following the completion of remediation activities or until the results of sampling and analysis demonstrates that the concentration of mercury in the ambient air has stabilized to preconstruction baseline levels.

#### 2.4 SAMPLING PROCEDURES

Real time measurements will be collected with the Lumex Mercury Vapor Analyzer. The instrument will be calibrated and operated in accordance with manufacturer's instructions and the protocol provided in Appendix A. Readings will be recorded in a field logbook used for all perimeter air monitoring purposes. Minimum related data that will be recorded include time, date, location description, temperature, and general weather.

Sampling will be performed using clean techniques following procedures provided by the laboratory. A protocol for composite sampling at fixed locations is provided in Appendix B.

#### 2.5 SAMPLE ANALYSIS

Real time measurements of mercury in ambient air will be provided by the Lumex mercury vapor analyzer, so laboratory analysis is not required. Composite sorbent trap samples will be analyzed by the laboratory for total mercury using CVAFS at a wavelength of 253.7 nm (equivalent of USEPA 1631). Laboratory SOPs and quality assurance and quality control procedures are provided in Appendix C. The concentration of mercury in ambient air will be calculated from the total mercury (vapor and particulate phase) collected at each location divided by the measured volume of air pulled through the sample trap.

#### 3. REFERENCES

Convivum Renewable Energy, 2011. Port of Bellingham Met Tower Final Data Report.

EPA. 2003. Air/Superfund National Technical Guidance Study Series, Volume VI – Guidance for Ambient Air Monitoring at Superfund Sites. April, 2003.

# APPENDIX A: PROTOCOL FOR COLLECTING AND ANALYZING MERCURY VAPOR IN AIR WITH A LUMEX RA-915 LIGHT MERCURY ANALYZER

# Protocol for Collecting and Analyzing Mercury Vapor In Air With A Lumex RA-915+Mercury Analyzer

January 2003

Revised March 7, 2003

# Section 1 Introduction

This protocol is a guideline for operating the Lumex RA-915 for mercury vapor analysis. It is based on a protocol obtained from the Maine Department of Environmental Protection that was intended to be a Standard Operating Procedure for their measurement of mercury vapor. The procedures are identical. This protocol document has only been modified to remove MEDEP-specific information and to address the needs of the HoltraChem Cell Process Dismantling Air Monitoring Program.

The Lumex RA-915 is applicable for ambient air testing of mercury vapor in the range of 20 ng/M<sup>3</sup> to 50,000 ng/M<sup>3</sup>. (50,000ng/M<sup>3</sup> = .05mg/M<sup>3</sup>, the PEL for mercury) For areas of higher concentration, a Jerome meter should be used. Do not directly expose the RA-915 to elemental mercury as this may permanently contaminate the instrument.

The Maine ambient air guideline for mercury is 300 ng/M<sup>3</sup>.

# Section 2 Purpose

The purpose of this document is to describe the procedure for collecting and analyzing air samples for mercury vapor analysis.

# Section 3 Responsibilities

Personnel conducting perimeter air monitoring and other site related air monitoring will be trained and must follow this procedure when using the Lumex RA-915 for mercury vapor analysis.

# **Section 4 Procedures**

### 4.1 Starting the Instrument

The instrument can be powered by either 120-v AC line current [with adapter cord], a battery pack in the instrument, or vehicle cigarette lighter adapter. The battery pack is intended for a maximum of 4 hours continuous use, and should be recharged using the included cord plugged into 120 volt AC line current. The instrument may be used with batteries if the battery indicator is flashing red. A steady red indicates the battery needs to be charged and AC power must be used to run the instrument. A supplemental battery pack is stored in the side pocket and can be plugged into the AC adapter cord port. The supplemental battery will provide an additional 2 hours use.

Pre-operational procedures:

- 1. Before operating the RA-915, conduct a visual inspection of the analyzer's component parts. The instrument may be used while in the carrying case.
- 2. Place the RA-915 in a horizontal position with the Palm monitor (controls and display screen) on top. The power switch will be the front end. (See photo 1)

- 3. Set the test cell control handle (on the side of instrument) to the **OFF** position. This can be accessed inside the side pocket of the carrying case.
- 4. Check to make sure Palm monitor is securely connected to the base unit.
- 5. The handle for optical bridge switch at the back and opposite the power switch should be pre-set to position III.
- 6. Turn on the power switch on front of the instrument. The Palm monitor will then show the Lumex version screen (See photo 2).
- 7. Press the "Ent" button on the Palm monitor. The MAIN MENU display will appear. There will be an \* next to the words MAIN MENU.
- 8. Press [3-5 sec] and release the Lamp Ignition button on front of the machine. When the lamp lights the \* next to the words MAIN MENU will disappear. Repeat this step as necessary to light the lamp.
- 9. Allow the instrument to warm up for 5 minutes prior to testing.

#### 4.2 Menu Screens

The MAIN MENU will have the following options:

- Parameter Used to change parameter settings (See below).
- On Stream Used to analyze background and environmental samples.
- On-time Not used for air analysis
- Test Used to verify instrument calibration.
- Settings Used to save new parameter settings or restore factory settings. This should not normally be used.

To select an option, highlight the option and push the Ent button.

To return to the main menu, push the Esc button.

Parameter settings for air analysis should generally follow preset values. The following settings have been stored:

Parameter	Value	Units
Average Time	1	seconds
Baseline Cor Time	20	seconds
Frame Time	10	seconds

Integr. Time	120	seconds
Low limit	20	ng/M <sup>3</sup>
High limit	300	ng/M <sup>3</sup>

#### 4.3 Background Air Analysis

Prior to taking the instrument to a potentially contaminated site, a background air sample should be analyzed to demonstrate that the instrument reading is below the reporting limit for this instrument,  $20 \text{ ng/M}^3$ .

A background sample must be taken at the beginning and end of each analysis day. All results must be below  $20 \text{ ng/M}^3$ . Do not proceed until this condition has been met.

For this analysis the Lumex RA-915 should be operated in the ON STREAM mode as described below:

#### Operation in ON STREAM (AIR ANALYSIS) mode

- 1. The optical bridge handle should already be in the OFF position as described in the starting instructions above [Section 4.1].
- 2. Use the arrow buttons, on the Palm monitor indication unit to select the ON STREAM mode and press the Ent button. This will switch the compressor on, and the zero signal will be measured. The following will occur on the display
  - The current S value, which corresponds to the mercury concentration in the pumped air in ng/M3, is displayed in the upper right of the palm monitor.
  - The Si level is also displayed below the S value. This result [Si] corresponds to the value S averaged over a given time range.
  - The bottom right displays a countdown [in seconds] of the time over which S values were averaged. The current setting is for values to be averaged over 10 seconds.
  - An **Alarm!!** Message is displayed across the top of the screen if the mercury concentration exceeds the ambient air guideline.
- 3. Press the **Ent** button is a second time: three Si readings and S<sub>c</sub> [the average of these three Si readings] are displayed. In this mode three 10-second average readings are repeated, averaged, and displayed with the corresponding relative deviation [R] in the measurements.
  - The average,  $S_c = (S1+S2+S3)/3$ .
  - The relative deviation of three measured concentrations is displayed as R

 $R = 100^{(max (S1,S2,S3) - min(S1,S2,S3))/S_{ave}}$ %.

- If S<sub>c</sub> is less than the parameter "Low limit" (20 ng/M<sup>3</sup>), "< 20" is displayed.
- 4. Record the three Si readings, S<sub>c</sub> and R for the background sample in a field notebook and any analysis record developed for the current sampling event.
- 5. If the background reading does not fall below  $20 \text{ ng/M}_3$ , remove the intake hose and repeat the procedure to determine whether the intake hose is contaminated.
- 6. To quit the On Stream mode, press the **ESC** button, which causes the air pump to switch off. The device switches over to the standby mode waiting for the next command. The message MAIN MENU appears on the Palm display.

#### 4.8 Calibration Verification

The instrument calibration must be verified on each analysis day prior to analyzing samples, and again at the end of the day.

The calibration is considered verified if the relative deviation [designated with R on the instrument] is below 20%.

Calibration verification is measured in the TEST mode as described below:

#### **Operation in the TEST Mode (Serviceability Check)**

- 1. Use arrow buttons on the indication unit to select the (TEST) mode and press the **Ent** button. After the instrument measures the zero signal the display will show the message "Enter Test Cell."
- 2. Set the test cell handle on the side of the instrument to the ON position, and wait for 20 seconds before pressing the **Ent** button. The following will be displayed:
  - The current S value, which represents the measured mercury concentration in the test cell in ng/M<sup>3</sup>;
  - The Sk value, which represents the mercury concentration which should be measured based on the test cell temperature; (See table on page 21 of the RA-915+ Operation Manual for reference);
  - The average measured mercury concentration [Si];
  - The relative deviation [R] of the measured value average [Si] from the theoretical value is automatically calculated by: R= 100\* | (Si- Sk)/Sk |; and
  - A countdown [in seconds] of the time over which Si values were averaged. The current setting is for values to be averaged over 10 seconds.

- The message *"Temperature"* is displayed across the top, if the temperature of the test cell is beyond the admissible temperature range for proper operation of the analyzer.
- 3. Record the Si, Sk, and R values associated with the calibration check in a field notebook and any analysis record developed for the current sampling event.
- 4. If the relative deviation [R] of the measured values Si from its table value is below 20%, the RA-915+ analyzer is ready for operating, otherwise see "Maintenance" in the Operation Manual.
- 5. To quit the TEST mode, press the ESC button whereupon the analyzer switches over to the standby mode for the removal of the test cell. The display will show the message Remove Test Cell. Remove test cells and press the ESC button again and the analyzer switches over to the standby mode waiting for the next command. The message appearing on the display reads MAIN MENU.

#### 4.5 Analysis

1. Allow the Lumex RA-915 to equilibrate to site temperature.

Sample locations should be selected in accordance with the pools of the parameter air -monitoring plan and considering the meterological conditions (e.g. wind directions of the day. It is important to note that environments with high levels of mercury are not suitable for the Lumex RA-915. Several precautions should be taken at possibly contaminated sites:

- Use a Jerome meter to delineate areas possibly contaminated above 0.05 mg/M<sup>3</sup>.
- If a Jerome meter is not available, start the investigation outside the possibly contaminated areas and work toward the contaminated areas stopping when the mercury readings exceed the calibration range of the instrument [0.05 mg/M<sup>3</sup>].
- Do not place the instrument on any potentially contaminated area, including floors or surfaces where mercury has been spilled.
- Do not place the inlet sample tube on any potentially contaminated surface.
- 2. Air temperature should also be measured and recorded concurrently with the mercury sample results. A digital thermometer is included in the travel case with the mercury analyzer for this use.
  - 1. For this analysis the Lumex RA-915 should be operated in the ON STREAM mode as described in section 4.3, making sure to record the three Si values, Sc, and R in a field notebook and any analysis record developed for the current sampling event.

- 5. Check a [low] background sample and calibration verification at the end of the sampling day.
- 6. To turn the instrument off press the Esc key to go to the main menu. Then turn the power toggle switch off. If the instrument was operated on battery power, the battery must be recharged prior to storing the instrument. Storing the instrument with an uncharged battery may cause damage to the battery.

#### 4.6 Instrument Maintenance and Storage

#### 4.6.1 Maintenance procedures for the analyzer include

- 1. Daily [when in use] visual inspection;
- 2. Periodic preventive maintenance;

All the maintenance operations should be duly recorded in the analyzer log.

**Daily [when in use] inspection is performed in the work place and involves** visual inspection of the analyzer and serviceability check. The service ability check consists of a background air check for contamination (section 4.3) and a calibration verification check (section 4.4).

# Periodic prevention maintenance is performed in the work place and involves:

- 1. Quarterly:
- Checking the fastening of the body covers;
- Checking the connectors for cleanness;
- Checking the state of the cables;
- Checking the dust filter: A small dust filter is located inside the intake hose attachment port. This filter should be checked on a quarterly basis [sooner if used in high dust areas] and replaced if the dust filter has turned color from white to brown and appears to be clogged. To remove the filter for inspection/ replacement, use a pair of tweezers; and
- The built-in absorption filter (located in the left-hand inlet on the front –wall of the base unit) should be replaced as needed. Typically this will be once or twice per year. If the instrument is used often, or in a mercury environment above 10,000ng/M<sup>3</sup> for a period of time the filter should be replaced more often.
- 2. Annually prevention maintenance is recommended. It is performed by OhioLumex and involves recalibration and checking the RA-915+ for conformity to the technical specifications. For further information refer to the Operation Manual. OhioLumex is in the process of updating the recommended maintenance

procedures for this instrument. When these updates are completed the maintenance section of this SOP will be modified to reflect any changes.

#### 4.6.2 Storage

- The instrument should be stored in a low mercury [<20 ng/M<sup>3</sup>] atmosphere at temperatures between 40°F and 100°F with relative humidity less than 80%. If it is inadvertently stored below 32°F, it should be taken to and kept at a temperature of 60°F or higher for up to 24 hours [temperature dependent] prior to use.
- When the analyzer is used with battery power, the battery must be recharged before returning the instrument to storage. Storage of a discharged battery for 3 days may permanently damage the battery.

### 4.8 Documentation

All sampling activities must be documented either in a field notebook or on preprinted sampling worksheets. At a minimum the following items must be documented:

- Project name;
- Date and time of sample;
- Background air results;
- Calibration verification results;
- Sample location;
- Name of person[s] performing air sampling/analysis;
- Temperature;
- Mercury result;
- Any special considerations or sampling conditions;

# 4.8 Quality Assurance/Quality Control4.81 QA Sample Collection

Collection and analysis of the following QA samples is mandatory:

Background sample - A background air sample should be taken outside of the site and in a location where there is no [low] mercury contamination. Results must be below 20 ng/ M<sup>3</sup>. This sample is taken to ensure that the instrument is free of contamination. At a minimum, background samples should be taken at the beginning an end of each sampling day. If the instrument is taken into an environment where mercury vapor concentrations exceed the calibration range of

the instrument [50,000 ng/  $M^3$ ] a background sample must be re-analyzed before continuing with the sampling event.

- Calibration verification The instrument calibration must be verified at the beginning and end of each sampling day. The calibration is considered verified if the relative deviation [designated with R on the instrument] is below 20%. The instrument must be returned to the factory for calibration yearly, and when calibration falls outside the designated range.
- Duplicate samples Each time a sample is analyzed the instrument automatically takes three 10 second average readings [Si] and averages the three readings to arrive at a result [Sc]. A relative deviation [displayed as R] is also calculated by the instrument by the following formula:

 $R = 100*(max(S1,S2,S3) - min(S1,S2,S3))/S_c$ 

 $S_c = (max(S1,S2,S3) + min(S1,S2,S3))/2$ 

#### 4.8.2 Deviations from Protocol

All deviations from the procedures outlined in this or in any other protocol must be documented in field notes.

## Section 5 References

- 1. Multifunctional Mercury Analyzer RA-915+ Operation Manual, OhioLumex Co, Inc. Analytical Equipment, Cleveland, Ohio, 2001.
- 2. Protocol for collecting and analyzing mercury vapor in air with a Lumex RA-915+Mercury Analyzer. Maine Department of Environmental Protection Standard Operating Procedure Hg01, April 2, 2002.

# APPENDIX B: TOTAL ATMOSPHERIC HG SAMPLING VIA THE FRONTIER SOLID SORBENT METHOD

# Total Atmospheric Hg Sampling via The Frontier Solid Sorbent Method

January 2003

Revised March 7, 2003

# Total Atmospheric Hg Sampling via The Frontier Solid Sorbent Method

# Section 1 Introduction

The following protocol is designed to instruct sampling personnel on how to perform ambient air mercury sampling on a weekly basis. The focus of this document is to give guidance to ensure trace clean sample handling techniques are followed and a standard protocol is followed to provide consistency. This protocol is intended to provide all of the detail needed to perform this sampling effort.

# Section 2 Sampling Protocol

This protocol covers the following steps:

- 1. Obtain the Mercury Sampling Supply Kit.
- 2. Record the Total Atmospheric Hg Solid Sorbent Trap Final Sample Volume.
- 3. Approach the Collector Sampling Arms From Down Wind.
- 4. Make Observations While the Sample Media Is Still In The Sampling Arm.
- 5. Perform A Post Sample Leak Check of the Total Hg Sorbent Trap.
- 6. Release the vacuum after performing the Post Leak Check.
- 7. Retrieve the Total Hg Solid Sorbent Trap.
- 8. Store The Samples and Field Data Clean The Sampling Arms.
- 9. Deploy "Dummy" Sample Media and Set Sample Media Flow Rate.
- 10. Deploy A New Total Hg Solid Sorbent Sample Trap.
- 11. Fill Out The Field Data Sheet.
- 12. Final Preparations Before Leaving Site.

## 2.1 Obtain the Mercury Sampling Supply Kit

This Kit should have the following items:

a. **1** *set of double zip-lock bagged gloves*. These specialized gloves should always be double bagged, in protective zip-lock bags and most importantly, kept sealed. These are class-100 vinyl clean gloves and are virtually particle free. Dirt and dust particles can be a source of low-level mercury contamination. Ambient air mercury is typically low in mercury and therefore it is



critical to hand the samples with particle free gloves. As a special warning, do not use latex gloves as they attract dirt and dust. Further, do not use powdered latex gloves as the powder has a measurable amount of mercury. Let Frontier know when more class-100 clean gloves are needed.

- b. *Sealed Zip-Lock Bag With Previous Weeks Total Hg Solid Sorbent Trap's End Plugs.* These are the white, Teflon end plugs you removed from the previous weeks Hg sample trap before deploying for sampling. The Teflon end plugs will be going back into the ends of this sample trap, in order to seal, protect and store the trap, prior to shipping back to Frontier. It is therefore critical to keep these end plugs, sealed in the bag during the sample period, so they remain clean.
- c. *Total Hg Solid Sorbent Trap For Flow Rate Settings.* This is the Total Hg Solid Sorbent Trap, when hooked up to the sample line and deployed, will help set the flow rate dial on the sample box to the proper flow rate for Total Atmospheric Hg capture. This is so the actual Total Hg Sample Trap, when deployed and activated later by a timer, will start at the desired flow rate. It is not necessary, however it is good practice to keep the flow rate setting Total Hg Sample Trap in a zip-lock bag for protection. Keep this bag and Solid Sorbent Trap clearly labeled to ensure it is not mistaken as an actual sample.
- d. *Sealed Zip-Lock Bag Paper Towels.* Paper towels are basically particulate free, mercury free and are suitable to clean off the PVC sampling arms and locations where the sample media resides to ensure the new sample media is being deployed in a clean environment.
- e. *Spray Bottle of Formula 409 cleaner*. Formula 409 is mercury free (Frontier tested it) and is an effective surfactant/degreaser. It is used on the PVC sampling arms and areas where the sample media resides to remove wind blown dust and particles.
- f. *Field Data Sheets.* A field form to record critical sampling information fore each sample: Site ID (location where the sampling occurred), Sample Type, Sample Start Date, Sample End Date, Sample Start Time, Sample End Time, Flow Rate Settings, Zero Offset, and Sample Volume.

### 2.2 Record the Total Atmospheric Hg Solid Sorbent Trap Volume

The Final Sample Volume for the Total Hg Solid Sorbent Trap is adjacent to the LCD display identified as "TAHg" which stands for Total Atmospheric Mercury. Record the final sample volume on the field form.

# 2.3 Approach the Collector Sampling Arms From Down Wind

With the Sampling Supply Kit and Field Data Sheet, proceed to the sample platform. Carefully approach the sampling arm that is holding the Solid Sorbent Trap (approach from down wind if possible). Try to avoid standing directly upwind of the open sample media. The sampler can be a potential source for mercury as hair and dirt/dust can blow off clothing and into sample media



# 2.4 Make Observations While the Sample Media is Still in the Sampling Arm

Inspect equipment and sample media, make observations, and record on field data sheet:

- Does everything appear to be as deployed?
- Is the sample media still secured and in its proper place?
- Does the sample arms appear to have an unusual amount of particulate?
- Was the site secured (no sign of anyone getting into the secured platform)?
- Were there any unusual conditions during the test run (wildfires in the area, wind/dust storm, heavy rain)?

### 2.5 Perform A Post Sample Leak Check of the Total Hg Solid Sorbent Trap

The purpose of this effort is to ensure that there was no leak in the sample line during the sample event. A successful Post Sample Leak Check is a good quality assurance measure and gives confidence that the sample taken is of high quality. A Post Sample Leak Check that fails indicates that there was a leak somewhere in the sample line and the sample volume measured is not the true sample volume as some of the air that passed through the system did not go through the sample trap.

- a. Obtain the Sealed Zip-Lock bag with the Teflon end plugs from the Solid Sorbent Trap.
- b. Open the Zip-Lock bag and place the bag upright in the plastic container (that holds all of the supplies).
- c. Don a pair of clean gloves.
- d. Without touching anything with your glove, reach into the open Zip-Lock that contains the Teflon end plugs and obtain one.
- e. Place the Teflon End Plug into the inlet of the Total Hg Solid Sorbent trap that is currently deployed.
- f. Proceed back to the sample box and double check the final sample volume recorded on your field data sheet for accuracy. Verify recorded value on your field data sheet.
- g. Record the Zero Offset from the Mass Flow Meter. The Zero-Offset is the reading on the Mass Flow Meter when there is no flow through the system. The Mass Flow Meter should be on for at least 15 minutes and the flow control valve should be off (turned all the way to the right). Read the zero-offset value and record on the field data sheet. This value should be close to zero



and could be positive or negative. This value will be used to correct for electronic drift in the sample flow rate.

- h. Record the Final Zero-Offset value.
- i. Turn on the sample pump.
- j. Open the flow control valve. Open the valve slowly and open to <sup>3</sup>/<sub>4</sub> of the full open flow.
- k. With the Teflon end plug in place, you are now evacuating the air from the sample media all the way back through the sample line to the mass flow meter. The flow rate should go to zero if this is a successful leak check.
- 1. A failed leak check is any mass flow reading that exceeds 0.07 standard liters per minute. If this occurs, you will need to begin to leak check the system starting at the sample box and progressing through the sample line to the sample media.

# 2.6 Release the vacuum after performing your Post Leak Check

After performing the post leak check, there is a vacuum behind your sample media. **Do not release the vacuum at the sample trap!** The sudden decompression behind the sample trap could push the sample media out of the trap or at least disturb the sample beds. This could lead to the loss of a sample.

Release the vacuum, after the post leak check, by unscrewing the Teflon swage fitting on the face plate of the sample box, labeled as the inlet for the VHg (0-5 slpm) side of the sample box. Unscrew the swage nut holding the sample line that leads to the Total Hg Solid Sorbent Trap. Slowly pull up the ¼ inch sample line out of the swage fitting, just for a moment. Immediately replace the sample line and secure the Teflon nut. Do not over tighten the Teflon nut. Over tightening the nut can actually cause a leak. Hand tight pressure is sufficient to make a seal.

# 2.7 Retrieve the Total Hg Solid Sorbent Trap

- a. Proceed to the sample arm holding the Total Hg Solid Sorbent trap. The Total Hg Solid Sorbent Trap is "housed" at the end of the sampling arm, inside a plastic junction box. The side of the junction box has a panel that can be opened. This panel will gives access to the sorbent trap and where the sorbent trap is connected to the sample line that runs back to the sampling box. Open the "door" on the junction box by removing the top screw that secures the top of the access panel to the box.
- b. Allow the door of the sample box to rotate down and hang off the screw at the bottom of the box that is holding the panel in place
- c. Don a new pair of class-100 clean gloves.



- d. Retrieve the sealed Zip-Lock bag that you saved when you deployed the solid sorbent sample trap. The zip-lock bag should have one more Teflon end plug in it (this from when you removed both end plugs when you deployed the trap).
- e. Label the sample bag with the Sample ID using a "Sharpee" or some other indelible marker with the following convention:

#### TAHg - Site ID - Sample Date

TAHg = Signifies Total Atmospheric Hg

Site ID = the name of the sampling location (i.e., HMC for HoltraChem)

**Sample Date =** the date that the actual sample was taken

Example: TAHg-HMC -02-15-03

- f. With a gloved hand, gently disconnect the sample trap from the sample line.
- g. Insert the last Teflon end plug into the open end.
- h. The trap is now sealed. Place the trap in the zip-lock bag and seal the bag.
- i. Record the Final Sample volume on the field data sheet.
- j. Record the Final Zero Offset for the Total Hg trap.

## 2.8 Store the Samples and Field Data Sheets

Put the field data sheet in with the sample media and seal in a zip-lock bag. Place in the refrigerator. Keeping the filters in a cool, dark atmosphere ensures that Hg does not thermally desorb off the ambient air particulate. The Total Hg Sorbent Traps are very stable with regards to mercury and therefore do not require refrigeration, however, it is best to keep all of the sample media together during this storage phase.

Store the sample media in the refrigerator until the end of each month. After the end of the month, send the sample media on ice, to Frontier in a cooler. Directions for shipping the sample media are presented as step 3.0.

# 2.9 Clean the Sampling Arms

Clean the sample arms and in particular, clean the areas where the sample media are normally deployed. Using Formula 409, spray down the plastic sample arms with this particle and grease removing surfactant. Use paper towels to wipe down and remove the Formula 409 (along with the particles etc).



## 2.10 Deploy "Dummy" Sample Media and Set Sample Media Flow Rates

- a. Obtain the "dummy" sample media.
- b. Wearing gloves (for practice/habit purposes only) obtain the Solid Sorbent Trap. Remove the Teflon end plugs and connect the outlet of the trap to the Teflon sample line. Run the Inlet of the sample trap down into the bottom hole intended to expose the inlet of the Total Hg Sample Trap.
- c. Once the dummy sample media is secured, proceed to the sample box.
- d. Turn the flow rate control knob on the Mass Flow Meter all the way off (to the right) until it stops.
- e. Plug the power cord in for the pump that powers the Total Hg Sorbent Trap. This is the pump that is connected to the sample box via the  $\frac{1}{4}$ " sample line.
- f. Slowly adjust the flow rate to 4.0 slpm by rotating the flow control valve to the left. Allow the system to continue to flow and stabilize to as close to 4.0 slpm as possible.
- g. When the flow rate signal has stabilized for the Solid Sorbent Trap, unplug the power cord.
- h. You have now set the flow rate for the sample media, so do not change the setting of the flow control knob.
- i. Plug pump power cord into the timer.
- j. Set the timer for the desired time for sampling to start and end.
- k. **IMPORTANT:** The power for the sample box itself should be plugged directly into a power socket. The sample box will need to always have power supplied to it. Therefore, do not plug the sample box power into the timing system used to turn on the pumps.

# 2.11 Deploy A New Total Hg Solid Sorbent Sample Trap

- a. Wearing clean gloves, remove the Solid Teflon End Plugs on either end of the sample trap.
- b. Place the Solid Teflon End Plugs into the Ziplock bag that the trap came in and seal the bag.
- c. There is a sample flow direction arrow on each trap. This is critically important. The arrow on the sample trap indicates how you should attach the sample trap to the sample line.
- d. Obtain the sample line inside the plastic junction box. Connect the sample line to the back of the sample trap. Again, the arrow on the trap should be pointing towards the sample line.
- e. With the trap connected to the sample line, insert the open end (inlet end of the sample trap) down into the opening of the junction box. The inlet end of the IC trap should only be 1-2



inches down from the bottom of the junction box. Adjust the sample line so it holds the sample trap at the proper location below the junction box.

f. Rotate the plastic panel on the junction box back to cover the junction box opening. Secure the panel with the screw. Make sure that this panel is properly sealed to ensure that no rain or dust get blown into the junction box. This is to keep the IC trap as clean as possible.

### 2.12 Fill Out The Field Data Sheet

Obtain a new Field Data Sheet. Record the following in the appropriate locations of the field data sheet:

- a. Record the Start Zero Offset For the Total Hg Solid Sorbent Trap. The Mass Flow Meter will display an "at rest" zero flow value.
- b. Record the Date that the sample was set up in the field.
- c. Record the Date that the sample will actually be activated to start sampling.
- d. Record the Site ID.
- e. Record the Sample ID:

```
(e.g. TAHg - HMC - Sample End Date)
```

Store the Field Data Sheet in a zip-lock bag inside the supplies container.

# 2.13 Final Preparations Before Leaving Site

- 1. Double check to ensure that pumps are plugged into the timer system.
- 2. Double check to ensure that the timer is set for the proper time and date required for sampling.
- 3. Push the red Totalizer Button to the right of the Total Volume display. This zeros the volume that is stored in memory.
- 4. Double check to ensure that sample box is plugged directly into a power socket and not into the timer. You want to be sure the power is always on for the sample box, otherwise, the mass flow reading could be very noisy from suddenly being turned off and on.

# Section 3 Sample Shipment

- 1. The filter packs and Sorbent Traps should all be packed well with some sort of packing material like bubble wrap to ensure the traps are not crushed by the blue ice (coolant packs) that will be used to keep the filter packs cool.
- 2. Line the bottom and sides of cooler with ice packs.



- 3. Ensure that each filter pack and each solid sorbent trap have field data sheets.
- 4. Place the extremely well packed bubble wrapped filter packs and traps in the cooler. Ensure that all of the spaces in the cooler are filled with packing material to ensure that things do not shift. If there is room in the cooler, the ice packs will move around and could damage the sample traps.
- 5. Fill out and place a Chain of Custody Record (COCR) in with each cooler.
- 6. Ship the cooler to the following address:

Attention: Bob Brunette Frontier Geosciences Inc. 414 Pontius Avenue North Seattle, Washington 98109 Phone: 206 622 6960

7. Ship the cooler with a sufficient priority so it arrives the next day. This to ensure the cooler ice packs keep the filter packs cool all the way to Frontier. FedEx Standard Overnight service is recommended.

# Section 4 Sample Media Supply and Sampling Supplies

Frontier will supply the following on a monthly basis:

- Sample media;
- Vinyl clean gloves;
- Field Forms; and
- Chain of Custody Records.
- Formula 409 and paper towels purchase at local store.

# Section 5 Field Sampling Questions and Trouble Shooting

If you have problems with your equipment or have questions, please call the CDM Project Manager, Ernest Ashley at 1-800-343-7004, or the Frontier Project Manager, Bob Brunette toll free at: 1-877- 622-6960.

### **Section 6 References**

Gas and Particulate Phase Hg Ambient Air Sampling Via Frontier Solid Sorbent and Total Particulate Hg Method Frontier Geosciences, December 2002.





#### Total Atmospheric Mercury Field Sampling Record

	Sample ID	HMC	TAHg	<u>Sta. No.</u>	<u>mmddyy</u>	
	Sample ID		TAng			
Due Generikeer Leele Cherche		D	I <sup>2</sup> T			
Pre-Sampling Leak Check:		Post Sam	ping Lea	IK Uneck		
Sampling Media Data						
		Dat	e		Time	
Sample Me	dia Deployed:					
Sample Media Beg	an Sampling:					
Sample Media Stopp	ed Sampling:					
Sample Mee	lia Retrieved:					
Weather Conditions During	g Sampling:					
Temperature:	Wind Speed:	Wind Direction:				
Sampling Data						
Pre-Sampling Zero Offset <sup>1</sup> :						
Post-Sampling						
Final Sample Vol	Final Sample Volume (Liters):					
Observations:      All sampling equipment in good operating condition      See comments below						
Somplor Nome						
Sampler Name:	2	Signature:				

<sup>&</sup>lt;sup>1</sup> Zero Offset = Mass Flow Meter reading with no flow.

# APPENDIX C: LABORATORY ANALYTICAL SOPS

### Total Mercury Digest for Gas/Air Samples Collected on Frontier Sorbent Total Mercury (FSTM) Traps FGS-009.2

Frontier Geosciences Inc. 414 Pontius Avenue North Seattle, WA 98109

Originated by: Nicolas S Bloom and Eric M. Prestbo Revised by: Paul Laskowski January 3, 2000

Effective Date: June 27, 2000

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On June 27, 2000, this procedure was reviewed and validated by Michelle L. Gauthier, Laboratory Manager and Beverly H. van Buuren, Quality Assurance Program Director. Signatures are on file.

#### **1.0 SCOPE AND APPLICATION**

1.1. This method is a peer-reviewed, published procedure for the determination of total mercury in air and gas samples collected on dry sorbant traps. All samples must be subjected to an appropriate leaching step, as described herein, prior to analysis by CVAFS.

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1.2. The typical estimated method detection limit as derived from the standard deviation of the blank traps is 0.2 ng Hg/sample trap for a  $1m^3$  sample.

#### 2.0 SUMMARY OF METHOD

2.1. FSTM traps with air samples collected on them are subjected to a hot (50-60 °C) leaching with a HNO3/H2SO4 mixture of concentrated acids for 1.5 hrs. The leachate is then diluted up with BrCl on the day of analysis by CVAFS method and analyzed according to FGS-069.

#### **3.0 INTERFERENCES**

- 3.1. The carbon granules are not dissolved by this procedure, but experience shows that this strong acid leach is sufficient to extract all collected Hg that has adsorbed on the surface. It is critical, however, that the final solution contains a sufficient volume of strong acids to avoid re-adsorption of Hg to the carbon granules.
- 3.2. Due to the amount of FSTM compounds that leach into the digests, a maximum aliquot size of 1.0 mL for each digested trap is used. These compounds have the ability to overwhelm the acid traps on the CVAFS analyzer making for high blanks, and to destroy the gold sample collection traps.

#### 4.0 SAFETY

- 4.1. Personnel will don appropriate laboratory attire according to the Chemical Hygiene Plan. This includes, but is not limited to, laboratory coat, safety goggles, and latex gloves under clean gloves.
- 4.2. The toxicity or carcinogenicity of reagents used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. All laboratory personnel should refer to the MSDS for each chemical they are

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working with.

- 4.3. All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known diseasecausative agents. Frontier will reimburse the expense of Hepatitis A and B immunizations for any laboratory staff member who desires this protection.
- 4.4. Waste Management and Pollution Prevention: Any wastes generated by this procedure should be disposed of according to SOPs FGS-099 and FGS-100, which provide instruction on dealing with laboratory and client waste. Pollution prevention information can be found in the current Frontier Geosciences Hazardous Materials Management Plan (HMMP), which details and tracks various waste streams and disposal procedures.

#### **5.0 PROCEDURE**

- 5.1. Each FSTM trap has two sections; the first one exposed to the flow of the gaseous sample is referred to as the FSTM-A trap. The one behind the FSTM-A trap in the flow stream is referred to as the FSTM-B trap. Find the direction flow, usually indicated by an arrow somewhere on the trap, to determine which section is which.
- 5.2. In a clean hood, prepare a digestion vial with the appropriate sample ID and remove the lid. Neutralize as much of the static charge as you can that may reside on the rim and inside the vial with an appropriate anti-static device.
- 5.3. Pull all fittings and shrink fit tubing from the trap and, using a glass tube cutter, open the trap carefully just in front of the plug that holds in the FSTM-A granules. Neutralize as much of the static charge as you can that may reside on and within the FSTM trap.
- 5.4. Clean off the reaming rod with a squirt of ethanol on a kim-wipe. Use it to push the contents of the IC trap from the back of the FSTM-B section until the first foam plug falls into the digestion vial, along with all the FSTM granules up to the middle foam plug. Tap trap to rim of vial to release any loose granules. Hold trap opening over vial while pushing the FSTM-B trap back to it's starting

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position. This should release any granules that may be stuck to the middle foam plug.

- 5.5. If the FSTM-B trap for this sample is to be digested, clean off the reaming rod again and use it to push the rest of the contents into a separate digestion vial.
- 5.6. Cap off undigested FSTM-B traps into their tube using the red plastic end plugs that are distributed with the FSTM traps (return to labeled container if the label is not directly affixed to the trap itself.
- 5.7. Add the nitric/sulfuric mixture to the digestion vial immediately after collecting the trap sections into the vial, and cap tightly. This will prevent loss of the granule due to a buildup of static charge, which makes granules "jump" out of the vial.
- 5.8. After all the sample traps are collected into the digest vials, the digest vials are then heated for 2.0 hours on a hot plate, and then allowed to cool to room temp.
- 5.9. The samples are then diluted up to their digestion vial's capacity with BrCl. The carbon granules are not dissolved by this procedure, but experience shows that this strong acid leach is sufficient to extract all adsorbed Hg.

#### 6.0 QUALITY ASSURANCE

- 6.1. Maximum Sample Batch Size: 50 samples for standard level QC; 40 samples for high level QC.
- 6.2. Preparation Blanks: 30 percent of the FSTM-B traps are analyzed out of a batch of samples as both a check for breakthrough from the FSTM-A traps, and as the preparation blanks for correction by the mercury inherent in the reagents and on the FSTM traps. Three times the standard deviation is used to determine estimated MDL for each batch.
- 6.3. Analytical Duplicate: Two per batch, 25 RPD limit.

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6.4. Analytical Spike: Two per batch, 75-125% recovery limit. As there is no way to do digestion duplicates, a spike of 1.0 ng of mercury standard goes into the bubbler with the sample aliquot.

#### 7.0 CORRECTIVE ACTIONS

7.1. Since the entire sample is used in the digestion process, it is imperative to do the digestion correctly the first time. This primarily relates to limiting sources of contamination. Including, but not limited to, making sure none of the "A" trap FSTM particles are included in the digestion of the "B" trap, flushing repipettors a minimum of three times before adding reagents to samples, cleaning digestion tools (i.e. reaming rod) between each sample, changing clean gloves when appropriate, and in general following all ultra-clean procedures.

#### Total Mercury Analysis by Cold Vapor - Atomic Fluorescence Spectrometry (CV-AFS) FGS-069.2

Frontier Geosciences Inc. 414 Pontius Avenue North Seattle, WA 98109

Originated by: Sarah DuBord, Lucas Hawkins, Dustin Leen, and Amber Steward Originated: May 10, 2000

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Last Revision: June 8, 2001

Effective Date: June 11, 2001

On June 8, 2001, this procedure was reviewed and validated by Michelle L. Gauthier, Laboratory Manager and Beverly H. van Buuren, Quality Assurance Program Director. Signatures are on file

#### **1.0 SCOPE AND APPLICATION**

1.1. This SOP is designed to ensure that reproducible, traceable procedures are followed in the standardization of the total mercury analyzers and in the analysis

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of samples for total mercury, as well as to establish the bounds wherein data will be considered acceptable.

- 1.2. This method provides for the determination of total mercury in a wide range of matrices including aqueous, biologic, and geologic media. In general, using clean handling and reagents, the typical detection limit for the method is less than 0.2 ng/L for aqueous samples and 0.5 ng/L for digested solid samples or 0.5 ng/g as Hg.
- 1.3. This SOP consists of three aspects: (1) preparation of total mercury standard solutions, (2) calibration sequence of the total mercury analyzers, and (3) analysis of samples for total mercury as well as Hg (II) determination for tissues.

#### 2.0 SUMMARY OF METHOD

- 2.1. Preparation of Total Mercury Standards
  - 2.1.1. Mercury (Hg) standard solutions are prepared using ultra-clean class volumetric glassware and gravimetrically calibrated pipettors. All waters and reagents are pre-tested and must have very low Hg concentrations. All standard solutions, preparations, and calibrations must be logged in the Mercury Standard Logbook upon receipt or creation and given a unique identification number.
  - 2.1.2. Any standard, along with its original documents, should be labeled with the receipt date and the receiver's initials. All documentation should be given to the QA Office. The QA Office is responsible for maintaining standard records as well as updating the folders around the laboratory where copies are kept.
- 2.2. Total Mercury Analyzer Calibration Sequence
  - 2.2.1. The calibration sequence for the determination of total mercury consists of a 5-point curve (0.05 ng, 0.10 ng, 0.50 ng, 2.00 ng, and 4.00 ng), an initial calibration verification (ICV) standard, and an initial calibration blank (ICB).
  - 2.2.2. The calibration standard is made from a dilution of NIST-3133; in most cases, the higest calibration standard determines the range of sample concentrations that are considered valid.

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- 2.2.3. The ICV standard is a second-source standard that is made from diluted NIST 1641d; it verifies the accuracy of the standard used for the calibration curve.
- 2.2.4. The ICB is used to show that the system is low in total mercury as well as to allow for blank correction of standard curve.
- 2.3. Total Mercury Analysis
  - 2.3.1. All total mercury analyses receive a unique dataset identifier. This is composed of the instrument type and number, the date, and the calibration number for that day. The format is as follows: THG8-010224-1 where "THG" refers to a total mercury analyzer, "8" refers to total mercury analyzer number 8, "010224" refers to the date (February 24, 2001, in YYMMDD format), and "1" refers to the first calibration of that day.
  - 2.3.2. Total mercury analyses are split into two categories: waters and solids. For analysis of aqueous samples, an aliquot of oxidized sample is neutralized with hydroxylamine-hydrochloride (NH<sub>2</sub>OH-HCl) and added to a bubbler. For solids, an aliquot of digested sample is directly pipetted into the bubbler.
  - 2.3.3. For the analysis of waters or solids, stannous chloride  $(SnCl_2)$  is added to reduce the solution, and the bubblers are sealed with Keck Clips. Blanked gold traps are placed at the end of the soda-lime traps. The bubbler is purged with nitrogen  $(N_2)$  for 20 minutes. All gas that flows into the bubbler should only leave the system through the soda-lime trap and then gold trap.
  - 2.3.4. The gaseous mercury amalgamates to the gold traps, which are removed and sequentially placed, one at a time, in the analytical train. The gold trap is heated, thus releasing the mercury into the argon gas stream, which flows into the analyzer. As sample peaks are produced by the strip chart recorder, each peak is labeled with that sample's unique ID.

#### **3.0 INTERFERENCES**

- 3.1. Due to the high levels of acid and halogens (i.e., bromine) in digested solids, it is recommended that aliquots of no more then 5.0 mL (1.0 mL if hydrofluoric acid is present at significant concentrations) of the digestates be analyzed, unless otherwise instructed by a Project Manager (PM) or Group Leader.
- 3.2. When running digested solid samples, bubbler water should be changed and

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purged after a total of 10 mL of digestate has been added to the bubbler. This is done to avoid a build up of acidity and halogens in the bubbler water that can result in low sample recoveries as well as a drop in analyzer sensitivity. These interferences are most evident while analyzing digested QC samples.

- 3.3. Water has the potential to create recovery interference. To minimize, if not prevent interference from water, ensure that your soda-lime pre-traps remain dry. All handling of gold traps should be done using dry class-100 clean gloves to prevent water droplets from entering analytical train.
- 3.4. The presence of high concentrations of silver and/or gold can cause the SnCl<sub>2</sub> to precipitate out and adhere to the bubbler walls. High concentrations of these metals can sometimes be found in the matrix spike samples from digestion sets that are being shared with the Trace Metals Group. When analyzing digestates where the matrix spike samples have been spiked with silver or gold, the matrix spike samples should not be analyzed for mercury. Instead, an analytical spike/analytical spike duplicate (AS/ASD) should be analyzed.

#### 4.0 SAFETY

- 4.1. Personnel will don appropriate laboratory attire according to the Chemical Hygiene Plan. This includes, but is not limited to, laboratory coat, safety goggles, and latex gloves under clean gloves.
- 4.2. The toxicity or carcinogenicity of reagents used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Chemists should refer to the MSDS for each chemical they are working with.
- 4.3. All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known diseasecausative agents. Frontier will reimburse the expense of Hepatitis A and B immunizations for any laboratory staff member who desires this protection.
- 4.4. Waste Management and Pollution Prevention: Any wastes generated by this procedure should be disposed of according to SOPs FGS-099 and FGS-100,

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which provide instruction on dealing with laboratory and client waste. Pollution prevention information can be found in the current Frontier Geosciences Hazardous Materials Management Plan (HMMP), which details and tracks various waste streams and disposal procedures.

- 4.4.1. Quality Control procedures for aqueous samples
  - 4.4.1.1.A high-QA analytical batch is defined as 20 or fewer field samples, not including PBWs, ICB, CCV, CCB and MD/MS/MSD. A standard-QA batch is 25 or fewer field samples.
  - 4.4.1.2.A minimum three PBWs are to be performed per analytical day.
  - 4.4.1.3. A ML spike must be performed for each analytical day. For waters, the ML is 0.05 ng added to a clean bubbler. Similar to the analysis of the lowest standard. For all other preparations, the ML is digested with the sample set.
  - 4.4.1.4.One CRM (200  $\mu$ L of 1/200 diluted NIST 1641d) must be analyzed for each analytical day. In the case for waters, this requirement is fulfilled using the NIST 1641d for the ICV of the calibration.
  - 4.4.1.5.One CCV/CCB must be performed every 10 analytical runs for High-QA samples (can be stretched to 11-12 samples for Standard-QA).
  - 4.4.1.6.One Matrix Spike/Matrix Spike Duplicate (MS/MSD) must be performed every 20 samples (25 if standard QA). MS/MSDs are spiked at 1-5 times the ambient concentration, with 0.1 ng being the minimum spiking level. Sample aliquots for MS/MSDs should be the same as the ambient sample aliquot. Spikes are added to the split aliquots for volumes of 10 mL or greater. For less than 10 mL aliquots, spikes are added directly to the bubbler. **NEVER ADD SPIKE DIRECTLY TO ORIGINAL SAMPLE UNLESS OTHERWISE STATED.**
  - 4.4.1.7. A Matrix Duplicate (MD) is required every 20 samples (25 if standard QA). A Matrix Triplicate (MT) sample may be request by the PM. MD/MT should be performed using the same sample aliquots as the ambient sample

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- 4.4.1.8.A Project Manager may ask for a blank spike to be performed. Specific clients have mandatory spiking levels regarding blank spikes. Should a blank spike need to be analyzed, it needs to be 100 mL of reagent water with usually 1-2% BrCl and spiked at requested levels. The created blank spike would be treated just like a normal aqueous sample, and would be noted in the mercury standards logbook.
- 4.4.1.9. The analytical day must end with a CCV/CCB.
- 4.4.1.10. All standards used for analysis must be noted on the dataset spreadsheet.
- 4.5. Analysis of Frontier Sorbent Total Mercury (FSTM) Traps and Particulate Filters
  - 4.5.1. FSTM traps and particulate filters are digested and analyzed as if they were solids (see section 7.8.5), with a few exceptions. The FSTM matrix can cause recovery problems, as well as questionable blanks. Therefore there are a few modifications to the analysis and QC requirements. Due to the complexity of this analysis, inexperienced analyst should not analyze FSTM digests.
    - 4.5.1.1. FSTM traps or particulate filters should be digested according to SOP FGS-011.
    - 4.5.1.2.Aliquot sizes used for analysis should be 1 mL or less. 5 mL may be used at PM request, but not recommended. Should the bubbler contain more than 2 mL of digestate, the water must be changed and repurged before continuing analysis. It is recommended that the smallest possible aliquot sizes be used for this matrix, ensuring that you maintain quality data. All blanks, blank spikes, and low level/nondetect samples must be analyzed at the same aliquot size.
  - 4.5.2. At the end of this analysis, bubblers must be cleaned using potassium hydroxide (KOH).
- 4.6. Quality Control Requirements for IC Traps and Particulate Filters
  - 4.6.1. A minimum of three preparation blanks per batch: PBFSTM for FSTM traps, PBF for filters. PBFSTMs must use carbon tubes from the same lot number as the samples.

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- 4.6.2. A CCV/CCB must be performed every 10 analytical runs.
- 4.6.3. A blank MS/MSD pair digested at 100 ng/trap, using carbon tubes from the same lot number as the samples, must be analyzed for each batch.

#### **5.0 QUALITY ASSURANCE**

- 5.1. Analysts are to verify QC sample results in "real-time" as they come off the instrument. This allows for correction of any analytical problems immediately.
- 5.2. The acceptable recoveries must be met in order to consider a data set valid. Of particular importance to the client is Frontier's position that a **single non-compliant result on a QC sample does not automatically invalidate a data set**. All data points noted on the analysis day's spreadsheet as invalid for know reasons may be discarded if rerun during the same analysis day. In the event that the system becomes out of control during the analysis day, all results bracketed between valid QC data points shall still be considered valid.

QC Item	Acceptance Criteria		
ICV (CRM for aqueous samples)	80-120% recovery (Goal: 90-110%)		
ICB and CCBs	Individual limit of $\leq 0.25$ ng/L		
	(Instrument blanks are not blank corrected)		
CRM (for solids samples) or Blank Spike	75-125% recovery		
Calibration curve correlation coefficient	$r \ge 0.995$ , linear regression forced through		
(minimum 5 points)	zero		
CCVs	80-120% recovery		
PBWs	Each blank $\leq 0.25$ ng/L with SD $\leq 0.15$		
	ng/L; 3 per analytical day (3 per calibration		
	curve)		
PBSs	Mean $\leq 0.5$ ng (or $<0.1x$ lowest sample)		
	3 per analytical batch of $\leq 20$ samples		
	$(\leq 25 \text{ for standard QA})$		
PBTs	Mean $\leq 0.5$ ng (or $\leq 0.1$ x lowest sample)		
	3 per analytical batch of $\leq 20$ samples		
	$(\leq 25 \text{ for standard QA})$		
MS/MSD	75-125% recovery with RPD $\leq 25$		
	1 per analytical batch $\leq$ 20 samples		
	$(\leq 25 \text{ for standard QA})$		
MD/MT	$\leq$ 25 RPD/RSD		
Blank Spike (if requested)	75-125%		
Minimum level spike (ML)	1 per matrix per analytical day		

#### **Quality Control Limits for Determination of Total Hg \***

\*Client QC requirements may be more stringent. Please refer to project sheets prior to analysis.

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#### **6.0 CORRECTIVE ACTION**

- 6.1. The Quality Control data gathered throughout the analytical day provides an indication of overall data quality. Therefore, corrective action is required if quality assurance measures are outside of acceptable limits. First, a careful re-examination of the calculations is performed to assure that there are no numerical errors. The Project Manager is informed of any data issue, and they decide what, if any, corrective action, including reruns, is warranted. The Quality Assurance Officer oversees this process and has the final say in what corrective action is to be performed.
- 6.2. If insufficient sample volume remains to repeat analysis for samples analyzed after the last acceptable CCV, use best professional judgment to estimate values (usually through CCV correction). Bracket those samples from previous acceptable QC data points, and provide a narrative explanation on the dataset coversheet.
- 6.3. The above corrective actions apply only to events which have unknown causes. If the analyst is aware of the cause, no corrective action is necessary other that reanalyzing the sample.

#### 7.0 EQUATIONS

- 7.1. Total Mercury in Water
  - 7.1.1. Average all bubbler blanks (B) using the peak height/peak area values from the strip chart/integrator.
  - 7.1.2. The slope of the calibration curve (A) is calculated using the chart units per ng of mercury. A standard statistical package is used to determine the slope, using the five initial calibration points. The calibration points are first corrected by subtraction of the mean bubbler blank. The statistical program forces the regression line through zero (0,0). Average the results for the preparation blanks (PB), from the chart values of at least three preparation blanks.
  - 7.1.3. To calculate total mercury in waters (ng/L), use the following equations:

7.1.3.1.THg/Aliquot (ng) = [(Peak Height or Peak Area)-B]/A

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7.1.3.2.THg Gross (ng/L) =  $[(THg/Aliquot)/V_a/D]*(1000 \text{ mL/L})$ 

7.1.3.3.THg Net (ng/L) = [(THg Gross)/F]-(PB\*X)

Where:

- **B** is the average bubbler blank peak height or peak area (in units)
- A is the slope of the calibration curve (in ng/units)
- $V_a$  is the volume of sample analyzed (the aliquot size) in mL
- **D** takes into account any dilution of the sample and is expressed as a fraction (0.2 = 1/5 dilution)
- **F** equals: 1-[BrCl%/(1+BrCl%)]; F is expressed as a fraction which accounts for the dilution of a sample from the addition of BrCl. Taking a 100 mL aliquot of a sample preserved at 5% BrCl would result in F = 0.9524. This corresponds to the percentage of 100 mL volume which actual sample
- **PB** is the average of the preparation blanks in ng/L
- X is an integer related to the preservation level of the samples (i.e., X=2 for a sample which is preserved at 2% BrCl), thus accounting for the extra contribution of mercury in the BrCl
- 7.2. Total Mercury in Solids
  - 7.2.1. To calculate total mercury in a solid (ng/g), use the following equations:

7.2.1.1.THg/Aliquot (ng) = [(Peak Height Or Peak Area)-B]/A

7.2.1.2.THg Gross (ng/L) =  $[(THg/Aliquot)/V_a/D]*(1000 \text{ mL/L})$ 

7.2.1.3.THg/Digest (ng) = [(THg Gross-PB)\* $V_d/(1000 \text{ mL/L})$ ]

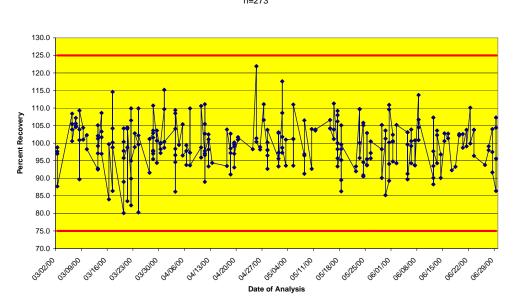
7.2.1.4.THg Solid (ng/g) = (THg/Digest)/m

Where:

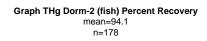
- **B** is the average bubbler blank peak height or peak area (in units)
- A is the slope of the calibration curve (in ng/units)

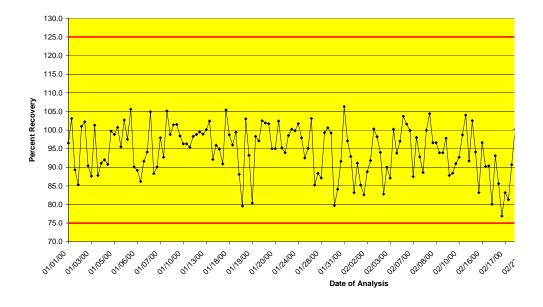
- $V_a$  is the volume of sample analyzed (the aliquot size) in mL
- $V_d$  is the final digested volume of the digest in mL
- **D** takes into account any dilution of the sample and is expressed as a fraction (0.2 = 1/5 dilution)
- **m** is the mass of the sample that can be expressed as either a dry or wet weight. (in grams)
- **PB** is the average of the preparation blanks in ng/L

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THg NIST 1641d Percent Recovery mean=99.7 n=273

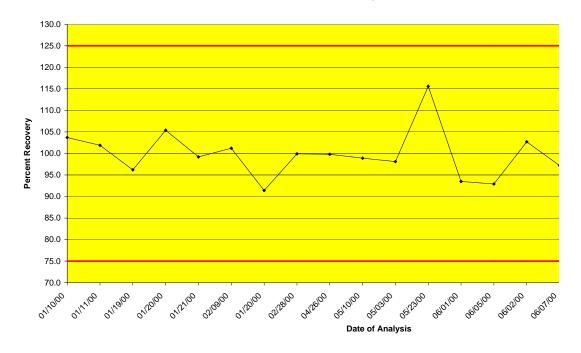




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#### THg NIST-2709 (soil) Percent Recovery mean=100.1 n=20



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