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General Electric Company
Pittsfield, Massachusetts

Field Sampling Plan / Quality Assurance Project Plan

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Volume II of III

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Volume II - Standard Operating Procedures for Field Based Activities

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Appendix A

Soil Sampling Procedures for Analysis of Volatile Organic Compounds (VOCs)

Soil Sampling Procedures for Analysis of Volatile Organic Compounds (VOCs)

I. Introduction

This standard operating procedure (SOP) describes the field sampling procedures to collect soil samples for the analysis of volatile organic compounds (VOCs). Soil samples will be collected in a manner that will minimize the loss of VOCs through volatilization and biodegradation. This SOP presents the procedures to collect soil and sediment samples for low-level (sample concentrations less than 200 µg/Kg, wet weight) and high-level (sample concentrations greater than 200 µg/Kg, wet weight) VOC analyses using field preservation techniques, the procedures to collect soil and sediment samples without field preservation, and the procedures for collection of soil and sediment samples using EnCore®, SoilCore®, or equivalent samplers. In order to minimize the handling of preservatives (i.e., sodium bisulfate or methanol) in the field, the preferred sample collection method for VOC analysis will employ the EnCore®, SoilCore®, or equivalent methods. The soil collection procedure to be utilized at each area will be presented in the project-specific work plan.

II. Materials

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

- Health and safety equipment (as required in the Health and Safety Plan);
- Photoionization detector (PID);
- Modified 10 - 30 mL plastic syringe;
    1. Cut off the injection tip,
    2. Depending upon the construction of the syringe, cut small air vents into the plunger or remove the rubber tip and retaining post.
- Stainless steel spatula;
- EnCore® Sampler T-Handle;
- Resealable-type bags;
- Handiwipes or lint-free paper towels;
- Field notebook;

- Appropriate sample containers depending upon specific methodology (4-oz glass jar with Teflon-lined cap, pre-preserved 40 mL vial with septum seal, EnCore®, SoilCore®, or equivalent Sampler); and
- Appropriate transport containers (coolers) with water ice and appropriate labeling, packing, and shipping materials.

III. Field Sampling

Option 1 - Collection of Unpreserved Sample

Step 1 - Soil samples are collected directly from the split-spoon using a stainless steel decontaminated spatula.

Step 2 - Samples are collected in 4-oz (120 mL) wide-mouth glass jars with Teflon-lined screw caps.

Step 3 - Wipe the threads of the sample jar with a handiwipe or lint-free paper towel, to ensure an adequate seal.

Step 4 - Place sample containers in a transportation cooler on ice immediately after collection. Package and label the sample containers following the procedures specified in Appendix L.

Option 2A - EnCore® Sampler

Step 1 - Place EnCore® sample container into the EnCore® T-handle.

Step 2 - Collect soil sample by pressing the EnCore® sample container into the soil to be collected.

Step 3 - Wipe the outside of the EnCore® sample container with a handiwipe or lint-free paper towel to ensure an adequate seal.

Step 4 - Using the T-handle, cap and lock the EnCore® Sampler for shipment.

Step 5 - If low-level VOC analysis is to be performed, repeat Steps 1 through 3 two additional times to collect a total of 3 samples (high-level VOC analysis only requires 1 EnCore® Sampler).

Step 6 - Collect additional soil in a 4-oz (120 mL) jar for percent moisture determination.

Step 7 - Place sample container in a transportation cooler on ice immediately after collection. Package and label the sample container following the procedures in Appendix L.

Option 2B - SoilCore® Sampler

Step 1 - Collect soil sample by pressing the SoilCore® sample container into the soil to be collected. A decontaminated stainless steel spatula may be used to assist this procedure.

Step 2 - Wipe the outside of the SoilCore® sample container with a handiwipe or lint-free paper towel to ensure an adequate seal.

Step 3 - Cap the filled end of the SoilCore® sample container.

Step 4 - Fill the opposite side of the SoilCore® sample container by pushing the empty side of the SoilCore® into the soil to be collected. A decontaminated stainless steel spatula may be used to assist this procedure.

Step 5 - Wipe the outside of the SoilCore® sample container with a handiwipe or lint-free paper towel to ensure an adequate seal.

Step 6 - Cap the filled end of the SoilCoreJ sample container.

Step 7 - Place the locking strap around both endcaps and pull the strap tight to secure endcaps. Check the position of endcaps after tightening to verify that the SoilCore® container is properly sealed.

Step 8 - Collect additional soil in a 4-oz (120 mL) jar for percent moisture determination.

Step 9 - Place sample container in a transportation cooler on ice immediately after collection. Package and label the sample container following the procedures in Appendix L.

Option 3 - Field Preservation

Step 1 - Order the appropriate number of pre-preserved sample containers (sodium bisulfate and/or methanol) from the laboratory. Instruct the laboratory to supply the sample container (with preservative) tare weights with the bottle order.

Step 2 - Prior to sample collection, weigh each sample container and compare the weight to the laboratory supplied tare weight to ensure that loss of preservative has not occurred. **Do not use sample containers that do not agree within ∀ 0.2 grams of the laboratory tare weight.**

Step 3 - Remove the plunger from a decontaminated sampler (modified plastic syringe).

Step 4 - Immediately after the surface of the soil is exposed to the atmosphere collect approximately 5.0 grams of sample by inserting the sampler into the soil. A decontaminated stainless steel spatula may be used to assist this procedure.

Step 5 - Carefully wipe the outside of the sampler with a handiwipe or lint-free towel.

Step 6 - Extrude the sample directly into the pre-preserved sample vial supplied by the laboratory. Avoid splashing the preservative solution.

Step 7 - Wipe the threads of the sample jar with a handiwipe or lint-free paper towel, to ensure an adequate seal.

Step 8 - Weigh the sample container and record the weight on the chain-of-custody form. Add a note on the chain-of-custody form to inform the laboratory to contact the project QA/QC Manager if the laboratory measured sample weight does not agree within ∀ 0.2 grams of the weight recorded on the chain-of-custody form.

Step 9 - For each sample location repeat steps 1 through 5 collecting two replicate samples for <u>each</u> method (low-level and/or high-level) in the appropriate sample vials.

Step 10- Using a stainless steel decontaminated spatula, place approximately 30 grams of soil from each location into a 4-oz glass jar for dry weight determination.

Step 11- Place sample containers in a transportation cooler on ice immediately after collection. Package and label the sample containers following the procedures specified in Appendices L and M. **Do not add tape, custody seals, or any other material to the pre-preserved sample containers (sodium bisulfate or methanol) that will alter the weight of the container.**

<u>NOTE</u> Soil samples which contain carbonate minerals may effervesce upon contact with the acidic preservative solution in the low concentration sample vials. If a rapid or vigorous reaction occurs discard the sample and collect the sample in a vial that does not contain the preservative solution.

III. Field Cleaning Procedures

Cleaning of VOC sampling equipment (e.g., stainless-steel sampling tools) is to follow procedures presented in Appendix W. The sampling equipment is to be cleaned prior to the start of sampling activities, between samples, and following the completion of sampling activities.

IV. Disposal Methods

Rinse water, PPE, and other residuals generated during the equipment cleaning procedures will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its ongoing disposal practices.

Appendix B

Soil Sampling Procedures for Analysis of Extractable Petroleum Hydrocarbons (EPH)/Volatile Petroleum Hydrocarbons (VPH)

Soil Sampling Procedures for Analysis of Extractable Petroleum Hydrocarbons (EPH)/Volatile Petroleum Hydrocarbons (VPH))

I. Introduction

This standard operating procedure (SOP) describes the field sampling procedures to collect soil samples for the analysis of volatile petroleum hydrocarbons (VPH) and extractable petroleum hydrocarbons (EPH). Soil samples must be collected in a manner that will minimize the loss of VPHs through volatilization and biodegradation. This SOP presents the procedures to collect soil and sediment samples for both VPH and EPH analyses. The VPH method involves the collection of 15 grams of soil in a modified plastic syringe and transfer of the soil into a 40 mL sample vial that has been preserved with 1 mL of methanol for every gram of sample. Alternatively, as directed by the project-specific work plan, VPH samples may be collected without field preservation by using EnCore®, SoilCore®, or equivalent Samplers. The EPH method involves the collection of soil in a 4-oz wide-mouth glass jar.

II. Materials

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

- Health and safety equipment (as required in the Health and Safety Plan);
- Photoionization detector (PID);
- Syringe (30 mL, with tip cut off);
- Stainless steel spatula;
- EnCore® Sampler T-handle;
- EnCore®, SoilCore®, or equivalent Sample Containers;
- Resealable-type bags;
- Handiwipes or lint-free paper towels;
- Field notebook;
- Appropriate sample containers (pre-preserved with methanol, as required); and
- Appropriate transport containers (coolers) with water ice and appropriate labeling, packing, and shipping materials.

III. Field Sampling Procedures

VPH

Option 1 - Field Preservation

Step 1 - Order the appropriate number of pre-preserved sample containers (15 mL of methanol per sample vial) from the laboratory. Instruct the laboratory to supply the sample container (with preservative) tare weights with the bottle order.

Step 2 - Prior to sample collection, weigh each sample container and compare the weight to the laboratory supplied tare weight to ensure that loss of preservative has not occurred. **Do not use sample containers that do not agree within ∀ 0.2 grams of the laboratory tare weight.**

Step 3 - Soil samples must be collected in a manner that minimizes sample handling and agitation. This is accomplished by using a 30-mL plastic syringe with the end sliced off. The syringe is pushed into the soil sample collected via split-spoon methods until full.

Step 4 - Extrude approximately 15 grams of soil into the pre-preserved sample vial.

Step 5 - Wipe the threads of the sample vial with a handiwipe or lint-free paper towel, to ensure an adequate seal.

Step 6 - Weigh the sample container and record the weight on the chain-of-custody form. Add a note on the chain-of-custody form to inform the laboratory to contact the project QA/QC Manager if the laboratory measured sample weight does not agree within ∀ 0.2 grams of the weight recorded on the chain-of-custody form.

Step 7 - Collect additional soil in a 4-oz (120 mL) jar for percent moisture determination.

Step 8 - Place sample containers in a transportation cooler on ice immediately after collection. Package and label the sample containers following the procedures specified in Appendices L and M. **Do not add tape, custody seals, or any other material to the pre-preserved sample containers that will alter the weight of the container.**

Option 2A - EnCore®Sampler

Step 1 - Place EnCore® sample container into the EnCore® T-handle.

Step 2 - Collect soil sample by pressing the EnCore® sample container into the soil to be collected.

Step 3 - Wipe the outside of the EnCore® sample container with a handiwipe or lint-free paper towel to ensure an adequate seal.

Step 4 - Using the T-handle, cap and lock the EnCore® Sampler for shipment.

Step 5 - Collect additional soil in a 4-oz (120 mL) jar for percent moisture determination.

Step 6 - Place sample containers in a transportation cooler on ice immediately after collection. Package and label the sample container following the procedures in Appendix L.

Option 2B - SoilCore® Sampler

Step 1 - Collect soil sample by pressing the SoilCore® sample container into the soil to be collected. A decontaminated stainless steel spatula may be used to assist this procedure.

Step 2 - Wipe the outside of the SoilCore® sample container with a handiwipe or lint-free paper towel to ensure an adequate seal.

Step 3 - Cap the filled end of the SoilCore® sample container.

Step 4 - Fill the opposite side of the SoilCore® sample container by pushing the empty side of the SoilCore® into the soil to be collected. A decontaminated stainless steel spatula may be used to assist this procedure.

Step 5 - Wipe the outside of the SoilCore® sample container with a handiwipe or lint-free paper towel to ensure an adequate seal.

Step 6 - Cap the filled end of the SoilCore® sample container.

Step 7 - Place the locking strap around both endcaps and pull the strap tight to secure endcaps. Check the position of endcaps after tightening to verify that the SoilCore® container is properly sealed.

Step 8 - Collect additional soil in a 4-oz (120 mL) jar for percent moisture determination.

EPH

Step 1 - Soil samples are collected directly from the split-spoon using a stainless steel, decontaminated, spatula.

Step 2 - Samples are collected in 4-oz (120 mL) wide-mouth glass jars with Teflon-lined screw caps.

Step 3 - Wipe the threads of the sample jar with a handiwipe or lint-free paper towel, to ensure an adequate seal.

Step 4 - Place sample containers in a transportation cooler on ice immediately after collection. Package and label the sample containers following the procedures specified in Appendix L.

IV. Field Cleaning Procedures

Cleaning of EPH/VPH sampling equipment (e.g., stainless-steel sampling tools) is to follow the procedures presented in Appendix W. The sampling equipment is to be cleaned prior to the start of sampling activities, between samples and following the completion of sampling activities.

V. Disposal Methods

Rinse water, PPE, and other residuals generated during the equipment cleaning procedures will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its on-going disposal practices.

Appendix C

Soil Boring Installation and Soil Sampling Procedures

Soil Boring Installation and Soil Sampling Procedures

I. Introduction

This Standard Operating Procedure (SOP) describes the field sampling procedures to install soil borings and to collect soil samples (incorporating as appropriate, the soil sampling procedures for volatile organic compound (VOC) or extractable petroleum hydrocarbon/volatile petroleum hydrocarbon (EPH/VPH) analyses described in Appendices A and B, respectively). Soil samples may be collected through a variety of mechanisms, typically the hollow-stem auger drilling method, the driven casing drilling method, or a direct push technique. In situations where physical site features limit the use of drill rigs, soil borings will be completed with hand-driven equipment or a portable power auger depending on the required depth and subsurface material. A detailed discussion of the selection of drilling methods is presented in Appendix Y. Samples of subsurface material encountered during the drilling of soil borings will typically be collected continuously to the required depth of the boring, or as directed by the supervising geologist or technician, using the methods described in Section III below.

Personnel will also be responsible for documenting drilling events in the field log notebook. Only qualified personnel (e.g., holds degree in engineering, geology, or related science, and/or has at least two years of relevant experience) will provide descriptions of soil samples. The drilling contractor will be responsible for obtaining accurate and representative samples, informing the supervising geologist of changes in drilling pressure and loss of circulation, and keeping a separate general log of soils encountered, including blow counts (i.e., the number of blows from a soil sampling drive weight [140 pounds] required to drive the split-spoon sampler in 6-inch increments), if applicable.

II. Equipment and Materials

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

- Health and safety equipment (as required in the Site Health and Safety Plan [HASP]);
- Cleaning equipment (as required in Appendix W);
- All drilling equipment required by the American Society of Testing and Materials (ASTM) document D1586, entitled *Standard Method for Penetration Test and Split-Barrel Sampling of Soils* (Annual Book of ASTM Standards, Volume 04.08), as applicable (see Section III);
- Appropriate sample containers and forms;
- Coolers with ice or "blue ice;"

- Photoionization detector (PID); and
- Field notebook.

III. Soil Boring Installation

General procedures for the installation of soil borings using a variety of drilling methodologies are presented in Appendix Y. Additional details related to the primary drilling methods to be utilized (i.e., hollow-stem augers, driven casing, and direct-push methodologies) are provided below.

When hollow-stem augers or driven casing methodologies are employed, soil cores will be collected using standard 2-inch by 2-foot split-spoons driven by a 140-pound hammer or standard Shelby tubes, unless otherwise specified in the project-specific work plan. The split-spoons or Shelby tubes will be advanced to the depth specified in the project-specific work plan. Additional information regarding potential methods for collecting such cores may be found in ASTM Standard D1586 entitled "*Standard Method for Penetration Test and Split-barrel Sampling of Soils"* and ASTM Standard D6282-98 entitled "*Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations*," unless an alternate approach is specified in the project-specific work plan. Soil samples will be collected following the procedures described in the following sections, and those presented in Appendices A and B for VOCs and VPH/EPH, respectively. In addition, all drilling subcontractors will comply with the Commonwealth of Massachusetts requirement for licensing of monitoring well drillers.

Direct-push drilling methods also may be used to collect soil cores. Examples of this technique include the Diedrich ESP vibratory probe system or AMS Power Probe™ dual tube system. Environmental probe systems typically use a hydraulically operated percussion hammer. Depending on the equipment used, the hammer delivers 140 to 350 foot pounds of energy with each blow. The hammer, operated at 1,200 blows per minute, provides the force needed to penetrate very stiff to medium-dense soil formations. The hammer simultaneously advances and outer steel casing which contains a disposable plastic liner that is utilized to collect soil samples. Soil samples will be collected following the procedures described in the following sections, and those presented in Appendices A and B for VOCs and VPH/EPH, respectively.

At locations where the soil sampler cannot be advanced to the total depth specified in the project work plan due to subsurface refusal, a minimum of three attempts will be made to advance the boring to the total depth at nearby locations. Similarly, if soil sample recovery is less than 50% for the target analytical sampling interval specified in the project-specific work plan, a minimum of three attempts will be made to collect additional soil from the same sampling interval. However, this additional sampling need not be conducted at areas where GE and EPA field representatives agree that the nature of the subsurface materials are not likely to allow proper sample recovery (e.g., coarse gravel, loose fine sands, concrete rubble, fill, etc.).

The proper starting depth of all surficial soil and surficial soil boring samples will be dependent on location and surface cover. The initial soil sampling interval will generally start at the soil interface, not at the top of vegetation, gravel, and pavement or building floor. For example, if soil samples are to be collected at a location consisting of a 6-inch thick gravel lot, the 0- to 1-foot soil sample will be collected from the first foot of soil beginning just below the base of the gravel rather than either collecting 6 inches of gravel along with the underlying soil, or discarding the gravel, but only collecting soil from 6 inches to 1 foot below ground surface (bgs). In some cases, the starting point of soil sampling will be dependent on future design considerations for the area in question (i.e., 0- to 1-foot surface samples may not be necessary at areas where a soil cover will be installed, or conversely, the 0- to 1-foot surface samples may be collected at depths beginning 1 foot below thick concrete floors that will be subject to removal). Special circumstances such as these will be described in project-specific work plans.

IV. Subsurface Soil Sampling Procedures

Step 1 - As samples are collected, qualified personnel will describe each soil sample. Additional information regarding procedures to identify soil types may be found in ASTM Standard D2488-00, entitled "*Standard Practice for Description and Identification of Soils* (Visual-Manual Procedure)." Soil descriptions will be entered in the field notebook or on the Subsurface Log (Attachment C-1) for the following parameters:

- soil type;
- color;
- percent recovery;
- moisture content;
- texture;
- grain size and shape;
- consistency;
- blow counts, if collected; and
- miscellaneous observations.

A common soil sample description format should be utilized in the field notes, such as:

Color; primary constituent (underlined or capitalized); secondary constituent(s) designated by "and" (if approximately 50 % of the sample, should only be utilized if a second primary constituent is identified), "some" (if approximately 30% to 50% of the sample), "little" (if approximately 10% to 30% of the sample), and/or "trace" (if less than 10% of the sample);

description of consistency; moisture content; miscellaneous observations; and initial interpretations (capitalized in parentheses).

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

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

In addition, the boring logs must identify the specific depth of the fill/native soil interface (if present) and will provide a detailed description of any debris observed in the fill. This is particularly important for borings taken during investigations of fill properties outside the CD Site. Observations of staining, sheens, or other potential indicators of impacted soil should also be described in detail, including the starting and ending depths of such observations.

Each sampling interval will be recorded based on any spaces or gaps and the recovery of the core. For example, if a soil sampler is advanced from 4 to 8 feet bgs, but the soil recovery is only 2.5 feet, the log will indicate that the description only applies to 4 to 6.5 feet bgs (i.e., assume that lower portion of the soil sample was not recovered), unless reasons to infer otherwise are evident in the sample or adjacent samples. If soil sample recovery for the target analytical sampling interval is less than 50%, additional sampling attempts will be made, as specified in Section III of this Appendix.

Step 2 - Samples are placed in appropriate sample containers for PID field screening as specified in Appendix N. PID screening may be performed to identify potentially impacted zones, or, in some cases, to determine which samples will receive analysis for VOCs and/or other Appendix IX+3 constituents, as discussed below.

Step 3 - If the project-specific work plan requires the collection of one or more samples from a given boring for analysis of Appendix IX+3 constituents or one or more groups thereof (e.g., VOCs), such sample(s) will be identified and collected. The selection of such sample(s) will be based on the specifications of the project-specific work plan regarding which sample(s) should be analyzed for such constituents, given the objectives of the investigation. The following procedures will be utilized in this selection, where applicable:

- In many cases, the project-specific work plan will specify the target depth increment to be analyzed for Appendix IX+3 constituents for the purpose of obtaining a spatially distributed range of depth increments. However, it may also provide that some modifications to the specified location/depth may be made in the field considering PID readings or visual observations (e.g., evidence of staining, presence of oil, etc.). In such cases, the following procedures will be followed:

- If no other samples exhibit PID readings or visual evidence of contamination significantly greater that the depth increment specified in the work plan, the work plan-specified increment will be selected.

- If another sample exhibits a significantly higher PID reading or more visual evidence of contamination (e.g., staining, presence of oil, odor, etc.) than the depth increment specified in the work plan, then such other sample will be selected for Appendix IX+3 analysis, unless similar soil samples from comparable depth increments have been or will be analyzed for Appendix IX+3 constituents at nearby sampling locations. In the event that such a modification is made to the work plan-specified depth increment at a given location, then a corresponding modification should be made at another sampling location within the investigation area (nearby if possible) to maintain the depth and spatial distribution of Appendix IX+3 samples specified in the work plan.

- For analytical depth increments which encompass multiple soil sample intervals, the sample which exhibits the highest PID reading within the selected depth increment will be analyzed for VOCs, while the entire depth increment will be utilized for analysis of the remaining Appendix IX constituents (see Section V below).

- If separate, distinct zones of elevated head space readings are encountered the Project Manager should be contacted to discuss sampling options. In these cases, typically one interval would be selected for Appendix IX+3 analysis according to the criteria discussed above, and an additional sample may be taken from the other zone for VOC analysis, unless similar soil samples from comparable depth increments have been or will be analyzed for Appendix IX+3 constituents at nearby sampling locations.

- When the project-specific work plan specifies that sample selection for VOC analysis is to be based on PID screening, a sample which exhibits a reading of 10 PID units or higher will be collected as specified in Appendix A and analyzed for VOCs by EPA Method 8260B (unless otherwise specified in the work plan). If such readings are encountered at multiple soil sample/PID screening intervals within a boring, only the sample which exhibits the highest PID reading within the boring will be analyzed for VOCs, unless separate and distinct zones of elevated PID readings are identified. If separate, distinct zones of elevated head space readings are encountered, the Project Manager should be contacted to discuss sampling options.

- When the project-specific work plan specifies that the selection of a sample for Appendix IX+3 analysis is to be based on PID screening, the following protocol will be followed unless otherwise specified in the work plan: The sample which exhibits the highest PID reading for each boring will be analyzed for Appendix IX+3 constituents. If no samples exhibit PID readings significantly higher than background, then a sample will be selected based on visual inspection (i.e., staining, presence of oil, odor, etc). If there are no visual indications of the presence of hazardous materials, then the sample located at the water table interface will be selected for Appendix IX+3 analyses.

- When using PID screening to determine the sample to be analyzed for VOCs or Appendix IX+3 constituents, such samples may be collected using either of the two following procedures:

  Option 1 - The samples for potential VOC/Appendix IX+3 analyses may be collected from each depth interval at the same time that the PID screening samples are collected. After completion of the soil boring, the selected sample will be sent to the laboratory for analysis and the remaining samples will be discarded.

  Option 2 - Alternatively, the complete soil boring can be screened to determine the interval with the highest PID reading prior to the collection of the VOC/Appendix IX+3 sample. After screening is completed and the VOC/Appendix IX+3 sample interval has been determined, another sample can be collected from the appropriate depth interval for VOC/Appendix IX+3 analysis by installation of another soil boring adjacent (slightly off-set) to the original boring.

Step 4 - Sample containers will be labeled, stored on site, and transported to the appropriate testing laboratory. Label all sample containers with the following:

- site;
- project number;
- boring number;
- sample interval;
- date;
- time of sample collection; and
- initials of sampling personnel.

Step 5 - The samples will be handled, packed, and shipped in accordance with the procedures set forth in Appendix L.

V. Soil Sample Compositing

In certain instances, representative soil samples from several depth increments may be composited into a single sample for subsequent analyses. This approach will be utilized for a number of future soil investigations related to the implementation of the CD -- notably, sampling several potential depth increments in the upper 15 feet bgs (e.g., 1- to 6-foot and 6- to 15-foot, or 1- to 3-foot, 3- to 6-foot, 6- to 10-foot, and 10- to 15-foot depth increments, depending on the project location and work plan requirements). In such instances, the following protocols will be used to support the performance of composite sample collection and analysis:

Step 1 - As soil samples from individual sample depth increments (e.g., 2-foot depth increments) are collected, a representative sample will be placed into a glass sample jar for subsequent PID screening.

Step 2 - The remainder of the soil sample will be placed into a clean, stainless steel bowl for subsequent compositing and homogenizing with other samples from the specified composite depth interval. Using this process, the appropriate composite samples will be obtained -- each representing the specified analytical sample depth increment. Each of these composite samples will be analyzed for PCBs.

Step 3 - In addition to analysis for PCBs, a sample from one or more of the composite samples may be submitted for Appendix IX+3 analyses, excluding VOCs. The composite sample to be submitted will be selected as described above in Section IV, Step 3.

Step 4 - Prior to compositing the discrete sample depth interval within the composite sample interval with the highest PID reading will be sampled for Appendix IX VOC analysis, if a VOC sample is required from that interval. Duplicate VOC samples should be collected as close as practical to the original VOC sample depth.

VI. Surficial Soil Sampling

Surficial soil samples will be collected using a hand-driven split-spoon sampler, a stainless steel bucket auger, or a spade and scoop as determined by the field team depending on the subsurface material. Samples of material encountered during this operation will be collected in 6-inch or 12-inch increments as indicated in the respective work plan.

VII. Surficial Soil Sampling Materials

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

- Health and safety equipment (as required by the HASP);
- Cleaning equipment (as required in Appendix W);
- Teflon® sheeting or stainless-steel tray;
- Appropriate sample containers and forms;
- Coolers with ice or “blue ice;”
- Hand operated soil sampling kit (split-spoon);
- Stainless-steel bucket auger;
- Brass push rod;
- Spatula or knife;
- Hand spade;
- Stainless steel scoop;
- Stainless steel spoon;
- 6-foot rule; and
- Field notebook.

VIII. Surficial Soil Sampling Procedures

The following procedures will be employed to collect surficial soil samples:

Step 1 - If the sample location is a grassed area or an area that exhibits overlying material (i.e., gravel, leaves, roots), the sod or overlying material should be removed and the underlying soil should be collected. The sod refers to the grass and dense root matter below the grass, including the soil within the dense root matter. Replace the sod following sample collection.

Step 2 - Secure a representative sample from the appropriate depth and place into a suitable sample jar.

Step 3 - If PID screening is being performed for selection of Appendix IX+3 samples, obtain a split sample for screening with a PID for VOCs, using the procedures set forth in Appendix N, and take a PID reading as soon as possible after sample preparation.

Step 4 - Label all sample containers with the following:

- site;
- project number;
- location number;
- depth of sample;
- date;
- time of sample collection; and
- initials of sampling personnel

Step 5 - Handle, pack, and ship the samples using the chain-of-custody procedures in accordance with Appendix L.

Step 6 - Record all appropriate information in the field notebook and on the proper forms.

IX. Duplicate Sample Collection

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

X. Survey

A field survey control program will be conducted using standard instrument survey techniques to document the boring, surficial soil, or floodplain sampling location and elevation. Generally, to accomplish this, a local control baseline will be set up. If specified in the work plan, this local baseline control can then be tied into the appropriate vertical and horizontal datum such as the National Geodetic Vertical Datum (NGVD) of 1929 and the State Plane Coordinate System. At a minimum, the elevation of floodplain soil samples will be determined using NGVD-1929.

XI. Field Cleaning Procedures

Cleaning of sampling equipment is to follow the procedures specified in Appendix W. The sampling equipment is to be cleaned prior to the start of sampling activities, between samples, and following the completion of sampling activities. In addition, tools utilized in the handling and opening of sampling equipment, such as wrenches for opening split-spoon samplers or knives for cutting direct-push sample liners, are to be cleaned with a non-phosphate soap and water prior to the start of sampling activities, between boreholes, and following the completion of sampling activities, at a minimum.

XII. Disposal Methods

Rinse water, PPE, and other residuals generated during the equipment cleaning procedures will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its ongoing disposal practices.

Attachment C-1

Subsurface Log

Date Start/Finish: / Drilling Company: Driller's Name: Drilling Method: Bit Size: Auger Size : Rig Type: Spoon Size:	Northing: Easting: Well Casing Elev.: ft. Corehole Depth: ft. Borehole Depth: ft. Ground Surface Elev.: ft. Descriptions by:	Well No. Client: Site:

DEPTH	ELEVATION	Sample Depth Sample Number	Sample/Int/Type	Blows/6 In.	N	Recovery (ft.)	PID (ppm) Headspace	Geotechnical Test	Geologic Column	Stratigraphic Description	Well Construction
gs elevation ft.										GROUND SURFACE	
5											
10											
15											

Remarks:

Water Levels		
Date / Time	Elevation	Depth

Appendix D

Groundwater Purging and Sampling Procedures for Monitoring Wells

Groundwater Purging and Sampling Procedures for Monitoring Wells

I. Introduction

Groundwater samples will be collected from monitoring wells to evaluate groundwater quality. The protocol presented in this Appendix describes the procedures to be used to purge monitoring wells and collect groundwater samples. This protocol has been developed in accordance with the EPA Region I *Low Stress (Low Flow) Purging and Sampling Procedures for the Collection of Groundwater Samples from Monitoring Wells* (Attachment D-1) for use in 1.5-inch diameter wells and greater. In addition, should GE desire to change to a markedly different sampling methodology, GE will submit a proposed standard operating procedure for the new methodology for EPA approval prior to implementing such a change, if the procedure is not already incorporated into the *Field Sampling Plan/Quality Assurance Project Plan* (FSP/QAPP). An alternate sampling procedure incorporating the usage of passive diffusion bags is presented in Appendix AA.

Both filtered and unfiltered groundwater samples may be collected using this low-flow sampling method. Filtered samples will be obtained either by using a 0.45-micron disposable filter in the field or by having the laboratory filter the samples prior to analysis. No wells will be sampled until well development has been performed in accordance with the procedures presented in Appendix S-1 or S-2, unless that well has been sampled or developed within the prior 1-year time period. Groundwater samples will not be collected within a 1-week time period following well development.

II. Materials

Specific to this activity, the following materials (or equivalent) shall be available:

- Site plan, well construction records, prior groundwater sampling records (if available);
- Sampling pump, capable of maintaining a minimum pumping rate of 0.1 L/min, which may consist of one or more of the following:
  - Submersible pump (e.g., Grundfos Redi-Flo 2);
  - Peristaltic pump (e.g., SERIES I or II Geopump™); and/or
  - Bladder pump (e.g., QED Sample Pro);
- Teflon® tubing or Teflon®-lined polyethylene tubing of an appropriate size for the pump being utilized (or alternate tubing that is shown through equipment blank sampling to not add contaminants to the sample and that is approved by EPA before the sampling event);

- Silastic tubing for use around the rotor head of a peristaltic pump (to minimize gaseous diffusion, the inside diameter of the tubing used at the pump rotor head must be the same as the inside diameter of tubing placed in the well);
- Water level probe (e.g., Solinst Model 101);
- Water quality (temperature/pH/specific conductivity/ORP/turbidity/dissolved oxygen) meter and flow-through measurement cell, which may consist of several brands including:
  - YSI 556 or Professional Plus Multi-Parameter Instrument;
  - Hydrolab Series 3 or Series 4a Multiprobe and Display; and/or
  - Horiba U-22 or U-50 Water Quality Monitoring System;
- Supplemental turbidity meter (e.g., Horiba U-22 or Hach 2100P);[1]
- Transparent, small-volume flow-through-cells (250 mL or less preferred);
- Appropriate water sample containers (supplied by the laboratory);
- Appropriate blanks (trip blank supplied by the laboratory);
- 0.45-micron disposable filters;
- Graduated cylinder (size according to flow rate) and stopwatch;
- Large graduated bucket to be used to record total water purged; and
- Sampling shade for tubing in the summer.
- Sample container labels (Attachment D-3)

Note that in the future, GE may acquire different makes/models of some of this equipment if the listed makes/models are no longer available, or as a result of general upgrades or additional equipment acquisitions. In the event that GE uses a different make/model of the equipment listed, GE will utilize an equivalent type of equipment (e.g., pumps, flow-through analytical cells), and will note the specific make/model of the equipment utilized during a sampling event on the Groundwater Sampling Log provided as Attachment D-2.

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[1] Turbidity measurements collected with multi-parameter meters have been shown to sometimes be unreliable due to fouling of the optic lens of the turbidity meter within the flow-through cell. A supplemental turbidity meter will be utilized to verify turbidity data during purging if such fouling is suspected. Note that industry improvements may eliminate the need for these supplemental measurements in the future.

The maintenance requirements for the above equipment generally involve decontamination or periodic cleaning, battery charging, and proper storage, as specified by the manufacturer. For operational difficulties, the equipment will be serviced by a qualified technician.

III. Procedure

Groundwater will be purged and sampled from the wells using an appropriate pump. Peristaltic pumps may be utilized if the depth to water is within the sampling range of a peristaltic pump (approximately 25 feet). Otherwise, submersible pumps or bladder pumps will be utilized provided the well is constructed with a casing diameter greater than or equal to 2 inches (the minimum well diameter capable of accommodating such pumps). For smaller diameter wells where the depth to water is below the sampling range of a peristaltic pump, alternative sampling methods (i.e., bailing) will be utilized to purge and sample the groundwater. Purge water will be collected and containerized.

1. Perform calibration of field instruments according to procedures in Attachment O-1 of Appendix O.

2. Measure initial depth to groundwater and use recent historical measurements for the depth to bottom in order to calculate the approximate water column and volume in the well prior to placement of pumps.[2] If a submersible or bladder pump is being utilized, slowly lower pump, safety cable, tubing, and electrical lines into the well to a depth corresponding to the appropriate intake depth. If a peristaltic pump is being utilized, slowly lower the sampling tubing into the well to a depth corresponding to appropriate intake depth. Generally, the pump intake will be set at the approximate center of the saturated screen section of the monitoring well. Exceptions to this placement should be made in the following situations:

   - For wells with screens greater than 10 feet in length, the pump intake should be no greater than five feet below the top of the saturated screen depth, unless otherwise specified in approved work plans or modified due to observed field conditions (e.g., slow recharging wells or wells demonstrating excessive drawdown at minimal pumping rates).

   - For wells that are screened across soil types of dramatically varying permeabilities (i.e., wells screened across the intersection between unconsolidated granular deposits and glacial till), the pump intake should be aligned with the unit of greater permeability, which is presumably supplying most of the groundwater to the well.

   - For slow-recharging wells, the pump intake may be lowered to a level necessary to obtain groundwater samples, or to purge the well dry prior to recharge and sample collection.

   The pump intake or sampling tube should be kept at least 2 feet above the bottom of the well to prevent mobilization of any sediment present in the bottom of the well.

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[2] As described in Step 10 below, a final depth to bottom should be determined following sample collection.

3. Measure the water level again with the pump in the well before starting the pump. Start pumping the well at 200 to 500 milliliters (ml) per minute. The pump rate should be adjusted to cause little or no water level drawdown in the well (less than 0.3 feet below the initial static depth to water measurement, if possible) and the water level should stabilize. As stated in the Attachment D-1, stabilization of indicator field parameters (i.e., turbidity, temperature, specific conductance, pH, etc.) can be used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 nephelometric turbidity units (NTU) and stable drawdowns of less than 0.3 feet, while desirable, are not mandatory. The water level should be monitored every 3 to 5 minutes (or as appropriate) during pumping if the well diameter is of sufficient size to allow such monitoring. Care should be taken not to break pump suction or cause entrainment of air in the sample. Record pumping rate adjustments and depths to water. If necessary, pumping rates should be reduced to the minimum capabilities of the pump to avoid pumping the well dry and/or to ensure stabilization of indicator parameters. A steady flow rate should be maintained to the extent practicable. Groundwater sampling records from previous sampling events (if available) should be examined to provide an estimate of the optimum pumping rate and anticipated drawdown for the well in order to more efficiently reach a stabilized pumping condition.

   If the recharge rate of the well is very low, alternative purging techniques should be utilized, which will vary based on the well construction and screen position. For wells screened across the water table, if the water level drops more than 0.3 feet below the initial depth to water, the pump rate can be decreased to the minimum flow rate of 100 mL/min, or as close to that level as possible, until the level stabilizes and monitoring for stabilization of field indicator parameters can commence. If the water level does not stabilize in a reasonable amount of time (e.g., one hour), the flow rate may be temporarily increased to 500 mL min for 5 minutes to more efficiently reduce the water level to the point of stabilization. Following the 5-minute interval with an increased rate, the flow should be reduced back to the minimum rate to check whether the well will begin to recharge while pumping continues. If so, purging should continue at the minimum flow rate until the water level stabilizes and all other field parameters have stabilized. If the level of stabilization is near the base of the well, the use of passive diffusion bags for VOC sample collection (Appendix AA) should be considered.

   For wells screened across the water table, if the water level cannot be stabilized using the lowest possible flow before the well is completely dewatered, purging should be terminated and the well allowed to recharge to a level which provides sufficient volume for the planned analyses before sampling. For wells screened entirely below the water table, the well should be pumped until a stabilized level (which may be below the maximum displacement goal of 0.3 feet) can be maintained and monitoring for stabilization of field indicator parameters can commence. If a stabilization level cannot be maintained with the lowest achievable flow rate, the well should be pumped until the drawdown is at a level slightly higher than the bentonite seal above the well screen. Sampling should commence after one well volume has been removed and the well has recovered sufficiently to permit collection of samples.

4. During purging, monitor the field indicator parameters (e.g., turbidity, temperature, specific conductance, pH, dissolved oxygen, oxidation reduction potential [ORP]) every three to five minutes (or as appropriate). Field indicator parameters will be measured using a flow-through analytical cell, although turbidity data will be collected prior to the flow-through cell using a three-way valve confirmed by a separate turbidity meter. Record field indicator parameters on the Groundwater Sampling Log (Attachment D-2). The well is considered stabilized and ready for sample collection when turbidity values remain within 10% (or within 1 NTU if the turbidity reading is less than 10 NTU), the dissolved oxygen level remains within 10% (or within 0.1 mg/l if the dissolved oxygen level is less than 1.0 mg/l), the specific conductance and temperature values remain within 3%, ORP remains within 10 millivolts, and pH remains within 0.1 units for three consecutive readings collected at three to five minute intervals. If the field indicator parameters do not stabilize within one hour of the start of purging, but the groundwater turbidity is below the goal of 50 NTU and the values for all other parameters are within 10%, the well can be sampled. If the parameters have stabilized, but the turbidity is not in the range of the 50 NTU goal, the pump flow rate should be decreased to a minimum rate of 100 mL/min (or as close as possible) to reduce turbidity levels as low as possible. If the well has been pumped dry, sampling can commence once the volume in the well has recovered sufficiently to permit collection of samples. During extreme weather conditions, stabilization of field indicator parameters may be difficult to obtain. Modifications to the sampling procedures to alleviate these conditions (e.g., measuring the water temperature in the well adjacent to the pump intake) will be documented in the field notes. If other field conditions exist which preclude stabilization of certain parameters, an explanation of why the parameters did not stabilize will also be documented in the field logbook.

5. Complete the sample label (Attachment D-3) according to procedures in Appendix L and cover the label with clear packing tape to secure the label onto the container.

6. After the indicator field parameters have stabilized (or other above-mentioned criteria for sampling have been achieved), collect groundwater sample by diverting flow out of the discharge tubing into the appropriate labeled sample container. If a flow-through analytical cell is being used to measure field parameters, the flow-through cell should be disconnected after stabilization of the field indicator parameters and prior to groundwater sample collection. Under no circumstances should analytical samples be collected from the discharge of the flow-through cell. When the container is full, tightly screw on the cap. Samples should be collected in the following order: VOCs; TOC; SVOCs; metals and cyanide; others.

7. If sampling for total and filtered metals and/or PCBs, filtered and unfiltered samples will be collected. If the sample cannot be transferred to the laboratory for filtering, sample filtration for the filtered sample will be performed in the field utilizing an in-line filtration system. Install an in-line, disposable 0.45-micron particle filter on the discharge tubing after the appropriate unfiltered groundwater sample has been collected. Continue to run the pump until an initial volume of “flush” water has been run through the filter in accordance with the manufacturer’s directions (generally 100 to 300 ml). Collect filtered groundwater sample by diverting flow out of the filter into the appropriate labeled sample container. When the container is full, tightly screw on the cap.

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

9. Record on the Groundwater Sampling Log (Attachment D-2) or bound field book the time sampling procedures were completed, any pertinent observations of the sample (e.g., physical appearance, the presence of, or lack of, odors, sheens, effervescence), and the values of the stabilized field indicator parameters, as measured during the final reading during purging.

10. Remove pump and tubing from well, secure well, properly dispose of PPE and disposable equipment (see Section VI). Collect final depth to bottom measurement. Note: If water level probe does not collect measurements from the bottom of the probe, collected measurement should be adjusted appropriately.

11. If tubing is to be dedicated to a well, the tube shall be stored within the riser pipe. The open end of the tube shall not be allowed to contact the interior of the steel protective casing or well cap. The tube should be folded to a length that will allow the well to be capped and also facilitate retrieval of the tubing during later sampling events. A length of rope or string should be used to tie the tubing to the well cap.

12. Complete the procedures for packaging, shipping, and handling with associated chain-of-custody (Appendix L).

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

14. At end of day, perform calibration check of field instruments according to procedures in Appendix O.

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

1. Don appropriate personal protective equipment (PPE), as required by the site Health and Safety Plan (HASP).

2. Place plastic sheeting around the well.

3. Clean the sampling equipment with the procedures in Appendix W.

4. Open the well cover while standing upwind of the well. Remove well cap and place on the plastic sheeting. Insert photoionization detector (PID) probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in the field log. If the well headspace reading is less than 5 parts per million (ppm), proceed. If the headspace reading is greater than 5 ppm, screen the air within the breathing zone. If the breathing zone reading is less than 5 ppm, proceed. If the PID reading in the breathing zone is above 5 ppm, move upwind from well for

5 minutes to allow the volatiles to dissipate, and then repeat the breathing zone test. If the reading is still above 5 ppm, don appropriate respiratory protection in accordance with the requirements of the HASP. Record all PID readings. For wells which are part of the regular weekly or monthly monitoring program and prior PID measurements have not resulted in a breathing zone reading above 5 ppm, PID measurements will be taken semi-annually.

5. Measure the depth to water and determine depth of well through examination of drilling log data or by direct measurement. Calculate the volume of water in the well (in gallons) by using the length of the water column (in feet), multiplying by 0.163 for a 2-inch well or by 0.653 for a 4-inch well. For other well diameters, use the following formula:

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

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

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

8. Monitor the field indicator parameters (e.g., turbidity, temperature, specific conductance, pH, etc.). Field indicator parameters will be measured using a clean container such as a glass beaker or sampling cups provided with the instrument. A flow-through analytical cell should be utilized if a pump is utilized for purging and sampling to enable collection of dissolved oxygen and ORP data. Record field indicator parameters on the Groundwater Sampling Log (Attachment D-2).

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

10. If field parameters have not stabilized, five well volumes may be removed with measurement of initial and final groundwater parameters, at a minimum. If parameters have stabilized, proceed to step #12.

11. If the recharge rate of the well is very low, wells screened across the water table may be bailed dry and sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection of samples. For wells screened entirely below the water table, the well should be only be bailed down to a level slightly higher than the bentonite seal above the well screen. The well should not be bailed completely dry in order to maintain the integrity of the seal. Sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection of samples. Field indicator parameters will be recorded again at the time of sample collection. For extremely low recharging wells, where multiple sampling attempts over several days are necessary to collect the required sample volume, field indicator parameters will be recorded each day that samples are collected, provided the well contains sufficient volume to conduct those measurements.

12. Following purging, allow water level in well to recharge to a sufficient level to permit collection of samples.

13. Complete the sample label according to procedures in Appendix L and cover the label with clear packing tape to secure the label onto the container.

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

15. The sample collection order (as appropriate) will be as follows:

    1) VOCs;
    2) TOC;
    3) SVOCs;
    4) Metals and cyanide; and
    5) Others.

16. When sampling for volatiles, water samples will be collected directly from the bailer into 40 mL vials with Teflon®-lined septa.

17. For other analytical samples, remove the cap from the large glass mixing container and slowly empty the bailer into the large glass mixing container. The sample for dissolved metals and/or filtered PCBs should either be placed directly from the bailer into a pressure filter apparatus or pumped directly from the bailer with a peristaltic pump, through an in-line filter, into the pre-preserved sample bottle.

18. Continue collecting sample until the mixing container contains a sufficient volume for all laboratory samples.

19. Mix the entire sample volume with the Teflon® stirring rod and transfer the appropriate volume into the laboratory jar(s). Secure the sample jar cap(s) tightly.

20. If sampling for total and filtered metals and/or PCBs, filtered and unfiltered samples will be collected. If the sample cannot be transferred to the laboratory for filtering, sample filtration for the filtered sample will be performed in the field utilizing a peristaltic pump prior to preservation. Install new medical-grade silicone tubing in the pump head. Place new Teflon® tubing into the sample mixing container and attach to the intake side of pump tubing. Attach (clamp) a new 0.45-micron filter (note the filter flow direction). Turn the pump on and dispense the filtered liquid directly into the laboratory sample bottles.

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

22. After sample containers have been filled, remove an additional volume of groundwater. Measure the pH, temperature, turbidity, and conductivity. Record on the Groundwater Sampling Log (Attachment D-2) or bound field book the time sampling procedures were completed, any pertinent observations of the sample (e.g., physical appearance, the presence of, or lack of, odors, sheens, etc.), and the values of the field indicator parameters.

23. Remove bailer from well, secure well, and properly dispose of PPE and disposable equipment (see Section VI).

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

25. Complete the procedures for packaging, shipping, and handling with associated chain-of-custody (Appendix L).

IV. Field Quality Control

In addition to the quality control samples to be collected in accordance with Table 4 of the FSP/QAPP, the following quality control procedures should be observed in the field:

- Samples should be collected from monitoring wells in order of increasing concentration (i.e. from wells with the lowest concentrations to those with the highest concentrations), to the extent known.

- Equipment blanks should include the pump and tubing (if using disposable tubing) or the pump only (if using tubing dedicated to each well).

- Equipment blanks should be collected after wells with higher concentrations (if known) have been sampled.

- All monitoring instrumentation shall be operated in accordance with manufacturer's instructions and the calibration procedures presented in Attachment O-1 of Appendix O. Instruments should be calibrated at the beginning of each day and the calibration should be verified at the end of each day.

V. Equipment Cleaning

All groundwater sampling equipment should be cleaned prior to use in the first well and after each subsequent well using procedures presented in Appendix W.

VI. Material Disposal

Materials generated during groundwater sampling activities, including disposable equipment, will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its ongoing disposal practices.

Attachment D-1

Low Stress (Low Flow) Purging and Sampling Procedures for the Collection of Groundwater Samples from Monitoring Wells (EPA Region 1 SOP, revised January 2010)

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I

LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUNDWATER SAMPLES FROM MONITORING WELLS

Quality Assurance Unit
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Prepared by: ____________________ 1/19/10
(Charles Porfert, Quality Assurance Unit) Date

Approved by: ____________________ 1-19-10
(Gerard Sotolongo, Quality Assurance Unit) Date

Revision Page

Date	Rev #	Summary of changes	Sections
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USE OF TERMS

Equipment blank: The equipment blank shall include the pump and the pump's tubing. If tubing is dedicated to the well, the equipment blank needs only to include the pump in subsequent sampling rounds. If the pump and tubing are dedicated to the well, the equipment blank is collected prior to its placement in the well. If the pump and tubing will be used to sample multiple wells, the equipment blank is normally collected after sampling from contaminated wells and not after background wells.

Field duplicates: Field duplicates are collected to determine precision of the sampling procedure. For this procedure, collect duplicate for each analyte group in consecutive order (VOC original, VOC duplicate, SVOC original, SVOC duplicate, etc.).

Indicator field parameters: This SOP uses field measurements of turbidity, dissolved oxygen, specific conductance, temperature, pH, and oxidation/reduction potential (ORP) as indicators of when purging operations are sufficient and sample collection may begin.

Matrix Spike/Matrix Spike Duplicates: Used by the laboratory in its quality assurance program. Consult the laboratory for the sample volume to be collected.

Poteniometric Surface: The level to which water rises in a tightly cased well constructed in a confined aquifer. In an unconfined aquifer, the potentiometric surface is the water table.

QAPP: Quality Assurance Project Plan

SAP: Sampling and Analysis Plan

SOP: Standard operating procedure

Stabilization: A condition that is achieved when all indicator field parameter measurements are sufficiently stable (as described in the "Monitoring Indicator Field Parameters" section) to allow sample collection to begin.

Temperature blank: A temperature blank is added to each sample cooler. The blank is measured upon receipt at the laboratory to assess whether the samples were properly cooled during transit.

Trip blank (VOCs): Trip blank is a sample of analyte-free water taken to the sampling site and returned to the laboratory. The trip blanks (one pair) are added to each sample cooler that contains VOC samples.

SCOPE & APPLICATION

The goal of this groundwater sampling procedure is to collect water samples that reflect the total mobile organic and inorganic loads (dissolved and colloidal sized fractions) transported through the subsurface under ambient flow conditions, with minimal physical and chemical alterations from sampling operations. This standard operating procedure (SOP) for collecting groundwater samples will help ensure that the project's data quality objectives (DQOs) are met under certain low-flow conditions.

The SOP emphasizes the need to minimize hydraulic stress at the well-aquifer interface by maintaining low water-level drawdowns, and by using low pumping rates during purging and sampling operations. Indicator field parameters (e.g., dissolved oxygen, pH, etc.) are monitored during purging in order to determine when sample collection may begin. Samples properly collected using this SOP are suitable for analysis of groundwater contaminants (volatile and semi-volatile organic analytes, dissolved gases, pesticides, PCBs, metals and other inorganics), or naturally occurring analytes. This SOP is based on Puls, and Barcelona (1996).

This procedure is designed for monitoring wells with an inside diameter (1.5-inches or greater) that can accommodate a positive lift pump with a screen length or open interval ten feet or less and with a water level above the top of the screen or open interval (Hereafter, the "screen or open interval" will be referred to only as "screen interval"). This SOP is not applicable to other well-sampling conditions.

While the use of dedicated sampling equipment is not mandatory, dedicated pumps and tubing can reduce sampling costs significantly by streamlining sampling activities and thereby reducing the overall field costs.

The goal of this procedure is to emphasize the need for consistency in deploying and operating equipment while purging and sampling monitoring wells during each sampling event. This will help to minimize sampling variability.

This procedure describes a general framework for groundwater sampling. Other site specific information (hydrogeological context, conceptual site model (CSM), DQOs, etc.) coupled with systematic planning must be added to the procedure in order to develop an appropriate site specific SAP/QAPP. In addition, the site specific SAP/QAPP must identify the specific equipment that will be used to collect the groundwater samples.

This procedure does not address the collection of water or free product samples from wells containing free phase LNAPLs and/or DNAPLs (light or dense non-aqueous phase

liquids). For this type of situation, the reader may wish to check: Cohen, and Mercer (1993) or other pertinent documents.

This SOP is to be used when collecting groundwater samples from monitoring wells at all Superfund, Federal Facility and RCRA sites in Region 1 under the conditions described herein. Request for modification of this SOP, in order to better address specific situations at individual wells, must include adequate technical justification for proposed changes. All changes and modifications must be approved and included in a revised SAP/QAPP before implementation in field.

BACKGROUND FOR IMPLEMENTATION

It is expected that the monitoring well screen has been properly located (both laterally and vertically) to intercept existing contaminant plume(s) or along flow paths of potential contaminant migration. Problems with inappropriate monitoring well placement or faulty/improper well installation cannot be overcome by even the best water sampling procedures. This SOP presumes that the analytes of interest are moving (or will potentially move) primarily through the more permeable zones intercepted by the screen interval.

Proper well construction, development, and operation and maintenance cannot be overemphasized. The use of installation techniques that are appropriate to the hydrogeologic setting of the site often prevent "problem well" situations from occurring. During well development, or redevelopment, tests should be conducted to determine the hydraulic characteristics of the monitoring well. The data can then be used to set the purging/sampling rate, and provide a baseline for evaluating changes in well performance and the potential need for well rehabilitation. Note: if this installation data or well history (construction and sampling) is not available or discoverable, for all wells to be sampled, efforts to build a sampling history should commence with the next sampling event.

The pump intake should be located within the screen interval and at a depth that will remain under water at all times. It is recommended that the intake depth and pumping rate remain the same for all sampling events. The mid-point or the lowest historical midpoint of the saturated screen length is often used as the location of the pump intake. For new wells, or for wells without pump intake depth information, the site's SAP/QAPP must provide clear reasons and instructions on how the pump intake depth(s) will be selected, and reason(s) for the depth(s) selected. If the depths to top and bottom of the well screen are not known, the SAP/QAPP will need to describe how the sampling depth will be determined and how the data can be used.

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU, and stable drawdowns of less than 0.3 feet, while desirable, are not mandatory. Sample collection

may still take place provided the indicator field parameter criteria in this procedure are met. If after 2 hours of purging indicator field parameters have not stabilized, one of three optional courses of action may be taken: a) continue purging until stabilization is achieved, b) discontinue purging, do not collect any samples, and record in log book that stabilization could not be achieved (documentation must describe attempts to achieve stabilization), c) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analytes, may reflect a sampling bias and therefore, the data may not meet the data quality objectives of the sampling event).

It is recommended that low-flow sampling be conducted when the air temperature is above 32°F (0°C). If the procedure is used below 32°F, special precautions will need to be taken to prevent the groundwater from freezing in the equipment. Because sampling during freezing temperatures may adversely impact the data quality objectives, the need for water sample collection during months when these conditions are likely to occur should be evaluated during site planning and special sampling measures may need to be developed. Ice formation in the flow-through-cell will cause the monitoring probes to act erratically. A transparent flow-through-cell needs to be used to observe if ice is forming in the cell. If ice starts to form on the other pieces of the sampling equipment, additional problems may occur.

HEALTH & SAFETY

When working on-site, comply with all applicable OSHA requirements and the site's health/safety procedures. All proper personal protection clothing and equipment are to be worn. Some samples may contain biological and chemical hazards. These samples should be handled with suitable protection to skin, eyes, etc.

CAUTIONS

The following cautions need to be considered when planning to collect groundwater samples when the below conditions occur.

If the groundwater degasses during purging of the monitoring well, dissolved gases and VOCs will be lost. When this happens, the groundwater data for dissolved gases (e.g., methane, ethene, ethane, dissolved oxygen, etc.) and VOCs will need to be qualified. Some conditions that can promote degassing are the use of a vacuum pump (e.g., peristaltic pumps), changes in aperture along the sampling tubing, and squeezing/pinching the pump's tubing which results in a pressure change.

When collecting the samples for dissolved gases and VOCs analyses, avoid aerating the groundwater in the pump's tubing. This can cause loss of the dissolved gases and VOCs in

the groundwater. Having the pump's tubing completely filled prior to sampling will avoid this problem when using a centrifugal pump or peristaltic pump.

Direct sun light and hot ambient air temperatures may cause the groundwater in the tubing and flow-through-cell to heat up. This may cause the groundwater to degas which will result in loss of VOCs and dissolved gases. When sampling under these conditions, the sampler will need to shade the equipment from the sunlight (e.g., umbrella, tent, etc.). If possible, sampling on hot days, or during the hottest time of the day, should be avoided. The tubing exiting the monitoring well should be kept as short as possible to avoid the sun light or ambient air from heating up the groundwater.

Thermal currents in the monitoring well may cause vertical mixing of water in the well bore. When the air temperature is colder than the groundwater temperature, it can cool the top of the water column. Colder water which is denser than warm water sinks to the bottom of the well and the warmer water at the bottom of the well rises, setting up a convention cell. "During low-flow sampling, the pumped water may be a mixture of convecting water from within the well casing and aquifer water moving inward through the screen. This mixing of water during low-flow sampling can substantially increase equilibration times, can cause false stabilization of indicator parameters, can give false indication of redox state, and can provide biological data that are not representative of the aquifer conditions" (Vroblesky 2007).

Failure to calibrate or perform proper maintenance on the sampling equipment and measurement instruments (e.g., dissolved oxygen meter, etc.) can result in faulty data being collected.

Interferences may result from using contaminated equipment, cleaning materials, sample containers, or uncontrolled ambient/surrounding air conditions (e.g., truck/vehicle exhaust nearby).

Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and/or proper planning to avoid ambient air interferences. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

Clean and decontaminate all sampling equipment prior to use. All sampling equipment needs to be routinely checked to be free from contaminants and equipment blanks collected to ensure that the equipment is free of contaminants. Check the previous equipment blank data for the site (if they exist) to determine if the previous cleaning procedure removed the contaminants. If contaminants were detected and they are a concern, then a more vigorous cleaning procedure will be needed.

PERSONNEL QUALIFICATIONS

All field samplers working at sites containing hazardous waste must meet the requirements of the OSHA regulations. OSHA regulations may require the sampler to take the 40 hour OSHA health and safety training course and a refresher course prior to engaging in any field activities, depending upon the site and field conditions.

The field samplers must be trained prior to the use of the sampling equipment, field instruments, and procedures. Training is to be conducted by an experienced sampler before initiating any sampling procedure.

The entire sampling team needs to read, and be familiar with, the site Health and Safety Plan, all relevant SOPs, and SAP/QAPP (and the most recent amendments) before going onsite for the sampling event. It is recommended that the field sampling leader attest to the understanding of these site documents and that it is recorded.

EQUIPMENT AND SUPPLIES

A. Informational materials for sampling event

A copy of the current Health and Safety Plan, SAP/QAPP, monitoring well construction data, location map(s), field data from last sampling event, manuals for sampling, and the monitoring instruments' operation, maintenance, and calibration manuals should be brought to the site.

B. Well keys.

C. Extraction device

Adjustable rate, submersible pumps (e.g., centrifugal, bladder, etc.) which are constructed of stainless steel or Teflon are preferred. Note: if extraction devices constructed of other materials are to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

If bladder pumps are selected for the collection of VOCs and dissolved gases, the pump setting should be set so that one pulse will deliver a water volume that is sufficient to fill a 40 mL VOC vial. This is not mandatory, but is considered a "best practice". For the proper operation, the bladder pump will need a minimum amount of water above the pump; consult the manufacturer for the recommended submergence. The pump's recommended submergence value should be determined during the planning stage, since it may influence well construction and placement of dedicated pumps where water-level fluctuations are significant.

Adjustable rate, peristaltic pumps (suction) are to be used with caution when collecting samples for VOCs and dissolved gases (e.g., methane, carbon dioxide, etc.) analyses. Additional information on the use of peristaltic pumps can be found in Appendix A. If peristaltic pumps are used, the inside diameter of the rotor head tubing needs to match the inside diameter of the tubing installed in the monitoring well.

Inertial pumping devices (motor driven or manual) are not recommended. These devices frequently cause greater disturbance during purging and sampling, and are less easily controlled than submersible pumps (potentially increasing turbidity and sampling variability, etc.). This can lead to sampling results that are adversely affected by purging and sampling operations, and a higher degree of data variability.

D. Tubing

Teflon or Teflon-lined polyethylene tubing are preferred when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics. Note: if tubing constructed of other materials is to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

PVC, polypropylene or polyethylene tubing may be used when collecting samples for metal and other inorganics analyses.

The use of 1/4 inch or 3/8 inch (inside diameter) tubing is recommended. This will help ensure that the tubing remains liquid filled when operating at very low pumping rates when using centrifugal and peristaltic pumps.

Silastic tubing should be used for the section around the rotor head of a peristaltic pump. It should be less than a foot in length. The inside diameter of the tubing used at the pump rotor head must be the same as the inside diameter of tubing placed in the well. A tubing connector is used to connect the pump rotor head tubing to the well tubing. Alternatively, the two pieces of tubing can be connected to each other by placing the one end of the tubing inside the end of the other tubing. The tubing must not be reused.

E. The water level measuring device

Electronic "tape", pressure transducer, water level sounder/level indicator, etc. should be capable of measuring to 0.01 foot accuracy. Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level "tape" at the start and end of each sampling event.

F. Flow measurement supplies

Graduated cylinder (size according to flow rate) and stopwatch usually will suffice.

Large graduated bucket used to record total water purged from the well.

G. Interface probe

To be used to check on the presence of free phase liquids (LNAPL, or DNAPL) before purging begins (as needed).

H. Power source (generator, nitrogen tank, battery, etc.)

When a gasoline generator is used, locate it downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate samples.

I. Indicator field parameter monitoring instruments

Use of a multi-parameter instrument capable of measuring pH, oxidation/reduction potential (ORP), dissolved oxygen (DO), specific conductance, temperature, and coupled with a flow-through-cell is required when measuring all indicator field parameters, except turbidity. Turbidity is collected using a separate instrument. Record equipment/instrument identification (manufacturer, and model number).

Transparent, small volume flow-through-cells (e.g., 250 mLs or less) are preferred. This allows observation of air bubbles and sediment buildup in the cell, which can interfere with the operation of the monitoring instrument probes, to be easily detected. A small volume cell facilitates rapid turnover of water in the cell between measurements of the indicator field parameters.

It is recommended to use a flow-through-cell and monitoring probes from the same manufacturer and model to avoid incompatibility between the probes and flow-through-cell.

Turbidity samples are collected before the flow-through-cell. A "T" connector coupled with a valve is connected between the pump's tubing and flow-through-cell. When a turbidity measurement is required, the valve is opened to allow the groundwater to flow into a container. The valve is closed and the container sample is then placed in the turbidimeter.

Standards are necessary to perform field calibration of instruments. A minimum of two standards are needed to bracket the instrument measurement range for all parameters except ORP which use a Zobell solution as a standard. For dissolved oxygen, a wet sponge used for the 100% saturation and a zero dissolved oxygen solution are used for the calibration.

Barometer (used in the calibration of the Dissolved Oxygen probe) and the conversion formula to convert the barometric pressure into the units of measure used by the Dissolved Oxygen meter are needed.

J. Decontamination supplies

Includes (for example) non-phosphate detergent, distilled/deionized water, isopropyl alcohol, etc.

K. Record keeping supplies

Logbook(s), well purging forms, chain-of-custody forms, field instrument calibration forms, etc.

L. Sample bottles

M. Sample preservation supplies (as required by the analytical methods)

N. Sample tags or labels

O. PID or FID instrument

If appropriate, to detect VOCs for health and safety purposes, and provide qualitative field evaluations.

P. Miscellaneous Equipment

Equipment to keep the sampling apparatus shaded in the summer (e.g., umbrella) and from freezing in the winter. If the pump's tubing is allowed to heat up in the warm weather, the cold groundwater may degas as it is warmed in the tubing.

EQUIPMENT/INSTRUMENT CALIBRATION

Prior to the sampling event, perform maintenance checks on the equipment and instruments according to the manufacturer's manual and/or applicable SOP. This will ensure that the equipment/instruments are working properly before they are used in the field.

Prior to sampling, the monitoring instruments must be calibrated and the calibration documented. The instruments are calibrated using U.S Environmental Protection Agency Region 1 *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity),* January 19, 2010, or latest version or from one of the methods listed in 40CFR136, 40CFR141 and SW-846.

The instruments shall be calibrated at the beginning of each day. If the field measurement falls outside the calibration range, the instrument must be re-calibrated so that all measurements fall within the calibration range. At the end of each day, a calibration check is performed to verify that instruments remained in calibration throughout the day. This check is performed while the instrument is in measurement mode, not calibration mode. If the field instruments are being used to monitor the natural attenuation parameters, then a calibration check at mid-day is highly recommended to ensure that the instruments did not drift out of calibration. Note: during the day if the instrument reads zero or a negative number for dissolved oxygen, pH, specific conductance, or turbidity (negative value only), this indicates that the instrument drifted out of calibration or the instrument is malfunctioning. If this situation occurs the data from this instrument will need to be qualified or rejected.

PRELIMINARY SITE ACTIVITIES (as applicable)

Check the well for security (damage, evidence of tampering, missing lock, etc.) and record pertinent observations (include photograph as warranted).

If needed lay out sheet of clean polyethylene for monitoring and sampling equipment, unless equipment is elevated above the ground (e.g., on a table, etc.).

Remove well cap and if appropriate measure VOCs at the rim of the well with a PID or FID instrument and record reading in field logbook or on the well purge form.

If the well casing does not have an established reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook (consider a photographic record as well). All water level measurements must be recorded relative to this reference point (and the altitude of this point should be determined using techniques that are appropriate to site's DQOs.

If water-table or potentiometric surface map(s) are to be constructed for the sampling event, perform synoptic water level measurement round (in the shortest possible time) before any purging and sampling activities begin. If possible, measure water level depth (to 0.01 ft.) and total well depth (to 0.1 ft.) the day before sampling begins, in order to allow for re-settlement of any particulates in the water column. This is especially important for those wells that have not been recently sampled because sediment buildup in the well may require the well to be redeveloped. If measurement of total well depth is not made the day before, it should be measured after sampling of the well is complete. All measurements must be taken from the established referenced point. Care should be taken to minimize water column disturbance.

Check newly constructed wells for the presence of LNAPLs or DNAPLs before the initial sampling round. If none are encountered, subsequent check measurements with an interface probe may not be necessary unless analytical data or field analysis signal a worsening situation. This SOP cannot be used in the presence of LNAPLs or DNAPLs. If NAPLs are present, the project team must decide upon an alternate sampling method. All project modifications must be approved and documented prior to implementation.

If available check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use final pump dial setting information) from previous event(s). If changes are made in the intake depth or extraction rate(s) used during previous sampling event(s), for either portable or dedicated extraction devices, record new values, and explain reasons for the changes in the field logbook.

PURGING AND SAMPLING PROCEDURE

Purging and sampling wells in order of increasing chemical concentrations (known or anticipated) are preferred.

The use of dedicated pumps is recommended to minimize artificial mobilization and entrainment of particulates each time the well is sampled. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each

sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

A. Initial Water Level

Measure the water level in the well before installing the pump if a non-dedicated pump is being used. The initial water level is recorded on the purge form or in the field logbook.

B. Install Pump

Lower pump, safety cable, tubing and electrical lines slowly (to minimize disturbance) into the well to the appropriate depth (may not be the mid-point of the screen/open interval). The Sampling and Analysis Plan/Quality Assurance Project Plan should specify the sampling depth (used previously), or provide criteria for selection of intake depth for each new well. If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of particulates present in the bottom of the well.

Pump tubing lengths, above the top of well casing should be kept as short as possible to minimize heating the groundwater in the tubing by exposure to sun light and ambient air temperatures. Heating may cause the groundwater to degas, which is unacceptable for the collection of samples for VOC and dissolved gases analyses.

C. Measure Water Level

Before starting pump, measure water level. Install recording pressure transducer, if used to track drawdowns, to initialize starting condition.

D. Purge Well

From the time the pump starts purging and until the time the samples are collected, the purged water is discharged into a graduated bucket to determine the total volume of groundwater purged. This information is recorded on the purge form or in the field logbook.

Start the pump at low speed and slowly increase the speed until discharge occurs. Check water level. Check equipment for water leaks and if present fix or replace the affected equipment. Try to match pumping rate used during previous sampling event(s). Otherwise, adjust pump speed until there is little or no water level drawdown. If the minimal drawdown that can be achieved exceeds 0.3 feet, but remains stable, continue purging.

Monitor and record the water level and pumping rate every five minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump to ensure stabilization of the water level. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" somewhat as pump flow adjustments are made. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. If the initial water level is above the top of the screen do not allow the water level to fall into the well screen. The final purge volume must be greater than the stabilized drawdown volume plus the pump's tubing volume. If the drawdown has exceeded 0.3 feet and stabilizes, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are collected.

Avoid the use of constriction devices on the tubing to decrease the flow rate because the constrictor will cause a pressure difference in the water column. This will cause the groundwater to degas and result in a loss of VOCs and dissolved gasses in the groundwater samples.

Note: the flow rate used to achieve a stable pumping level should remain constant while monitoring the indicator parameters for stabilization and while collecting the samples.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (e.g., bladder, peristaltic), and/or the use of dedicated equipment. For new monitoring wells, or wells where the following situation has not occurred before, if the recovery rate to the well is less than 50 mL/min., or the well is being essentially dewatered during purging, the well should be sampled as soon as the water level has recovered sufficiently to collect the volume needed for all anticipated samples. The project manager or field team leader will need to make the decision when samples should be collected, how the sample is to be collected, and the reasons recorded on the purge form or in the field logbook. A water level measurement needs to be performed and recorded before samples are collected. If the project manager decides to collect the samples using the pump, it is best during this recovery period that the pump intake tubing not be removed, since this will aggravate any turbidity problems. Samples in this specific situation may be collected without stabilization of indicator field parameters. Note that field conditions and efforts to overcome problematic situations must be recorded in order to support field decisions to deviate from normal procedures described in this SOP. If this type of problematic situation persists in a well, then water sample collection should be changed to a passive or no-purge method, if consistent with the site's DQOs, or have a new well installed.

E. Monitor Indicator Field Parameters

After the water level has stabilized, connect the "T" connector with a valve and the flow-through-cell to monitor the indicator field parameters. If excessive turbidity is anticipated or encountered with the pump startup, the well may be purged for a while without connecting up the flow-through-cell, in order to minimize particulate buildup in the cell (This is a judgment call made by the sampler). Water level drawdown measurements should be made as usual. If possible, the pump may be installed the day before purging to allow particulates that were disturbed during pump insertion to settle.

During well purging, monitor indicator field parameters (turbidity, temperature, specific conductance, pH, ORP, DO) at a frequency of five minute intervals or greater. The pump's flow rate must be able to "turn over" at least one flow-through-cell volume between measurements (for a 250 mL flow-through-cell with a flow rate of 50 mLs/min., the monitoring frequency would be every five minutes; for a 500 mL flow-through-cell it would be every ten minutes). If the cell volume cannot be replaced in the five minute interval, then the time between measurements must be increased accordingly. Note: during the early phase of purging emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments followed by stabilization of indicator parameters. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings are within the following limits:

Turbidity (10% for values greater than 5 NTU; if three Turbidity values are less than 5 NTU, consider the values as stabilized),
Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),
Specific Conductance (3%),
Temperature (3%),
pH (± 0.1 unit),
Oxidation/Reduction Potential (±10 millivolts).

All measurements, except turbidity, must be obtained using a flow-through-cell. Samples for turbidity measurements are obtained before water enters the flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values measured within the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and continue monitoring activities. Record start and stop times and give a brief description of cleaning activities.

The flow-through-cell must be designed in a way that prevents gas bubble entrapment in the cell. Placing the flow-through-cell at a 45 degree angle with the port facing upward can help remove bubbles from the flow-through-cell (see Appendix B Low-Flow Setup Diagram). All during the measurement process, the flow-through-cell must remain free of any gas bubbles. Otherwise, the monitoring probes may act erratically. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must remain submerged in water at all times.

F. Collect Water Samples

When samples are collected for laboratory analyses, the pump's tubing is disconnected from the "T" connector with a valve and the flow-through-cell. The samples are collected directly from the pump's tubing. Samples must not be collected from the flow-through-cell or from the "T" connector with a valve.

VOC samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

If the pump's flow rate is too high to collect the VOC/dissolved gases samples, collect the other samples first. Lower the pump's flow rate to a reasonable rate and collect the VOC/dissolved gases samples and record the new flow rate.

During purging and sampling, the centrifugal/peristaltic pump tubing must remain filled with water to avoid aeration of the groundwater. It is recommended that 1/4 inch or 3/8 inch (inside diameter) tubing be used to help insure that the sample tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use the following procedure to collect samples: collect non-VOC/dissolved gases samples first, then increase flow rate slightly until the water completely fills the tubing, collect the VOC/dissolved gases samples, and record new drawdown depth and flow rate.

For bladder pumps that will be used to collect VOC or dissolved gas samples, it is recommended that the pump be set to deliver long pulses of water so that one pulse will fill a 40 mL VOC vial.

Use pre-preserved sample containers or add preservative, as required by analytical methods, to the samples immediately after they are collected. Check the analytical methods (e.g. EPA SW-846, 40 CFR 136, water supply, etc.) for additional information on preservation.

If determination of filtered metal concentrations is a sampling objective, collect filtered water samples using the same low flow procedures. The use of an in-line filter (transparent housing preferred) is required, and the filter size (0.45 μm is commonly used) should be based on the sampling objective. Pre-rinse the filter with groundwater prior to sample collection. Make sure the filter is free of air bubbles before samples are collected. Preserve the filtered water sample immediately. Note: filtered water samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in groundwater for human health or ecological risk calculations.

Label each sample as collected. Samples requiring cooling will be placed into a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

G. Post Sampling Activities

If a recording pressure transducer is used to track drawdown, re-measure water level with tape.

After collection of samples, the pump tubing may be dedicated to the well for re-sampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.1 ft.), if not measured the day before purging began. Note: measurement of total well depth annually is usually sufficient after the initial low stress sampling event. However, a greater frequency may be needed if the well has a "silting" problem or if confirmation of well identity is needed.

Secure the well.

DECONTAMINATION

Decontaminate sampling equipment prior to use in the first well and then following sampling of each well. Pumps should not be removed between purging and sampling operations. The pump, tubing, support cable and electrical wires which were in contact with the well should be decontaminated by one of the procedures listed below.

The use of dedicated pumps and tubing will reduce the amount of time spent on decontamination of the equipment. If dedicated pumps and tubing are used, only the initial sampling event will require decontamination of the pump and tubing.

Note if the previous equipment blank data showed that contaminant(s) were present after using the below procedure or the one described in the SAP/QAPP, a more vigorous procedure may be needed.

Procedure 1

Decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump and tubing. The pump may be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior and electrical wires must be rinsed with the decontaminating solutions, as well. The procedure is as follows:

Flush the equipment/pump with potable water.

Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.

Flush with potable or distilled/deionized water to remove all of the detergent solution. If the water is recycled, the water must be changed periodically.

Optional - flush with isopropyl alcohol (pesticide grade; must be free of ketones {e.g., acetone}) or with methanol. This step may be required if the well is highly contaminated or if the equipment blank data from the previous sampling event show that the level of contaminants is significant.

Flush with distilled/deionized water. This step must remove all traces of alcohol (if used) from the equipment. The final water rinse must not be recycled.

Procedure 2

Steam clean the outside of the submersible pump.

Pump hot potable water from the steam cleaner through the inside of the pump. This can be accomplished by placing the pump inside a three or four inch diameter PVC pipe with end cap. Hot water from the steam cleaner jet will be directed inside the PVC pipe and the pump exterior will be cleaned. The hot water from the steam cleaner will then be pumped from the PVC pipe through the pump and collected into another container. Note: additives or solutions should not be added to the steam cleaner.

Pump non-phosphate detergent solution through the inside of the pump. If the solution is recycled, the solution must be changed periodically.

Pump potable water through the inside of the pump to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.

Pump distilled/deionized water through the pump. The final water rinse must not be recycled.

FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the groundwater samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. Quality control samples include field duplicates, equipment blanks, matrix spike/matrix spike duplicates, trip blanks (VOCs), and temperature blanks.

FIELD LOGBOOK

A field log shall be kept to document all groundwater field monitoring activities (see Appendix C, example table), and record the following for each well:

Site name, municipality, state.

Well identifier, latitude-longitude or state grid coordinates.

Measuring point description (e.g., north side of PVC pipe).

Well depth, and measurement technique.

Well screen length.

Pump depth.

Static water level depth, date, time and measurement technique.

Presence and thickness of immiscible liquid (NAPL) layers and detection method.

Pumping rate, drawdown, indicator parameters values, calculated or measured total volume pumped, and clock time of each set of measurements.

Type of tubing used and its length.

Type of pump used.

Clock time of start and end of purging and sampling activity.

Types of sample bottles used and sample identification numbers.

Preservatives used.

Parameters requested for analyses.

Field observations during sampling event.

Name of sample collector(s).

Weather conditions, including approximate ambient air temperature.

QA/QC data for field instruments.

Any problems encountered should be highlighted.

Description of all sampling/monitoring equipment used, including trade names, model number, instrument identification number, diameters, material composition, etc.

DATA REPORT

Data reports are to include laboratory analytical results, QA/QC information, field indicator parameters measured during purging, field instrument calibration information, and whatever other field logbook information is needed to allow for a full evaluation of data usability.

Note: the use of trade, product, or firm names in this sampling procedure is for descriptive purposes only and does not constitute endorsement by the U.S. EPA.

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U.S Environmental Protection Agency, Region 1, *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity),* January 19, 2010 or latest version.

U.S Environmental Protection Agency, EPA SW-846.

U.S Environmental Protection Agency, 40 CFR 136.

U.S Environmental Protection Agency, 40 CFR 141.

Vroblesky, Don A., Clifton C. Casey, and Mark A. Lowery, Summer 2007, Influence of Dissolved Oxygen Convection on Well Sampling, *Ground Water Monitoring & Remediation* 27, no. 3: 49-58.

APPENDIX A
PERISTALTIC PUMPS

Before selecting a peristaltic pump to collect groundwater samples for VOCs and/or dissolved gases (e.g., methane, carbon dioxide, etc.) consideration should be given to the following:

- The decision of whether or not to use a peristaltic pump is dependent on the intended use of the data.
- If the additional sampling error that may be introduced by this device is NOT of concern for the VOC/dissolved gases data's intended use, then this device may be acceptable.
- If minor differences in the groundwater concentrations could effect the decision, such as to continue or terminate groundwater cleanup or whether the cleanup goals have been reached, then this device should NOT be used for VOC/dissolved gases sampling. In these cases, centrifugal or bladder pumps are a better choice for more accurate results.

EPA and USGS have documented their concerns with the use of the peristaltic pumps to collect water sample in the below documents.

- "Suction Pumps are not recommended because they may cause degassing, pH modification, and loss of volatile compounds" *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001, December 1987.
- "The agency does not recommend the use of peristaltic pumps to sample ground water particularly for volatile organic analytes" *RCRA Ground-Water Monitoring Draft Technical Guidance*, EPA Office of Solid Waste, November 1992.
- "The peristaltic pump is limited to shallow applications and can cause degassing resulting in alteration of pH, alkalinity, and volatiles loss", *Low-flow (Minimal drawdown) Ground-Water Sampling Procedures*, by Robert Puls & Michael Barcelona, April 1996, EPA/540/S-95/504.
- "Suction-lift pumps, such as peristaltic pumps, can operate at a very low pumping rate; however, using negative pressure to lift the sample can result in the loss of volatile analytes", USGS Book 9 Techniques of Water-Resources Investigation, Chapter A4. (Version 2.0, 9/2006).

APPENDIX B

SUMMARY OF SAMPLING INSTRUCTIONS

These instructions are for using an adjustable rate, submersible pump or a peristaltic pump with the pump's intake placed at the midpoint of a 10 foot or less well screen or an open interval. The water level in the monitoring well is above the top of the well screen or open interval, the ambient temperature is above 32°F, and the equipment is not dedicated. Field instruments are already calibrated. The equipment is setup according to the diagram at the end of these instructions.

1. Review well installation information. Record well depth, length of screen or open interval, and depth to top of the well screen. Determine the pump's intake depth (e.g., mid-point of screen/open interval).

2. On the day of sampling, check security of the well casing, perform any safety checks needed for the site, lay out a sheet of polyethylene around the well (if necessary), and setup the equipment. If necessary a canopy or an equivalent item can be setup to shade the pump's tubing and flow-through-cell from the sun light to prevent the sun light from heating the groundwater.

3. Check well casing for a reference mark. If missing, make a reference mark. Measure the water level (initial) to 0.01 ft. and record this information.

4. Install the pump's intake to the appropriate depth (e.g., midpoint) of the well screen or open interval. Do not turn-on the pump at this time.

5. Measure water level and record this information.

6. Turn-on the pump and discharge the groundwater into a graduated waste bucket. Slowly increase the flow rate until the water level starts to drop. Reduce the flow rate slightly so the water level stabilizes. Record the pump's settings. Calculate the flow rate using a graduated container and a stop watch. Record the flow rate. Do not let the water level drop below the top of the well screen.

If the groundwater is highly turbid or colored, continue to discharge the water into the bucket until the water clears (visual observation); this usually takes a few minutes. The turbid or colored water is usually from the well being disturbed during the pump installation. If the water does not clear, then you need to make a choice whether to continue purging the well (hoping that it will clear after a reasonable time) or continue to

the next step. Note, it is sometimes helpful to install the pump the day before the sampling event so that the disturbed materials in the well can settle out.

If the water level drops to the top of the well screen during the purging of the well, stop purging the well, and do the following:

> Wait for the well to recharge to a sufficient volume so samples can be collected. This may take awhile (pump maybe removed from well, if turbidity is not a problem). The project manager will need to make the decision when samples should be collected and the reasons recorded in the site's log book. A water level measurement needs to be performed and recorded before samples are collected. When samples are being collected, the water level must not drop below the top of the screen or open interval. Collect the samples from the pump's tubing. Always collect the VOCs and dissolved gases samples first. Normally, the samples requiring a small volume are collected before the large volume samples are collected just in case there is not sufficient water in the well to fill all the sample containers. All samples must be collected, preserved, and stored according to the analytical method. Remove the pump from the well and decontaminate the sampling equipment.

If the water level has dropped 0.3 feet or less from the initial water level (water level measure before the pump was installed); proceed to Step 7. If the water level has dropped more than 0.3 feet, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are be collected.

7. Attach the pump's tubing to the "T" connector with a valve (or a three-way stop cock). The pump's tubing from the well casing to the "T" connector must be as short as possible to prevent the groundwater in the tubing from heating up from the sun light or from the ambient air. Attach a short piece of tubing to the other end of the end of the "T" connector to serve as a sampling port for the turbidity samples. Attach the remaining end of the "T" connector to a short piece of tubing and connect the tubing to the flow-through-cell bottom port. To the top port, attach a small piece of tubing to direct the water into a calibrated waste bucket. Fill the cell with the groundwater and remove all gas bubbles from the cell. Position the flow-through-cell in such a way that if gas bubbles enter the cell they can easily exit the cell. If the ports are on the same side of the cell and the cell is cylindrical shape, the cell can be placed at a 45-degree angle with the ports facing upwards; this position should keep any gas bubbles entering the cell away from the monitoring probes and allow the gas bubbles to exit the cell easily (see Low-Flow Setup Diagram). Note,

make sure there are no gas bubbles caught in the probes' protective guard; you may need to shake the cell to remove these bubbles.

8. Turn-on the monitoring probes and turbidity meter.

9. Record the temperature, pH, dissolved oxygen, specific conductance, and oxidation/reduction potential measurements. Open the valve on the "T" connector to collect a sample for the turbidity measurement, close the valve, do the measurement, and record this measurement. Calculate the pump's flow rate from the water exiting the flow-through-cell using a graduated container and a stop watch, and record the measurement. Measure and record the water level. Check flow-through-cell for gas bubbles and sediment; if present, remove them.

10. Repeat Step 9 every 5 minutes or as appropriate until monitoring parameters stabilized. Note at least one flow-through-cell volume must be exchanged between readings. If not, the time interval between readings will need to be increased. Stabilization is achieved when three consecutive measurements are within the following limits:

Turbidity (10% for values greater than 5 NTUs; if three Turbidity values are less than 5 NTUs, consider the values as stabilized),
Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),
Specific Conductance (3%),
Temperature (3%),
pH (± 0.1 unit),
Oxidation/Reduction Potential (±10 millivolts).

If these stabilization requirements do not stabilize in a reasonable time, the probes may have been coated from the materials in the groundwater, from a buildup of sediment in the flow-through-cell, or a gas bubble is lodged in the probe. The cell and the probes will need to be cleaned. Turn-off the probes (not the pump), disconnect the cell from the "T" connector and continue to purge the well. Disassemble the cell, remove the sediment, and clean the probes according to the manufacturer's instructions. Reassemble the cell and connect the cell to the "T" connector. Remove all gas bubbles from the cell, turn-on the probes, and continue the measurements. Record that the time the cell was cleaned.

11. When it is time to collect the groundwater samples, turn-off the monitoring probes, and disconnect the pump's tubing from the "T" connector. If you are using a centrifugal or peristaltic pump check the pump's tubing to determine if the tubing is completely filled with water (no air space).

All samples must be collected and preserved according to the analytical method. VOCs and dissolved gases samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

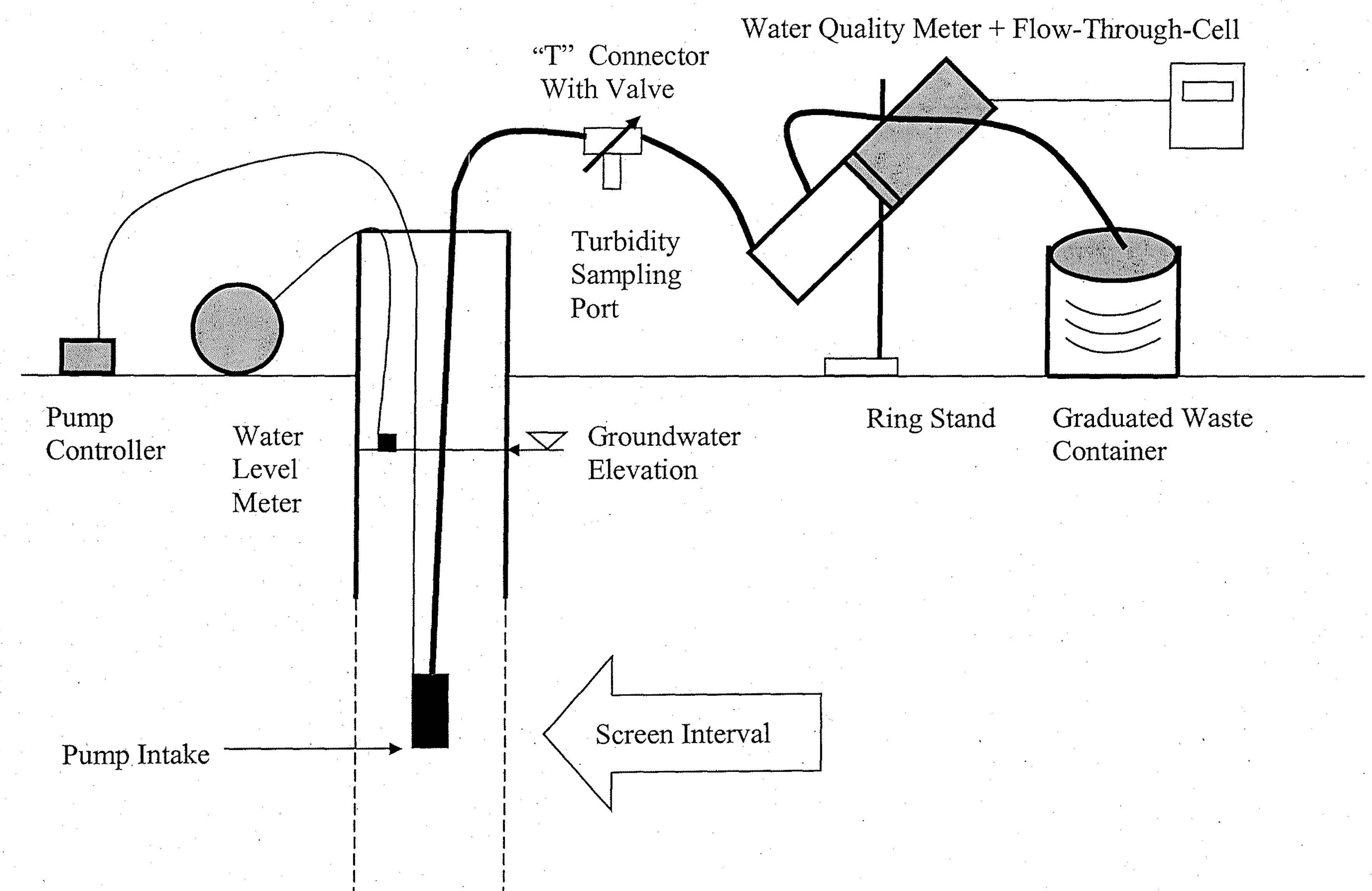
If the pump's tubing is not completely filled with water and the samples are being collected for VOCs and/or dissolved gases analyses using a centrifugal or peristaltic pump, do the following:

> All samples must be collected and preserved according to the analytical method. The VOCs and the dissolved gases (e.g., methane, ethane, ethene, and carbon dioxide) samples are collected last. When it becomes time to collect these samples increase the pump's flow rate until the tubing is completely filled. Collect the samples and record the new flow rate.

12. Store the samples according to the analytical method.

13. Record the total purged volume (graduated waste bucket). Remove the pump from the well and decontaminate the sampling equipment.

Low-Flow Setup Diagram



APPENDIX C

EXAMPLE (Minimum Requirements)

WELL PURGING-FIELD WATER QUALITY MEASUREMENTS FORM

Location (Site/Facility Name)________________________________
Well Number________________Date____________________
Field Personnel________________________________
Sampling Organization________________________________
Identify MP________________________________

Depth to _______/_______ of screen
(below MP) top bottom
Pump Intake at (ft. below MP)_________
Purging Device; (pump type)__________
Total Volume Purged ____________

Clock Time 24 HR	Water Depth below MP ft	Pump Dial[1]	Purge Rate ml/min	Cum. Volume Purged liters	Temp. °C	Spec. Cond.[2] µS/cm	pH	ORP[3] mv	DO mg/L	Turb-idity NTU	Comments
Stabilization Criteria					3%	3%	±0.1	± 10 mv	10%	10%	

1. Pump dial setting (for example: hertz, cycles/min, etc).
2. µSiemens per cm(same as µmhos/cm)at 25°C.
3. Oxidation reduction potential (ORP)

Attachment D-2

Groundwater Sampling Log

Attachment D-2

GROUNDWATER SAMPLING LOG

Well No. ______________________
Key No. ______________________
PID Background (ppm) ______________________
Well Headspace (ppm) ______________________

Site/GMA Name ______________________
Sampling Personnel ______________________
Date ______________________
Weather ______________________

WELL INFORMATION

Reference Point Marked? Y N
Height of Reference Point ________ Meas. From ________
Well Diameter ________
Screen Interval Depth ________ Meas. From ________
Water Table Depth ________ Meas. From ________
Well Depth ________ Meas. From ________
Length of Water Column ________
Volume of Water in Well ________
Intake Depth of Pump/Tubing ________ Meas. From ________

Reference Point Identification:
TIC: Top of Inner (PVC) Casing
TOC: Top of Outer (Protective) Casing
Grade/BGS: Ground Surface

Redevelop? Y N

Additional well maintenance needed? Y N (if yes, describe below)

Sample Time ______________________
Sample ID ______________________
Duplicate ID ______________________
MS/MSD ______________________
Split Sample ID ______________________

Required	Analytical Parameters:	Collected
( )	VOCs (Standard List)	( )
( )	VOCs (Expanded List)	( )
( )	SVOCs	( )
( )	PCBs (Unfiltered)	( )
( )	PCBs (Filtered)	( )
( )	Metals/Inorganics (Unfiltered)	( )
( )	Metals/Inorganics (Filtered)	( )
( )	Total Cyanide (Unfiltered)	( )
( )	Total Cyanide (Filtered)	( )
( )	PAC Cyanide (Filtered)	( )
( )	PCDDs/PCDFs	( )
( )	Pesticides/Herbicides	( )
( )	Natural Attenuation	( )
( )	Other (Specify)	( )

EVACUATION INFORMATION

Pump Start Time ________
Pump Stop Time ________
Minutes of Pumping ________
Volume of Water Removed ________
Did Well Go Dry? Y N

Evacuation Method: Bailer ( ) Bladder Pump ( )
Peristaltic Pump ( ) Submersible Pump ( ) Other/Specify ( )
Pump Type: ______________________
Samples collected by same method as evacuation? Y N (specify)

Water Quality Meter Type(s) / Serial Numbers: ______________________

Time	Pump Rate (L/min.)	Total Gallons Removed	Water Level (ft TIC)	Temp. (Celsius) [3%]*	pH [0.1 units]*	Sp. Cond. (mS/cm) [3%]*	Turbidity (NTU) [10% or 1 NTU]*	DO (mg/l) [10% or 0.1 mg/l]*	ORP (mV) [10 mV]*

* The stabilization criteria for each field parameter (three consecutive readings collected at 3- to 5-minute intervals) is listed in each column heading.

OBSERVATIONS/SAMPLING METHOD DEVIATIONS ______________________

____

____

____

SAMPLE DESTINATION

Laboratory: ______________________
Delivered Via: ______________________
Airbill #: ______________________

Field Sampling Coordinator: ______________________

Attachment D-2

GROUNDWATER SAMPLING LOG

Well No. ____________________

Site/GMA Name ____________________

Sampling Personnel ____________________

Date ____________________

Weather ____________________

WELL INFORMATION - See Page 1

Time	Pump Rate (L/min.)	Total Gallons Removed	Water Level (ft TIC)	Temp. (Celsius) [3%]*	pH [0.1 units]*	Sp. Cond. (mS/cm) [3%]*	Turbidity (NTU) [10% or 1 NTU]*	DO (mg/l) [10% or 0.1 mg/l]*	ORP (mV) [10 mV]*

* The stabilization criteria for each field parameter (three consecutive readings collected at 3- to 5-minute intervals) is listed in each column heading.

OBSERVATIONS/SAMPLING METHOD DEVIATIONS ____________________

Attachment D-2

GROUNDWATER SAMPLING LOG

Well No. ______________________

Site/GMA Name ______________________
Sampling Personnel ______________________
Date ______________________
Weather ______________________

WELL INFORMATION - See Page 1

OBSERVATIONS/SAMPLING METHOD DEVIATIONS ______________________

Attachment D-3

Sample Container Label

Attachment D-3

ARCADIS

Project #		Date
Sample I.D.		
Sample Type ☐ Soil/Sediment ☐ Water	Collection Mode ☐ Composite ☐ Grab	Time
Analysis		
Sampler(s)	Preservative	

Appendix E

Surface Water Sampling Procedures

Surface Water Sampling Procedures

I. Introduction

This appendix specifies several types of surface water sampling procedures. These include procedures for collecting surface water samples for subsequent chemical analysis; procedures for obtaining velocity profile measurements at selected river/stream cross-sections; and procedures for installing and using maximum-minimum thermometers to measure water column temperature.

II. Surface Water Sampling for Chemical Analysis

This section specifies the procedures for collecting surface water samples for chemical analysis. Several methods for collecting surface water samples are available, depending on the type of surface water to be sampled (i.e., rivers, streams, discharges, ponds, or impoundments). Regardless of the sample collection method used, sampling will not take place during precipitation events (unless so specified in the project-specific work plan), and samples will be obtained beginning with the most downstream location and proceeding upstream.

<u>Materials</u>

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

- Health and safety equipment (as required by the Health and Safety Plan);
- Cleaning equipment (as required in Appendix W);
- Boat;
- Rope;
- Surveyor's rod and/or 6-foot rule;
- Duct tape;
- Measuring tape;
- Thermometer;
- Electromagnetic velocity meter;
- Large glass or stainless steel mixing container;
- Beaker or equivalent glass measuring device;
- Field notebook;
- Conductivity/temperature meter;

- pH meter;
- 0.45 micron Versapor membrane in-line disposable filter;
- Appropriate blanks (trip), if necessary;
- Appropriate sampling containers and forms;
- Appropriate preservatives (as required);
- Coolers with ice or "blue" ice; and
- Appropriate water sampler as specified in the project-specific work plan, which may include following:
- Surface water grab sampler (Attachment E-1) consisting of a 1,000 mL beaker, adjustable clamp, and two-or three-piece telescoping aluminum tube or an equivalent acceptable sampling device; or
- Kemmerer stainless steel bottle sampler (Attachment E-2).

<u>Procedures</u>

A. The following procedures will be used to obtain grab samples:

Step 1 - Identify surface water sampling location on appropriate sampling log sheet (Attachment E-3) and/or field notebook along with other appropriate information;

Step 2 - Don health and safety equipment (as required by the Health and Safety Plan);

Step 3 - Clean the sampling equipment in accordance with the procedures in Appendix W;

Step 4 - Assemble the water grab sampler (Attachment E-1). Make sure that the sampling beaker and the bolts and nuts that secure the clamp to the pole are tightened properly;

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

Step 6 - Retrieve the water sampler from the surface water with minimal disturbance;

Step 7 - Remove the cap from the large glass or stainless steel mixing container and slightly tilt the mouth of the container below the sampling device;

Step 8 - Empty the sampler slowly, allowing the sample stream to flow gently down the side of the container with minimal entry turbulence;

Step 9 - Continue delivery of the sample until the mixing container contains a sufficient volume for all laboratory samples;

Step 10 - Mix the entire sample volume with the Teflon® stirring rod and transfer the appropriate volume into the laboratory sample jar. (Volatile samples will not be homogenized.) Preserve samples as specified in Table 1 of the FSP/QAPP;

Step 11 - When sampling for volatiles, surface water samples will be collected directly from the water sampler to 40 mL vials with Teflon® liners;

Step 12 - The sample collection order (as appropriate) will be as follows:

1. VOCs;
2. TOC;
3. SVOCs;
4. Metals and cyanide; and
5. Others.

Step 13 - If sampling for total and filtered metals, a filtered and unfiltered sample will be collected. Sample filtration for the filtered sample will be performed in the field utilizing a peristaltic pump prior to preservation. Install new medical-grade silicone tubing in the pump head. Place new Teflon® tubing into the sample mixing container and attach to the intake side of pump tubing. Attach (clamp) a new 0.45 micron filter to the discharge side of the pump tubing (noting the correct filter flow direction). Turn the pump on and dispense the filtered liquid directly into the laboratory sample bottles. Document sample collection on Peristaltic Pump Sampler Field Log (Attachment E-4).

Step 14 - If sampling for total and filtered PCBs, two samples must be collected, one of which will be filtered by the laboratory prior to analysis;

Step 15 - Secure the sample jar cap(s) tightly;

Step 16 - Label all sample containers as appropriate, as discussed in Appendix L;

Step 17 - After sample containers have been filled, fill a beaker or glass container with the water sample and measure the pH and conductivity, as discussed in Appendix O. Alternatively, direct measurements for pH and conductivity can be taken at the approximate mid-depth location as detailed in Appendix O;

Step 18 - Measure the water temperature at the approximate mid-depth location, and measure the ambient air temperature;

Step 19 - Record required information on the appropriate forms and/or field notebook; and

Step 20 - Handle, pack, and ship the samples in accordance with the procedures in Appendix L.

B. To obtain surface water samples at depth from lakes (including Silver Lake), ponds, and impoundments with water depth greater than 6 to 8 feet, a Kemmerer sampler (Attachment E-4) will be used (for all analytes.) To use the Kemmerer sampler, the Kemmerer bottle is lowered to the approximate mid-depth location and the device trigger is released causing the sample vessel to be filled. After the sample vessel is filled, the Kemmerer sampler is slowly raised to minimize disturbance to the sample. Repeat Steps 7 through 20 from Procedure A after sample vessel is retrieved.

III. Velocity Profile Measurement Procedures

The following materials will be required for this activity:

- Health and safety equipment (as required by the Health and Safety Plan);
- Field notebook and pen;
- Calculator;
- Boat;
- Rope;
- Surveyor's rod;
- Duct tape;
- Measuring tape; and
- Electromagnetic velocity meter.

Note: Based on extensive past experience in obtaining velocity measurements in the Housatonic River, the electromagnetic velocity meter is the most appropriate flow measurement device for measuring velocity in the river's different flow regimes and channel configurations.

The following procedures will be used to determine the velocity profile at river/stream cross sections:

Step 1 - Don personal protective equipment (as required in the Health and Safety Plan).

Step 2 - Extend rope across the river/steam.

Step 3 - Measure the width of the river/stream, then divide and mark into equally spaced measurement locations. For rivers/streams less than 30 feet in width, the spacing should be 5 feet. For rivers/streams between 30 feet and 100 feet in width, the spacing should be 10 feet; and for rivers/streams greater than 100 feet in width, the spacing should be 20 feet.

Step 4 - Calibrate velocity meter as per manufacturer's specifications.

Step 5 - Lower the surveyor's rod and measure and record the water depth to the nearest 0.1 foot at each measurement location.

Step 6 - Velocities will be determined using the two-point method. Attach the velocity meter probe to the surveyor's rod, measure, and record the velocity in feet per second at depths equaling 0.2 and 0.8 times the total river depth at each measurement location. Average the two velocity measurements to obtain the average velocity for that vertical section.

Step 7 - Record all measurements in field notebook.

Step 8 - Calculate the river flow rate by multiplying the average velocity reading for a particular vertical section times the area represented by the portion of the total cross-section extending half-way to the adjacent vertical sections (i.e., the Avelocity-area method@). The total flow rate is the sum of the flow of the partial sections.

$$Q_T = V_1 A_1 + V_2 A_2 + ... + V_n A_n$$

Where: Q_T = Total flow in cubic feet per second

V_{1-n} = Average velocity for a vertical section in feet per second

A_{1-n} = Cross-section area extending half-way to the adjacent vertical sections in square feet.

IV. Maximum-Minimum Thermometer Gauging

At several sites on the Housatonic River, the temperatures of the water column will be measured and recorded throughout a given season through the use of maximum-minimum (max-min) thermometers. Following installation, the thermometers will be checked periodically to monitor temperatures. The purpose of such measurements is to provide information regarding potential impacts of water temperature on fish distribution in the river.

The following materials will be required for this activity:

- Max-min thermometers;
- Wooden stake, rebar, and/or wire for fastening thermometer to stream bottom;
- String;
- Waders; and
- Appropriate data sheets.

The following procedures will be followed in installing the max-min thermometers and using them to record water temperature:

Step 1 - Each thermometer should be placed at a fixed point in the river that allows it to be submerged throughout the summer at both high and low flows. The location should be somewhat hidden to discourage tampering, but should be conveniently located to facilitate reading.

Step 2 - The thermometer should be tied with string to a fixed object so that if it becomes dislodged, it will not be lost downstream.

Step 3 - The thermometer should be read periodically (as specified in the work plan), recording the maximum and minimum temperatures that have occurred since the last reading as well as the present temperature.

Step 4 - The max-min markers in the thermometer should be reset to the current temperature after being read. The thermometer should then be returned to the stream.

V. Duplicate Sample Collection

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

VI. Survey

A field survey control program will be conducted using standard instrument survey techniques to document the surface water sampling locations when necessary to have record of the exact location. Generally, to accomplish this, a local control baseline will be set up. This local baseline control may then be tied into the

appropriate vertical and horizontal datum such as the National Geodetic Vertical Datum of 1929 and the State Plane Coordinate System.

VII. Equipment Cleaning

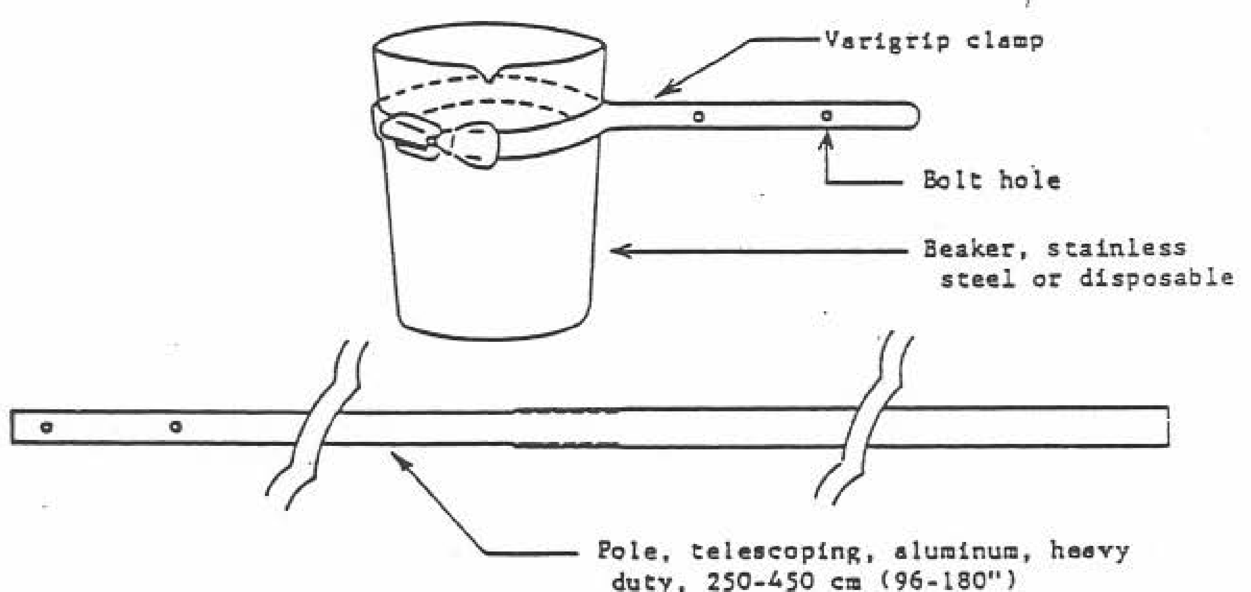
Equipment cleaning will occur at the beginning of each sampling event and between each sampling location as described in Appendix W.

VIII. Disposal Methods

Rinse water, PPE, and other residuals generated during the equipment cleaning procedures will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its ongoing disposal practices.

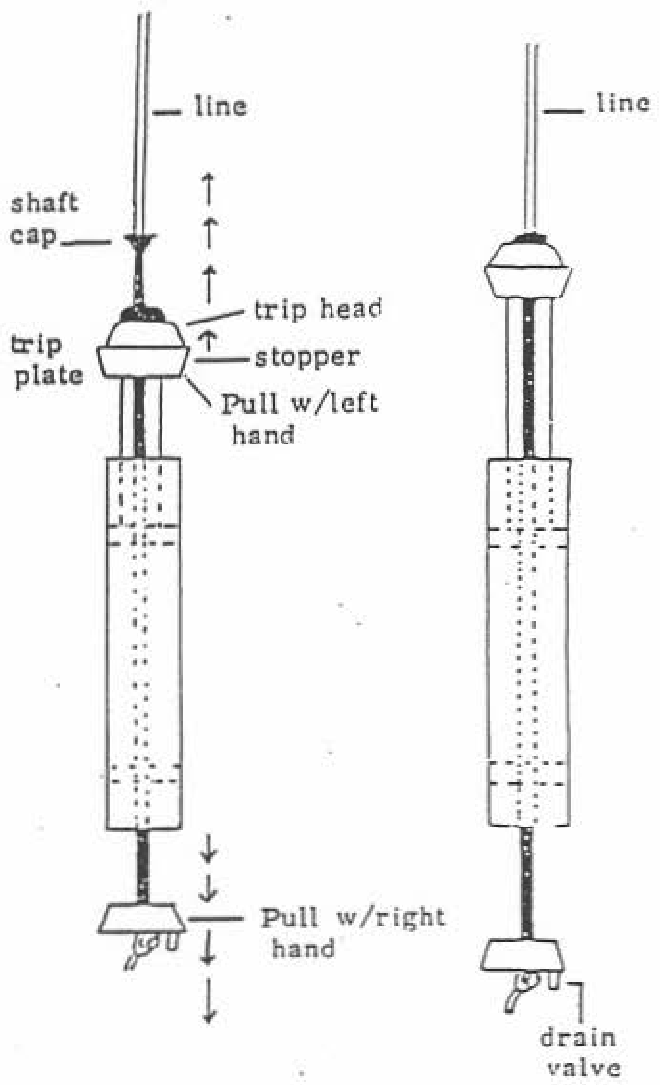
Attachment E-1

Surface Water Grab Sampler


Varigrip clamp
Bolt hole
Beaker, stainless
steel or disposable
Pole, telescoping, aluminum, heavy
duty, 250-450 cm (96-180")

Attachment E-2

Kemmerer Bottle


line
shaft cap
trip head
trip plate
stopper
Pull w/left hand
Pull w/right hand
line
drain valve

Attachment E-3

Surface Water Grab Sample Field Log

Surface Water Grab Sample Field Log

Client ____________________

Site ____________________

Project No. ____________________

Sampling Personnel ____________________

Date ____________________

Time ____________________

Weather ____________________

I. Sample Location

Sample depth ________

Approximate flow rate ____________________

Volume of glass beaker ____________________

II. Surface Water Sampling Information

Distance from bank sampled ____________________

Depth below surface of water removed ____________________

III. Physical Appearance of Sample

Color ____________________

Odor ____________________

Suspended Soils ____________________

Film ____________________

IV. Container Analysis

V. Surface Water Characteristics

Color ________

Temperature ________

Suspended Solids ____________________

Film ____________________

Odor ____________________

Attachment E-4

Peristaltic Pump Sampler Field Log

Peristaltic Pump Sampler Field Log

Location ___________ Date ____________, 19__ Sampled by: _____________

Location No. _______ Gauge Reading ________________________________

Measured Water Depth (D) ________ ft Air _______°F Water _________°F

Weather _________________________ Flow Conditions _______________

Sample ID	Date	Time	Sample Depth (feet off bottom)	Analysis

Comments ____

Appendix F

Sediment Sampling Procedures

Sediment Sampling

I. Introduction

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

If specified in the project-specific work plan, reconnaissance and sediment probing will be conducted prior to sediment sampling in rivers, streams, ponds, or impoundments to identify areas of significant sediment deposition. Sediment probing will be accomplished by floating in a boat and/or by wading along shallow areas and physically probing with a metal rod for sediment deposition areas. Sediment sampling locations will be selected from data collected during the field during reconnaissance.

Procedures to be utilized for sediment harvesting, sediment traps, and settleability testing are provided as Attachments F-1, F-2, and F-3, respectively.

II. Sediment Reconnaissance/Probing Procedures

A. Materials

The following materials will be available as required during sediment reconnaissance activities:

- health and safety equipment, as required by the *Health and Safety Plan* (HASP);
- boat;
- flagging;
- field notebook;
- surveyor's rod or 6-foot rule;
- measuring tape; and
- metal rod graduated for sediment depth measurement.

B. Procedures

Step 1 Identify the site limits (area to be probed) and locate position on aerial photographs or detailed mapping.

Step 2 Don personal protective equipment (PPE), as required by the HASP.

Step 3 Begin physically probing for sediments with a metal rod by floating in a boat and/or by wading along the identified area. Probe the bottom at regular intervals (i.e., if in a stream area, probe along both sides of channel and across the mid-section of the stream) to identify the location of significant sediment deposits. Soft areas which are penetrable with the rod will be considered sediment deposits. As sediment deposits are located, each will be marked with flagging and plotted on an aerial photograph or detailed mapping of the probed area.

Step 4 Probe the sediment deposit area to determine the approximate average sediment depth;

Step 5 Obtain the approximate measurements of the sediment deposits to determine surface area.

Step 6 Record the following information in the field record book: approximate location, date, personnel, weather, average sediment depth, length and width of sediment deposit, average water depth cover, stream width, sediment type, type of depositional environment, and any other pertinent comments.

III. Sediment Sampling

A. Materials

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

- health and safety equipment, as required by the HASP;
- cleaning equipment, as required in Appendix W;
- boat;
- Teflon® sheet or stainless steel tray;
- duct tape;
- Lexan® tubing with end caps;
- brass push rod;
- graduated rod for sediment depth measurement;
- hacksaw;
- vacuum pump;
- end cap with appropriate fitting for vacuum pump attachment;

- Teflon® tubing;
- 6-foot rule or survey rod;
- transport container with ice or "blue" ice;
- appropriate sample containers and forms; and
- field notebook.

B. Procedures

Step 1 Identify the proposed sample location on the sampling log sheet (Attachment F-4) and/or field notebook, along with other appropriate information collected during sediment sampling activities.

Step 2 Don PPE, as required by the HASP.

Step 3 At each sample location, lower a section of Lexan® tube until it just reaches the top of sediment. Measure the depth of water. (Sections of Lexan® tube may need to be spliced together in deep water locations.)

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

Step 5 Drive the tube several more inches using a stainless steel core driver block and measure the distance. This procedure is performed to obtain a "plug" at the bottom of the core and prevent the loose sediment from escaping.

Step 6 Place a vacuum pump on the top end of the Lexan® tube (using a modified end cap with a fitting for attachment of the vacuum pump) and create a vacuum to prevent the sediments/plug from escaping. The vacuum is applied to the water column on top of the sediment core and does not directly affect the sediment sample to minimize the potential loss of volatile organic compounds (VOCs). In addition, when VOC samples are to be collected, the application time and magnitude of the vacuum will be minimized to the extent practical.

Step 7 Slowly pull the tube from the sediment, twisting it slightly as it is removed (if necessary).

Step 8 Before the tube is fully removed from the water, place a cap on the bottom end of the tube while it is still submerged.

Step 9 Keeping the tube upright, wipe the bottom end dry, seal the cap with duct tape, and label. Measure the length of sediment recovered and evaluate the integrity of the core. If the core is not suitably intact, repeat coring procedure within 5 to 10 feet of the first location attempted.

Step 10 Transport the core sample to the shore.

Step 11 While still keeping the core upright, use a hacksaw to make a horizontal cut in the tube approximately 1 inch above the sediment. After cutting, carefully pour off any excess standing water.

Step 12 Re-cap the cut end of the tube, seal the cap with duct tape, and mark this end as “top.”

Step 13 Wipe the tube dry.

Step 14 Place a sample label on the tube.

Step 15 Record the following information on both the tube and on the cap: 1) sample number; 2) sampling date; and 3) sampling time.

Step 16 Place the core sample upright in a container on ice.

Step 17 Repeat the above procedures until all core samples are collected (for the sampling event or the sampling day).

Step 18 Sediment cores will be extruded from the Lexan® tubing onto a Teflon® sheet or stainless steel tray. Describe and record sample description. After extrusion, scrape the top of the core with a decontaminated stainless steel spatula to remove any Lexan® saw chips that may have accumulated during the tubing cutting procedure.

Step 19 Cores will be sectioned into depth-proportioned increments as specified in the work plan. If sampling for VOCs, the core section will be placed immediately into the sample jar (without compositing) following extrusion from the Lexan® tubing. VOC samples will be collected following the procedures in Appendix A.

Step 20 Core sections may be frozen to facilitate sectioning when sediment is extremely loose.

Step 21 The saw or knife used to section the core should be cleaned (as described in Appendix W) between each cut.

Step 22 Prepare equipment blank samples at the frequency specified in Table 4 of the *Field Sampling Plan/ Quality Assurance Project Plan (*FSP/QAPP) by collecting distilled/deionized water that has been used to rinse the hacksaw and a representative section of the Lexan® tubing.

Step 23 Label all sample containers with: 1) site; 2) project number; 3) location number; 4) sample interval; 5) date; 6) time of core collection; and 7) names of sampling personnel.

Step 24 Handle, pack, and ship the samples in accordance with the procedures in Appendix L.

Step 25 Record all appropriate information in the field notebook and sampling log form(s).

IV. Duplicate Sample Collection

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

V. Survey

A field survey control program will be conducted using standard instrument survey techniques to document the sediment sampling location. Generally, to accomplish this, a local control baseline will be set up. This local baseline control may then be tied into the appropriate vertical and horizontal datum such as the National Geodetic Vertical Datum of 1929 and the State Plane Coordinate System.

VI. Equipment Cleaning

Equipment cleaning of the saw or knife used for core sample sectioning will be performed between each cut as described in Appendix W. Equipment cleaning of any sampling equipment which is re-used at another sample location will be performed as described in Appendix W.

VII. Disposal Methods

Rinse water, PPE, and other residuals generated during the equipment cleaning procedures will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its on-going disposal practices.

Attachment F-1

Sediment Harvesting Procedures

Attachment F-1: Sediment Harvesting Procedures

I. Introduction

The procedures below are for surface water filtration and suspended solids sample preparation related to the harvesting of surface water suspended solids.

II. Materials

The following equipment will be utilized, as necessary during sediment harvesting:

- health and safety equipment, as required by the *Health and Safety Plan* (HASP);
- cleaning equipment;
- surface water composite sample (5 gallons) collected at mid-depth and mid-channel utilizing a peristaltic pump and the procedures in Appendix E;
- stainless steel filter holder;
- stainless steel forceps;
- compressed nitrogen gas tank;
- appropriate sample containers;
- appropriate forms and/or field notebook;
- insulated coolers with ice; and
- 0.7-micron glass fiber filters.

III. Filtration and Sample Preparation Procedures

Step 1 Remove 0.7-micron pore-size glass fiber filter from aluminum foil with stainless steel forceps.

Step 2 Place the glass fiber filter onto the stainless steel filter plate.

Step 3 Assemble filter holder.

Step 4 Add one liter of sample to the filter assembly reservoir.

Step 5 Attach hose from the compressed nitrogen canister to filter assembly and pressurize the filter assembly reservoir.

Step 6 When the one liter quantity of water is filtered, turn off nitrogen gas supply and release pressure from the reservoir.

Step 7 Repeat Steps 4 through 6 until flow rate through filter is significantly reduced due to clogging of the filter by sediments.

Step 8 When the filter becomes clogged, complete the filtering of the water present in the reservoir, turn off nitrogen gas supply, release pressure, and disassemble the filter holder.

Step 9 With forceps, gently remove the filter from the filter plate and place in aluminum foil wrap.

Step 10 Record volume of water filtered through filter.

Step 11 Repeat Steps 1 through 10 until entire volume of water sample is filtered.

Step 12 Label wrap filter for shipment in cooler to the laboratory in accordance with procedures in Appendix L.

Step 13 Handle, pack, and ship cooler using the chain-of-custody procedures in accordance with Appendix L.

Attachment F-2

Sediment Trap

Attachment F-2: Sediment Trap Procedures

This protocol describes the procedures to collect a representative sample of newly deposited sediments by using a fixed-location sediment trap.

A. Materials

The following materials will be available, as required, during installation and retrieval of the sediment trap.

- health and safety equipment, as required by the *Health and Safety Plan* (HASP);
- boat;
- sediment trap (construction detailed below);
- weight (for mooring the trap);
- buoy;
- rope;
- end caps for Lexan® cylinder;
- wide-mouth glass sample jar;
- glass rod;
- distilled water; and
- transport container with ice.

B. Procedure for Installing the Sediment Trap

Step 1 Identify the proposed sampling location.

Step 2 Measure depth to sediment/water interface (a minimum of approximately 5 feet is required).

Step 3 Place buoy in water.

Step 4 Slowly lower the sediment trap and attached mooring weight into the water until the weight rests on the bottom.

Step 5 Attach the rope from the sediment trap to the buoy.

C. Procedure for Retrieval of Sediment Trap and Collection of Sample

Step 1 Locate buoy.

Step 2 Slowly raise the sediment trap and mooring weight to the surface.

Step 3 Inspect the cylinder and record sediment depth and other notable observations.

Step 4 Cap each cylinder for transport to shore, taking care to avoid agitation.

Step 5 On shore, remove each cylinder from the sediment trap frame and decant (or drain) off excess water above the sediment.

Step 6 Transfer sediment/water mix to wide-mouth glass jar, compositing sediments from several cylinders until each jar is full.

Step 7 If sediment does not easily pour from a cylinder, dislodge sediments with a glass rod and rinse into sample jar with distilled water.

Step 8 From each sample jar, record the number of cylinders composited.

Step 9 Cap the sample jars and label according to procedures in Appendix L.

Step 10 Place the sample jars upright into a container with ice.

Step 11 Handle, pack, and ship the samples using the chain-of-custody procedures in accordance with Appendix L.

D. Construction of Sediment Trap

The proposed design of the sediment trap consists of a series of 2-inch-diameter, 15-inch-long Lexan® cylinders placed within a frame such that the collection area (top of cylinders) is 24 to 30 inches above the sediment bed. To provide sufficient surface collection area, multiple cylinders will be used for a single sediment trap. The frame will be attached to a weight to keep it moored and to a buoy to keep it suspended approximately 1 foot off the bottom.

Attachment F-3

Settleability Testing

SETTLING TEST PROCEDURES*

PART I: TESTING EQUIPMENT AND PROCEDURES

Test Objective

1. The objective of running settling tests on sediments to be dredged is to define, on a batch basis, their settling behavior in a large-scale, continuous-flow dredged material containment area. The tests provide numerical values for the design criteria which can be projected to the size and design of the containment area.

Test Equipment

2. The settling column shown in Figure 1 should be used for dredged material settling tests (Montgomery 1978). The column is constructed of 8-inch Plexiglas tubing and can be sectioned for easier handling and cleaning. Shop drawings of the column with bills of materials are available from the WES Environmental Laboratory.

Samples

3. Samples used to perform settling tests should consist of fine-grained (<No. 40 sieve) material. If coarse-grained (>No. 40 sieve) material present in the sample is less than 10 percent (dry weight basis), separation is not required prior to sedimentation testing. A composite of several sediment samples may be used to perform the tests if this is thought to be more representative of the dredged material. Approximately 15 gal of sediment is usually required for the tests.

* Material in this Appendix was adapted from Draft EM 1110-2-5027 "Confined Disposal of Dredged Material" (US Army Engineer Waterways Experiment Station 1985).

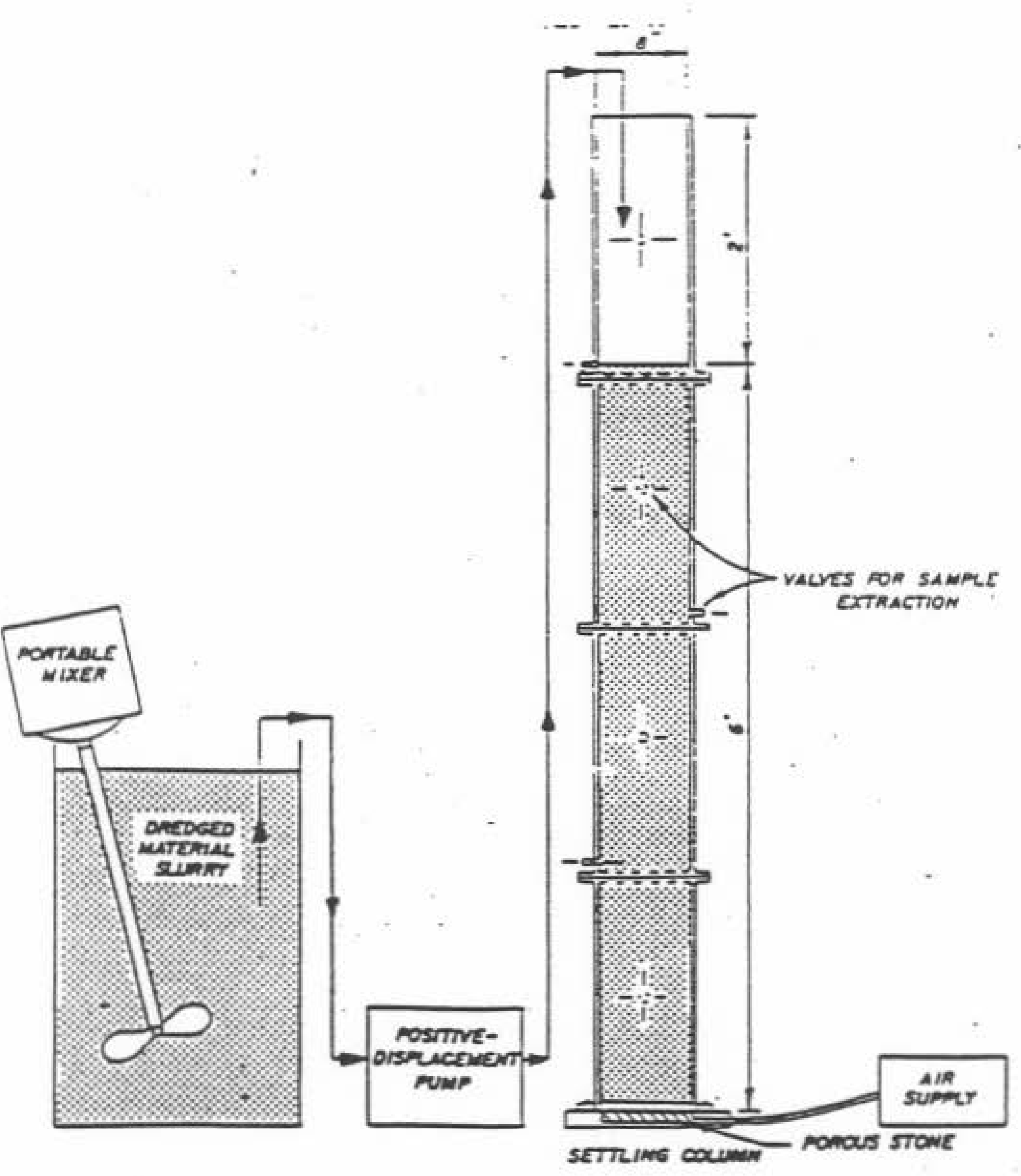
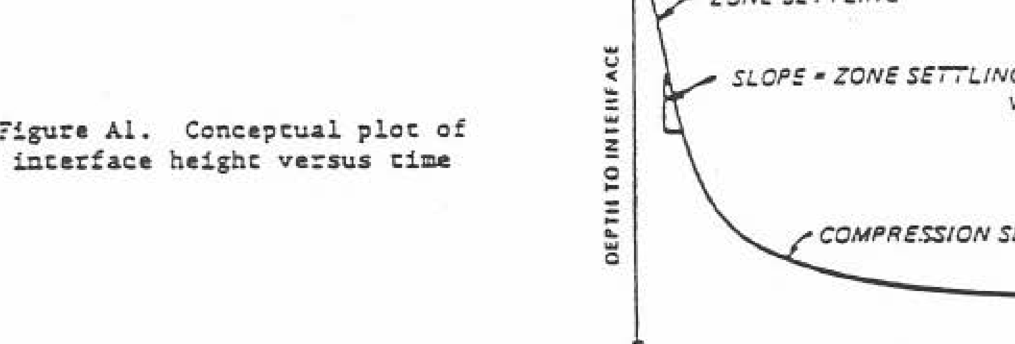


Figure 1. Schematic diagram of apparatus for settling tests

Figure A1. Conceptual plot of interface height versus time



Test Procedures

Pilot test

4. A pilot test conducted in a small graduated cylinder (1 ℓ is satisfactory) is a useful method for determining whether flocculent settling or zone settling processes will prevail during the initial settling. The pilot test should be run at a slurry concentration of approximately 150 g/ℓ. If an interface forms within the first few hours of the test, the slurry mass is exhibiting zone settling, and the fall of the interface versus time should be recorded. The curve will appear as shown in Figure A1. The break in the curve will define the concentration at which compression settling begins. Only concentrations lower than this transition calculation should be used for the zone settling test series in the 8-inch column. If no break in the curve is evident, the material began settling in the compression zone, and the pilot test should be repeated at a lower slurry concentration.

5. It should be emphasized that use of a small cylinder as in the pilot test is not acceptable for use in design. Wall effects for columns of small diameter affect zone settling velocities, and data obtained using small-diameter columns will not accurately reflect field behavior.

6. If no interface is observed in the pilot test within the first few hours, the slurry mass is exhibiting flocculent settling. In this case, the pilot test should be continued until an interface is observed between the turbid water above and more concentrated settled solids below. The concentration

the settled solids (computed assuming zero concentration of solids above) an indication of the concentration at which the material exhibits compres-on settling.

quired number of
lumn loadings for tests

7. Three types of settling tests may be needed to fully define the set-ling properties of the dredged material. However, in many cases the 8-in. ettling column used for the settling tests need only be loaded with slurry nce. A compression settling test is needed to define the volume which will e occupied in the disposal area by a newly deposited dredged material layer. lso, a flocculent settling test for either the slurry mass or for the super-atant water above any interface is required to predict effluent suspended olids concentrations. Both of these tests should be conducted at a slurry oncentration equal to the expected influent concentration. Therefore, only ne loading of the test column would be required to collect data for both pur-oses. A series of zone settling tests is required to define the minimum equired surface area needed for effective zone settling. For the zone set-ling test series, the pilot test will define the highest concentration which should be used for the series. If the column is initially loaded for this condition, the same material in the column can be used for the remaining tests by draining appropriate volumes of slurry (remixed following a test by agitat-ing with compressed air) and replacing the drained slurry with an equal volume of water of appropriate salinity.

PART II: SETTLING TESTS

Flocculent Settling Test

8. The flocculent settling test consists of measuring the concentration of suspended solids at various depths and time intervals in a settling column. If an interface forms near the top of the settling column during the first day of the test, sedimentation of the material below the interface is described by zone settling. In that case, the flocculent test procedure should be continued only for that portion of the column contents above the interface.

9. Information required to design a containment area in which flocculent settling occurs can be obtained using the following procedure:

a. Use a settling column such as the one shown in Figure 1. The slurry depth used in the test column should approximate the effective settling depth of the proposed containment area. A practical upper limit on the depth of the test is 6 ft. The column should be at least 8 in. in diameter, with sample ports at 0.5-ft intervals (minimum). The column should have provisions for slurry agitation with compressed air from the bottom to keep the slurry mixed during the column filling period.

b. Mix the sediment slurry to the desired suspended solids concentration selected to represent the expected concentration of the dredged material influent C_i. The slurry should be mixed in a container with sufficient volume to fill the test column. Field studies indicate that for maintenance dredging the fine-grained material concentration will average about 150 g/ℓ. This should be the concentration used in the test if better data are not available.

c. Pump or pour the slurry into the test column, using compressed air to maintain a uniform concentration during the filling period.

d. When the slurry is completely mixed in the column, cut off the compressed air and immediately draw off samples at each sample port and determine their suspended solids concentration. Use the average of these values as the initial slurry concentration at the start of the test. The test is considered initiated when the initial samples are drawn.

e. If an interface has not formed on the first day, flocculent settling is occurring in the entire slurry mass. Allow the slurry to settle and withdraw samples from each sampling port at regular time intervals to determine the suspended solids concentrations. Substantial reductions of suspended solids will occur

during the early part of the test, but reductions will lessen at longer retention times. Therefore, the intervals between sampling can be extended as the test progresses. Recommended sampling intervals (in hours) are: 1, 2, 4, 6, 12, 24, 48, etc. until the end of the test. As a rule, a 50-ml sample should be taken from each port. Continue the test until an interface of solids can be seen near the bottom of the column and the suspended solids concentration in the fluid above the interface is <1 g/ℓ. Tabulate test data and use them to plot a concentration profile diagram like the one shown in Figure A2.

f. If an interface forms the first day, zone settling is occurring in the slurry below the interface, and flocculent settling is occurring in the supernatant water. For this case, samples should be extracted from all side ports above the falling interface. The first of these samples should be extracted immediately after the interface has fallen sufficiently below the uppermost port to allow extraction without disturbing the slurry below the interface. This sample can usually be extracted within a few hours after initiation of the test, depending on the initial slurry concentration and the spacing of ports. Record the time of extraction and port depth below the surface for each port sample taken. As the interface continues to fall, extract samples from all ports above the interface at regular time intervals. As an alternative, samples can be taken above the interface at the desired depths using a pipette or syringe and tubing. As before, a suggested sequence of sampling intervals would be 1, 2, 4, 6, 12, 24, 48, 96 hr, etc. The samples should continue to be taken until the suspended solids concentration of the extracted samples shows no decrease. For this case, the suspended solids concentrations in the samples should be less than 1 g/ℓ, and filtration will be required to determine the concentrations. Tabulate the data, and plot a concentration profile diagram as shown in Figure A2. In computing the percentages remaining R for this case, the concentration of the first port sample taken above the falling interface is considered the initial concentration SS_o. Examples are shown in Appendix D.

Zone Settling Test

10. The zone settling test consists of placing a slurry in a sedimentation column, and measuring the height of the liquid-solids interface at various times. These data are plotted as depth-to-interface versus time. The slope of the constant settling velocity (straight-line) portion of the curve is the zone settling velocity, which is a function of the initial slurry concentration. A series of these tests is required if the material exhibits an

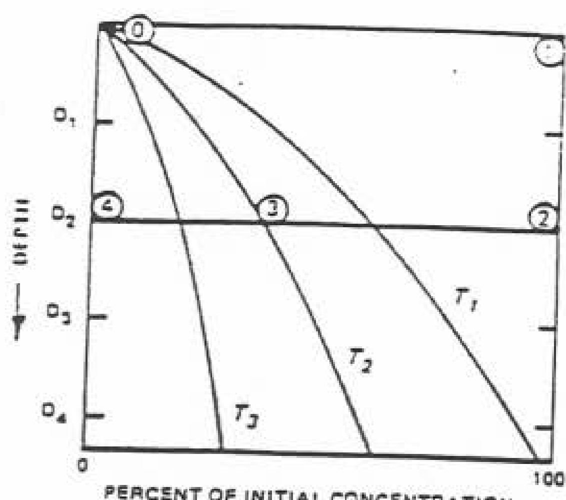


Figure A2. Conceptual concentration profile diagram

interface within the first day. The range of slurry concentrations used in the series should vary from a low of approximately 50 g/ℓ to a high concentration at which the slurry exhibits compression settling (determined by the pilot settling test) immediately.

11. Information required to design a containment area in which zone settling occurs can be obtained by using the following procedure:

- a. Use a settling column such as the one shown in Figure 1. It is important that the column diameter be sufficient to reduce the "wall effect," and that the test be performed with a test slurry depth near that expected in the field. Therefore, a 1-ℓ graduated cylinder should never be used to perform a zone settling test for sediment slurries representing dredged material.
- b. Mix the slurry to the desired concentration and pump or pour it into the test column. Air may not be necessary to keep the slurry mixed if the filling time is less than 1 min.
- c. Record the depth to the solid-liquid interface as a function of time. Measurements must be taken at regular intervals to gain data for plotting the depth-to-interface versus time curve as shown in Figure A1. It is important to take enough measurements to clearly define this curve for each test.
- d. Continue the measurements until sufficient data are available to define the maximum point of curvature of the curve which plots depth-to-interface versus time for each test. The tests may require from 1 to 3 days to complete.

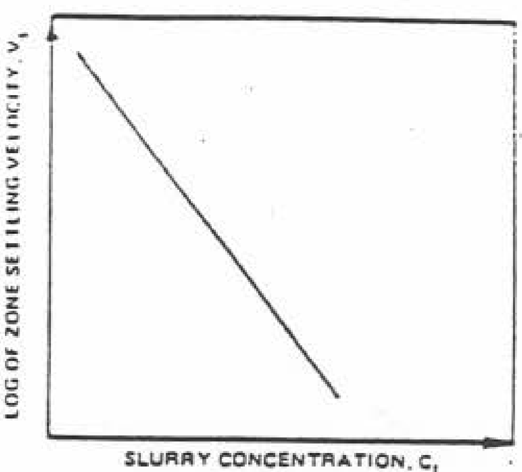


Figure A3. Conceptual plot of zone settling velocity versus concentration

e. Perform a minimum of four tests. Data from these tests are required to develop the curve of zone settling velocity versus concentration, as shown in Figure A3. Examples are shown in Appendix D.

Compression Settling Test

12. A compression settling test must be run to obtain data for estimating the volume required for initial storage of the dredged material. For slurries exhibiting zone settling, the compression settling data can be obtained from one of the series of zone settling tests, in which the depth of the interface versus time is recorded. The only difference is that the test is continued for a period of 15 days so that a relationship of concentration versus time in the compression settling range is obtained, as shown in Figure A4.

13. For slurries exhibiting flocculent settling behavior, the test used to obtain flocculent settling data can be used for the compression settling test if an interface is formed after the first few days of the test. If not, an additional test is required, with the initial slurry concentration for the test sufficiently high to initially induce compression settling. This concentration can be determined by the pilot test.

14. The following steps are used to develop the curve of concentration versus time:

a. Tabulate the interface depth H for various times of observation during the 15-day test period.

b. Calculate concentrations for various interface heights as follows:

$$C = \frac{C_i H_i}{H}$$

where

C = slurry concentration at time T, g/ℓ

C_i = initial slurry concentration, g/ℓ

H_i = initial slurry height, ft

H = height of interface at time T, ft

This assumes zero solids concentration in the water above the interface to simplify calculations.

c. Plot concentration versus time on log-log paper as shown in Figure A4.

d. Draw a straight line through the data points. This line should be drawn through the points representing the compression settling or consolidation zone, as shown in Figure A4.

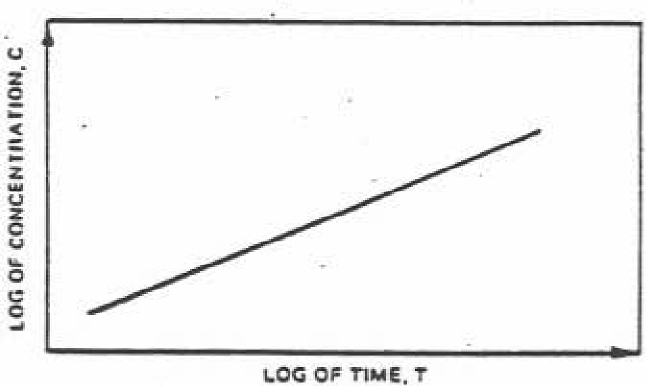


Figure A4. Conceptual time-versus-concentration plot

Attachment F-4

Sediment Sampling Log

SEDIMENT SAMPLING LOG

Client ____________
Site ____________

Project No. ____________
Sampling Personnel ____________
Date ____________
Time ____________
Weather ____________

I. Sample Location

____

____

____

II. River/Stream Sediment Sampling Information

Water Depth ____________ (in.)

Measured Sediment Depth ____________ (in.)

Full Depth of Penetration ____________ (in.)

Recovered Sediment Depth ____________ (in.)

III. Sample Sectioning Information

Section Number	Representative Length of Section	Actual Length of Section

IV. Sediment Characteristics

Color ____________

Material ____________

Grain Size ____________

Odor ____________

Remarks:

Appendix G

Light Non-Aqueous Phase Liquid (LNAPL)/Dense Non-Aqueous Phase Liquid (DNAPL) Sampling Procedures

Light Non-Aqueous Phase Liquid (LNAPL)/Dense Non-Aqueous Phase Liquid (DNAPL) Sampling Procedures

I. Introduction

Light non-aqueous phase liquid (LNAPL) and dense non-aqueous phase liquid (DNAPL) samples may be collected to facilitate laboratory characterization of these materials. Standard procedures for collecting LNAPL and DNAPL samples are presented in this Appendix.

II. Materials

The following materials will be available, as required, during LNAPL/DNAPL sampling:

- Photoionization detector (PID);
- Health and safety equipment (as required in the Health and Safety Plan);
- Cleaning Equipment (as required in Appendix W);
- Plastic sheeting;
- Field book or appropriate log forms;
- Absorbent pads;
- Peristaltic pump and pump tubing or bailer (stainless steel or Teflon7);
- Non-absorbent cord (polypropylene);
- Sample containers provided by laboratory;
- Insulated coolers, ice, and appropriate packing material;
- Resealable type bags;
- Sample labels, and chain-of-custody (COC) forms;
- Large heavy-duty garbage bags;
- Teflon7 tubing;
- Oil/water interface probe; and
- Monitoring well keys (if required).

III. Procedures

Step 1 - Review checklist and verify that the appropriate equipment has been assembled.

Step 2 - Open well and perform water level/oil thickness measurement procedures in accordance with Appendix Q.

Step 3 - Identify site and well location on sampling log sheets along with date, arrival time, and weather conditions. Identify the personnel and equipment utilized as well as other pertinent data requested on the logs (Attachment G-1).

Step 4 - Label all sample containers with date, time, well number, site location, and sampling personnel present.

Step 5 - Don a new pair of disposable gloves as required. These gloves will be used for the entire sampling event and are well specific.

Step 6 - LNAPL is to be sampled utilizing a Teflon7 or stainless steel bailer decontaminated in accordance with Appendix W. Alternatively, it may be removed utilizing a peristaltic pump with new Teflon7 tubing. If using a bailer, slowly lower bailer into the LNAPL layer, and then slowly retrieve the bailer to minimize disturbances to the NAPL layer. If using a peristaltic pump, slowly lower the tubing into the LNAPL layer and begin pumping. When finished, slowly remove tubing from the well.

Step 7 - DNAPL is to be sampled utilizing a peristaltic pump with new Teflon7 tubing or a Teflon7 bailer. The tubing should be slowly lowered through the overlying water column and into the DNAPL layer. When finished sampling, slowly remove tubing from the well to minimize disturbances to the NAPL layer. If the DNAPL lies below the effective depth at which a peristaltic pump can draw liquid, a weighted Teflon7 bailer or alternative pumping method (e.g., down-hole pump) should be used to collect the sample.

Step 8 - Obtain the LNAPL and or DNAPL sample needed for analysis with the pump or bailer and pour or pump the liquid directly from the sampling device into the appropriate container with proper label affixed and tightly screw on the cap.

Step 9 - Note the time on the sample label and sampling log.

Step 10 - Replace well cap and secure well.

Step 11 - Clean all sampling equipment in accordance with Appendix W or dispose of equipment (see Section IV below).

Step 12 - Collect all PPE and other wastes generated for disposal (see Section IV below).

Step 13 - Record required information on the appropriate forms and/or field notebook.

Step 14 - Handle, pack, and ship the samples in accordance with the procedures in Appendix L. LNAPL/DNAPL may require additional packaging and labeling procedures as specified in Appendix M.

IV. Disposal Methods

Waste materials generated during LNAPL/DNAPL sampling activities, including disposable equipment, will be disposed of in appropriate containers. Containerized waste will be disposed of by GE consistent with its on-going disposal practices.

Attachment G-1

LNAPL/DNAPL Sampling Field Log

ATTACHMENT G-1

Example

LNAPL/DNAPL Sampling Field Log

Project: ________________________________ Project No. ______________________________

Site Name: ________________________________ Sampling Personnel: ______________________________

____

____

____

Well No.: _________________________________ Date: __________________________

Time: __________________________

HNU/PID Reading:_______________________ Background __________Well

Weather: ____

I. WELL INFORMATION

Reference Point Marked on Casing: Y N
Length of inner casing: ____ above, below grade

Well Diameter: ______ TIC ______ TOC
Length of outer casing: _____ above, below grade

Well Depth
LNAPL Thickness - Vol. LNAPL Removed ________
DNAPL Thickness - Vol. LNAPL Removed ________
Water Thickness -

II. WELL SAMPLING

Lab Sample No.	Time Sampled	Material Sampled

III. MISC. OBSERVATIONS

____

____

____

____

Appendix H

Biota Sampling and Analysis Procedures

Biota Sampling and Analysis Procedures

This Appendix contains a series of attachments setting forth the standard operating procedures (SOPs) for certain types of biota sampling and analysis that may be conducted at the GE-Pittsfield/Housatonic River Site. The SOPs presented in Attachments H-1 through H-10 relate to biota sampling and analysis in Massachusetts. The SOPs presented in Attachments H-11 through H-14, prepared by the Academy of Natural Sciences of Drexel University (ANS) (formerly the Academy of Natural Sciences of Philadelphia) present procedures for collection and analysis of biota samples from the Connecticut portion of the Housatonic River. If the General Electric Company (GE) proposes to uses alternate procedures, or for other types of biota sampling (if appropriate), the sampling and analysis procedures will be presented in the project-specific work plan proposing such sampling or in other documents relating to such investigations.

Specifically, this appendix contains the following attachments:

- Attachment H-1 provides the SOP for the collection and processing of samples of aquatic vertebrates (fish and bullfrogs) for chemical analysis of their tissue.
- Attachment H-2 provides the SOP for the aging of fish samples to aid data interpretation.
- Attachment H-3a provides the SOP for macroinvertebrate community sampling using kick nets.
- Attachment H-3b provides the SOP for macroinvertebrate community sampling using a ponar dredge.
- Attachment H-3c provides the laboratory SOP from Test America for particle size analysis.
- Attachment H-4 provides the SOP for the collection of macroinvertebrate tissue.
- Attachment H-5 provides the SOP associated with benthic community assessment and laboratory taxonomic identification.
- Attachment H-6 provides the SOP for the performance of caged bivalve bioaccumulation studies.
- Attachment H-7 provides the SOP for the preparation and homogenization of biota tissue samples for chemical analysis.
- Attachment H-8 provides the SOP for the extraction and cleanup of PCBs from fish and biota material.

- Attachment H-9 provides the SOP for congener-specific PCB analysis using the Green Bay method.

- Attachment H-10 provides the SOP for the extraction and analysis of lipids from fish and biota material.

- Attachment H-11 provides the ANS SOP for presentation, fixation, and curation of fish samples from Connecticut.

- Attachment H-12 provides the ANS SOP for the preparation of fish samples from Connecticut for chemical analysis.

- Attachment H-13 provides the ANS SOP for the extraction and cleanup of fish tissue samples from Connecticut for PCB and pesticide analysis

- Attachment H-14 provides the ANS SOP for the quantification of individual PCB congeners, chlorinated pesticides, and individual compounds by capillary column gas chromatography in biota samples from Connecticut.

Attachment H-1

Sampling of Aquatic Vertebrates (Fish and Bullfrogs) for Tissue Analysis

Attachment H-1: Sampling of Aquatic Vertebrates (Fish and Bullfrogs) for Tissue Analysis

This attachment describes the methodologies to be used for the collection, storage, and preparation of aquatic vertebrate samples (i.e., fish and bullfrogs) for tissue analysis, unless otherwise provided in the project-specific work plan.

A. Materials

The following collection equipment and materials will be available, as required, during aquatic vertebrate sampling:

- Health and safety equipment (as required in the Health and Safety Plan [HASP]);
- Electrofishing equipment;
- Seine and sport fishing equipment;
- Chest waders;
- Floy gun and tags;
- Net;
- Heavy-duty aluminum foil;
- Coolers with ice or “blue” ice;
- Boat; and
- Appropriate forms/field notebook.

B. Collection Procedures

The following procedures will be used to obtain and handle fish and frog samples in the field.

1. The biota collection sites, species, and number of samples will be specified in the applicable work plan.
2. Fish will primarily be collected by using electrofishing methods. Where necessary, seining, netting, and/or methods may also be used.

3. Bullfrogs will be collected using angling methods.

4. All samples must remain whole and ungutted.

5. For each sample, the following information will be recorded in a field notebook:

   - Floy tag number - collection date;
   - total length for fish; total length of legs, fully extended, for frogs;
   - weight;
   - species;
   - sex (if known);
   - sample location;
   - other distinguishing features; and
   - sampler(s).

   Any unusual physical irregularities should also be noted.

6. If samples are required to be composited under the project-specific work plan, the number of individual organisms specified in that plan for each composite will be collected and composited. In the absence of such specification, a sufficient number of organisms will be included in the composite sample to meet minimum mass requirements for chemical analysis. Composite samples will be measured and weighed following project-specific requirements, but will generally include length measurements of the smallest and largest individual in the sample, and the total weight measurement of the entire sample.

7. Measurements of environmental contaminants in biota typically exhibit a wide range of variability due to the natural variation of the target population. This variability may be sufficiently great to limit the ability of the sampling study to determine differences among collection sites (i.e., reaches of the river). In order to minimize this variability, specimens of comparable length, body weight, and age will be collected whenever possible, except as otherwise provided in the project-specific work plan.

8. Each sample (or composite sample) will be wrapped individually in heavy-duty aluminum foil. The individually wrapped samples should be stored in freezer bags and sorted by sampling site. All specimens shall be stored on ice during transportation. Samples shall be packaged and labeled following the procedures presented in Appendix L.

C. Handling and Processing

Once the fish and/or frog samples have been transported to the analytical laboratory, the following procedures (or comparable procedures developed by the laboratory) apply:

1. Storage

   a. Samples should be stored by site on freezer shelves. Samples should be frozen within 24 hours from the time of receipt by the laboratory. Keeping the biota samples on ice in the field and during transportation, followed by freezing at the laboratory, should minimize any internal organ deterioration and the potential leaching of body fluids of those organs into the muscle tissue of the fillets.

2. Preparing to Grind

   a. Samples shall be thawed at room temperature for 24 hours prior to compositing. Fish and/or frog samples should be arranged by site, then species by increasing length.

   b. Prepare sample bottles – label year and sequence number on tape and wrap around each sample bottle. Consult the sample tags and/or chain-of-custody (COC) forms for specific parameters to be run.

3. Processing the Specimen Samples: All samples will be processed and prepared for analysis using the laboratory's standard operating procedure for preparation and homogenization of the relevant type of biota tissue samples, or as otherwise specified in the project-specific work plan.

Attachment H-2

Fish Aging Procedure

Attachment H-2: Fish Aging Procedure

If aging of fish samples is completed to aid in data interpretation, fish will be aged from scales collected generally from the region of the body immediately below the dorsal fin. The procedure to be utilized for fish aging is described below.

A. Field Collection and Handling of Scales

1. Prior to removal of the scales, mucus and dirt are to be removed from the fish by scraping the sampling area in the direction of the caudal fin with the back of a knife or edge of forceps.

2. Scales are to be removed from the location indicated above with a knife or forceps. The scales are then inserted into a coin envelope. Several scales will be removed from each fish. Scales from each fish are deposited in a single envelope.

3. Each sample envelope will be labeled with the collection date and sample number.

4. The instruments used to remove scales should be cleaned between samples to avoid cross-contamination of scale samples.

B. Preparation of Scale Samples for Reading

1. Fish can be aged from scales either by direct reading of the raw scales or from scale impressions if the raw scales are too thick, dirty, or pigmented to transmit sufficient light to facilitate interpretation.

2. Mounts of raw scales are prepared by mounting several scales, sculpted side up, between two glass slides. The slides are bound together with tape and labeled with the sample number and species of fish.

3. Regenerated scales should not be mounted.

4. Scale impressions can be made be impressing the sculptured side of several scales from a single sample into plastic or cellulose slides, or laminated polyethylene/vinyl material using a roller press. These impressions should also be permanently labeled with the sample number and species of the fish.

C. Aging Scales

1. Scales should be aged using a microprojector, with the sculpted surface of the scale or impression facing the light source.

2. Regenerated scales should not be aged.

3. Each age should be based on the reading of at least two scales, both aged by independent observers, and a consensus reached by consultation if different results occur.

4. Age should be reported as the number of annuli in Roman numerals.

Attachment H-3a

Macroinvertebrate Community Sampling Using Kick Nets

Attachment H-3a – Macroinvertebrate Community Sampling Using Kick Nets

I. Scope and Application

This document describes the general methodology that will be used in the field to collect macroinvertebrate community samples using a kick net.

II. Personnel Qualifications

Personnel conducting the macroinvertebrate sampling will be familiar with invertebrate sampling procedures.

III. Equipment List

Staff assigned the responsibility of collecting macroinvertebrate samples will be provided with the following information:

- Work documents (field sampling plan, health and safety plan, etc.)
- Water body name and site maps
- Number of samples to be collected
- Collecting and processing procedures
- Special instructions (if any)
- Sampling permits and licenses (if applicable)

The following collection equipment and materials will be available, as required, during benthic macroinvertebrate sampling:

- Personal protective equipment (as required by the health and safety plan)
- 9” by 18” rectangular dip net with 500-micron mesh
- 1-m square pipe (or equivalent) quadrat
- Mesh or sieve screens with 0.500 mm openings (U.S. Standard no. 35)
- Cooler(s)
- Bucket
- Sample jars, vials, and preservative (70% or greater alcohol solution)
- Forceps and magnifying glass

- Cleaning and decontamination materials
- Indelible ink markers
- Camera
- Hand-held GPS
- Physical characterization/water quality field data sheet
- Field notebook

IV. Cautions

Cautions include typical hazards associated with working around water (e.g., drowning, falling on slippery surfaces, etc.).

V. Health and Safety Considerations

Personnel will take proper precautions when working around water.

Personnel will use appropriate lifting techniques.

Personnel will work using the buddy system for safety.

VI. Procedure

Benthic macroinvertebrate samples will be collected using a kick net from riffle/run habitat as described below.

A. Collection

The following procedures describe the use of a kick net to collect invertebrate samples and are based on the protocols found in the US Environmental Protection Agency's (EPA's) Rapid Bioassessment Protocols (Barbour et al., 1999) and the 2000 Aquatic Habitat Assessment for the 1½ Mile Reach (Woodlot, 2000):

1. The field crew will don health and safety equipment (e.g., waders, gloves, etc.).

2. The field crew will identify the proposed sample location using GPS or topographic landmarks.

3. At each of the pre-determined sample locations, an area will be selected which includes riffle or run habitat.

4. Water quality data (temperature, dissolved oxygen, pH, and specific conductance) will be collected within one meter of the substrate surface once at each of the sample locations. Water depth, substrate type, and stream characteristics will be recorded at each location where a kick-net sample is collected (12 at each of the three sample locations).

5. Samples will be collected by holding the net stationary on the substrate starting at the downstream end of the riffle while disturbing (kicking or hand rolling and cleaning rocks as necessary) a one-meter square area defined by a quadrant immediately upstream of the net for approximately two minutes.

6. Upon completion of the two minutes of sampling, the net will be removed from the water column, allowed to drain free of water, and inspected for the presence of macroinvertebrates. Large debris (leaves, sticks, rocks, etc.) will be removed from the net and the remaining contents will be transferred to a labeled sample jar and preserved using at least 70 percent isopropyl alcohol.

7. This process will be repeated by moving upstream across the riffle in a zigzag pattern if possible until 12 samples are collected from 12 separate locations per each transect. The field leader may elect a different sampling pattern if necessitated by field conditions. The process will then be repeated at each of the remaining sample transects.

8. Each sample will be labeled with sampling date and collection location and will be counted to ensure that the correct number of samples has been taken.

In addition to the collection of these benthic macroinvertebrate samples, if the project-specific work plan so provides, sediment samples associated with the benthic community samples will be collected for analysis of grain size and total organic carbon (TOC). These samples will be collected in accordance with the sediment sampling procedures described in Appendix F of this Field Sampling Plan/Quality Assurance Project Plan or as otherwise provided in the project-specific work plan. These samples will be analyzed for grain size by ASTM Standard Method D422-63 (2007), using the laboratory SOP provided in Attachment H-3c unless otherwise provided in the project-specific work plan. Samples will be analyzed for TOC by the Lloyd Kahn Method, as incorporated in the SOP provided in Appendix HH, unless otherwise provided in the project-specific work plan.

B. Handling, Packing, and Shipping

The following identifies the procedures that will be used to handle, pack, and ship the benthic macroinvertebrate samples:

1. All samples will be given a sample identification number that will be recorded in the field notebook and that will correspond to the sample analysis, sampling date, and collection location.

2. Isopropyl alcohol is a DOT/IATA regulated material so a shipping determination will be performed by trained staff to determine the appropriate shipping procedure.

3. Samples containing isopropyl alcohol will either be packaged and shipped under the applicable DOT hazardous materials shipping protocol (not described in this document), or the samples will be drained of isopropyl alcohol prior to being offered for shipment and if drained, will be packed and shipped as described below.

   - A piece of 500-micron mesh will be cut and placed over the top of the sample jar containing the isopropyl alcohol sample mixture.

   - The jar will be inverted over a decon bucket and the free liquid will be allowed to slowly drain out of the sample jar into the bucket. Care will be taken to ensure that no organisms are lost during the draining.

   - After the isopropyl mixture has drained completely, turn the sample container right side up, put the piece of mesh used to strain the mixture in the jar (to ensure no organisms caught in the mesh will be lost) and tighten the lid.

4. Sample jars will be inspected to make sure that labeling is correct and that the sample containers are intact. Sample jars will be tightened and taped, if necessary.

5. Chain-of-custody forms, custody seals, address labels, and air-bill forms will be initiated. A copy of the completed chain-of-custody form and air-bill form will be retained by the sampler.

6. Coolers used for transport will be duct-taped at the drain plug on the outside and inside of the cooler.

7. Benthic macroinvertebrate samples will be placed upright in the bottom of the coolers with cushioning materials placed on top and in between samples (if applicable).

8. The completed chain-of-custody form will be placed into a plastic bag and duct-taped to the inside of the cooler lid.

9. The cooler will be closed and fastened with duct tape around the seam of the lid to prevent water leakage and with strapping tape around the entire cooler to prevent it from opening during transport.

10. A completed custody seal will be placed across the seam of the cooler lid. A completed address label will be placed on top of the cooler. Both will be taped-over using clear packing tape.

11. Samples will be shipped to the laboratory, and the laboratory will be notified of the shipment and will be contacted immediately following the arrival date to ensure that delivery has occurred.

VII. Waste Management

Project-derived waste will be disposed of in accordance with project requirements.

VIII. Data Recording and Management

Field notes will be recorded during sampling activities, and at a minimum, will include the following:

- Names of field crew and oversight personnel
- Sample location (GPS if specified)
- Date, time, and duration of sampling
- General weather conditions
- Substrate characterization
- General water quality parameters
- Sample information (including matrix, sampling method, sample mass, sample ID, sample date and time)
- Habitat description
- General observations of benthic macroinvertebrate abundance and diversity
- Photograph number when pictures are taken (if necessary)

The Macroinvertebrate Sampling Data Sheet (attached) will be completed.

IX. Quality Assurance

The macroinvertebrate sampling will be conducted consistent with the procedures outlined herein. Deviations from the plan will be discussed with the project manager prior to changing any field procedures.

X. References

Barbour, M.T., J. Gerritsen, B.D. Snyder, J.B. Stribling. 1999. *Rapid Bioassessment Protocols For Use in Streams and Wadeable Rivers: Periphyton, Benthic Macroinvertebrates, and Fish, Second Edition*. EPA 841-B-99-002. Prepared for the U.S. Environmental Protection Agency; Office of Water, Washington, D.C.; 7: 3-5.

Woodlot Alternatives, Inc. (Woodlot). 2000. *Aquatic Habitat Assessment: 1½ Mile Reach – GE-Pittsfield/Housatonic River Site, Pittsfield, MA*. September 2000.

Attachment H-3b

Benthic Community Sampling
Using Ponar Grab Sampler

Attachment H-3b: Benthic Community Sampling Using Ponar Grab Sampler

I. Introduction

The following procedures describe the general methodologies that will be used in the field to sample the benthic macroinvertebrate community using a ponar grab sampler. Prior to mobilizing in the field, staff assigned the responsibility of collecting benthic macroinvertebrate samples will be provided with the following information:

- Work documents (Field Sampling Plan, Health and Safety Plan [HASP], etc.);
- Water body name and site maps;
- Number of samples to be collected;
- Collecting and processing procedures;
- Special instructions (if any);
- Appropriate fisheries office contact; and
- Sampling permits and licenses.

II. Sampling Procedures

<u>Equipment</u>

The following collection equipment and materials will be available, as required, during benthic macroinvertebrate sampling:

- Personal protective equipment (PPE), as required by the HASP;
- Boat, engine, life jackets, anchors, buoys, and rigging;
- Water quality meter(s) to monitor temperature, pH, specific conductivity, turbidity, dissolved oxygen, and water velocity;
- Grab sampler with 6” by 6” sample area;
- Mesh or sieve screen (U.S. Standard No. 35);
- Sample jars, vials, and preservative (70% alcohol solution);
- Forceps and magnifying glass;
- Cleaning and decontamination materials;
- Insulated coolers;
- Tape (duct, strapping, and clear packing);

- Plastic sealable bags and indelible ink markers;
- Camera;
- Global Positioning System (GPS);
- Physical characterization/water quality field data sheet;
- Forms (chain-of-custody, custody seal, address label, and air-bill); and
- Field notebook.

Procedures

Benthic macroinvertebrate community samples will generally be collected using a sediment grab sampler and approved sampling techniques. The collection methods to be employed during benthic community sampling using this method are presented below.

1. The field crew will identify the proposed sample location using GPS or topographic landmarks, and will anchor the boat securely so that it will not drift due to water or wind currents.

2. Water quality data (temperature, dissolved oxygen, pH, specific conductance, turbidity, and water velocity) will be collected within 1 meter of the substrate surface. If sample locations are close together, these data will be recorded once for each general area.

3. At each sample location, the opened grab sampler will be lowered over the side of the boat and allowed to settle into bottom sediments. A hard pull on the rope will close the sediments inside the grab sampler. Care should be taken to avoid areas of significant debris (e.g., trash, woody detritus) that may diminish the ability to close the sampler jaws resulting in poor or non-representative recovery.

4. Retrieve the grab sampler into the boat and, where practicable, empty the grab sampler contents directly into a sieve. Where necessary due to excessive material recovery volumes, the sampler may be emptied into a larger receptacle (e.g., bucket, stainless steel bowl), with collected materials subsequently placed in the sieve in smaller subsamples. Prior to sieving, the recovered materials should be visually inspected for representativeness (i.e., similarity to other samples collected from the same location) and rejected if they are of insufficient volume or are otherwise not comparable to other samples from the same general area.

5. Sieve the benthic samples to isolate the benthic organisms. Hand transfer organisms and sediment matrix from the sieve to a labeled sample jar and preserve in the field using 70% isopropyl alcohol.

6. Repeat this process until the desired number of benthic samples per location is collected. Care will be taken so that successive grab sampler sampling does not reoccur over previous sampled areas.

In addition to the collection of these benthic macroinvertebrate samples, if the project-specific work plan so provides, sediment samples associated with the benthic community samples will be collected for analysis of grain size and total organic carbon (TOC). These samples will be collected in accordance with the sediment sampling procedures described in Appendix F of this Field Sampling Plan/Quality Assurance Project Plan or as otherwise provided in the project-specific work plan. These samples will be analyzed for grain size by ASTM Standard Method D422-63 (2007), using the laboratory SOP provided in Attachment H-3c unless otherwise provided in the project-specific work plan. Samples will be analyzed for TOC by the Lloyd Kahn Method, as incorporated in the SOP provided in Appendix HH, unless otherwise provided in the project-specific work plan.

Documentation

Field notes will be recorded during sampling activities, and at a minimum, will include the following:

- Names of field crew and oversight personnel;
- General weather conditions;
- Date, time, and sample location (GPS if specified);
- Sampling technique and duration;
- General observations of benthic abundance and diversity;
- Substrate characterization and water quality; and
- Photograph number when pictures are taken (if necessary).

The Macroinvertebrate Sampling Data Sheet (attached) will be completed.

III. Sample Processing, Packaging, and Shipping

The following procedures describe the general methodologies that will be used in the field to process (handle, preserve, pack, and ship) the benthic macroinvertebrate samples for laboratory analysis.

Handling

1. Benthic organisms, and the remaining sediment matrix that is isolated after sieving, will be preserved in the field using 70% isopropyl alcohol.

2. Each sample will be labeled with sampling date and collection location, and will be counted to ensure that the correct number of samples has been taken. All samples will be given a sample identification number that will be recorded in the field notebook, and will correspond to the sample analysis, sampling date, and collection location.

3. Samples will be inspected to make sure that labeling is correct and that the sample containers are intact. Benthic community sample jars will be tightened and taped, if necessary.

4. Chain-of-custody forms, custody seals, address labels, and airbill forms will be initiated. A copy of the completed chain-of-custody form and air-bill form will be retained by the sampler.

Packing

1. Coolers used for transport will be duct-taped at the drain plug on the outside and inside of the cooler.

2. Benthic macroinvertebrate samples will be placed upright in the bottom of separate coolers with cushioning materials placed on top.

3. The completed chain-of-custody form will be placed into a plastic bag and duct-taped to the inside of the cooler lid.

4. The cooler will be closed and fastened with duct tape around the seam of the lid to prevent water leakage and with strapping tape around the entire cooler to prevent it from opening during transport.

5. A completed custody seal will be placed across the seam of the cooler lid. A completed address label will be placed on top of the cooler. Both will be taped-over using clear packing tape.

6. An airbill (if appropriate) with shipper's and consignee's addresses will be affixed to top of cooler.

Shipping

1. Samples will be shipped to the laboratory by hand or by express carrier in a timely manner.

2. The laboratory will be notified of the shipment and will be contacted immediately following the arrival date to ensure that delivery has occurred.

IV. Equipment Decontamination

Reusable equipment that comes into contact with biota tissues will be cleaned prior to use and between samples using the following procedure:

1. Potable water rinse and wash with a detergent solution (i.e., Alconox).

2. Potable water rinse.

Materials generated during sampling activities (e.g., disposable gloves and other disposable equipment) will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its ongoing disposal practices.

V. Chain-of-Custody Procedures

All samples will be collected and handled in accordance with the chain-of-custody procedures summarized below:

1. Prior to relinquishing samples for packaging and shipment, one member of the sampling team will record relevant information (e.g., sample identification, instructions for sample processing, and/or chemical analyses) on a chain-of-custody form.

2. The samples will be packaged for shipment as described in Section III above.

Attachment H-3c

Laboratory SOP: Particle Size Analysis (provided by TestAmerica)

Title: Particle Size Analysis (ASTM D 2217 and D422-63)

Approval Signatures:

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SOP REVIEW FORM

SOP Number	Revision	Effective Date:	Title
GR-GT-006	5	03/17/10	Particle Size Analysis

Review Statement:

My signature signifies that I reviewed and compared the above referenced SOP against current bench practice.

Date	Reviewer	Job Title	Revision Needed	
			Yes[1]	No
04/03/12	Christopher Calldn	Dept Manager		X
04/03/12	Eric [illegible]	Analyst		X

[1:] *List the section for revision and the the reason using the revision summary page and attach to this cover sheet.*

QA Use Only:

X The SOP was reviewed and does not require revision. Attach this form to the SOP.

____ The SOP Revision will be made with a Change in Progress Attachment (CIPA).

____ The SOP Revision will be released as a new version of the SOP.

____ The SOP Revision requires method validation or demonstration of capability

____ The SOP Revision does not reqire method validation or demonatration of capability.

____ The SOP revison affects other SOPs that must now also be revised (List SOPs)

Kathleen Daigle
QA Signature

4/3/2012
Date

Comments:

BR-FQA023:12.22.11:0
TestAmerica Burlington

1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of particle size distribution in soils.

2.0 Summary of Method

A portion of sample is soaked in a dispersing agent then partitioned into separate portions, material retained on a #10 sieve and material passing the #10 sieve. The material retained on the #10 sieve is dried to constant weight then passed through a large size sieve stack; the material retained on each sieve is measured and recorded. Material passing the #10 sieve is subject to hydrometer analysis then passed through a small size sieve stack, the material retained on each sieve is measured and recorded. All measurements, large and small sieves and hydrometer readings and the hygroscopic moisture are used to establish the particle size distribution of the sample.

This SOP is based on the following reference methods:

- ASTM Standard D 2217 – 85 (Rapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

NOTE: ASTM D2217 was method was withdrawn without replacement by ASTM in 2007. A withdrawn standard is an ASTM standard that has been discontinued by the ASTM Sponsoring Committee responsible for the standard.

If the laboratory has modified the procedure from the reference method(s) a list of modifications will be provided in Section 16.0.

3.0 Definitions

Not Applicable

4.0 Interferences

Not Applicable

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Not Applicable

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Top-Loading Balance, capable of weight measurement to 0.01 g
- Mechanical Stirring Device and Dispersion Cup
- Thermometer: Accurate to 0.5°C
- Mortar and Rubber Tipped Pestle
- Sedimentation Cylinder(s) 1000 mL
- Hydrometer: ASTM 151H in specification E 100.
- Sieves, of the following size(s): Gilson Company, Inc. or equivalent
  3.0” (75.00 mm)
  2.0” (50.00 mm)
  1.5” (37.50 mm)
  1.0” (25.00 mm)
  3/4" (19.00 mm)
  3/8” (9.50 mm)
  # 4 (4.75 mm)
  #10 (2.00 mm)
  #20 (850.0 um)
  #40 (425 um)
  #60 (250.0 um)
  #80 (180.0 um)
  #100 (150.0 um)
  #200 (75.0 um)
- Drying Oven with temperature range of 60-110°C
- Stainless Steel Spatulas & Spoons
- Metal & Bristle Brushes
- Ro-Tap Sieve Shaker, W. S. Tyler or equivalent.
- Timing Device with second hand and capable of counting up to 25 hours

7.0 Reagents and Standards

- Reverse Osmosis (RO) water: In-House System
- Sodium Hexametaphosphate: ELE International or equivalent.

Sodium Hexametaphosphate Solution: Add 120 g of sodium hexametaphosphate and 2940 g of reagent water to a 1-gallon bottle. Add a stir rod to the container and place on a stir plate. Mix the solution until it is homogeneous. Assign an expiration date of 30 days from the date made

unless the parent reagent expires sooner in which case use the earliest expiration date. Store the prepared solution at ambient temperature.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Solid	Glass Jar w/ Teflon Lid	500 g	None	None	ASTM D422-63

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Equipment Calibration

Check the calibration of the balance on each day of use prior to use using at least 2 Class S weights that bracket the range of use. Record in the logbook designated for this purpose.

Check the temperature of the drying oven(s) each day of use, prior to use. Record in the logbook designated for this purpose.

NOTE: The QA Manager or her designee checks the calibration of liquid in glass thermometers annually against a NIST-traceable thermometer following the procedures given in laboratory SOP BR-QA-004. Electronic / digital thermometers that are battery-operated are checked quarterly using the same procedure.

Calibrate the hydrometers every two years following the procedure given in BR-GT-008.

Calibrate the sieves 6 months following the procedure given in BR-GT-008.

Calibrate the Ro-Tap sieve shaker every 12 months following the procedure given in BR-GT-008.

10.2 Hygroscopic Moisture Determination

Label an aluminum pan with the Lab ID for each sample. Tare the balance, weigh each pan and record the weight measurement in the spreadsheet.

Mix the sample with a stainless steel spatula. Measure at least 10-15 g of each sample into the labeled aluminum pan and record the weight of sample in the spreadsheet.

Place the pan + sample in an oven maintained at a temperature of 110ºC and dry the sample for at least 16 hours. Reweigh each pan and record the weight measurement in the spreadsheet.

Percent solids are calculated using the equation given in Section 11.0.

10.3 Sample Preparation

Use the calculated percent solids and the sample characteristic for each sample to determine the amount needed for analysis using Table 2. For example, if the calculated percent solids for a sample are 50% and the sample characteristic is sand, use 200 g for analysis. If there is an insufficient amount of sample available, initiate a nonconformance memo (NCM) and contact the PM for further instruction.

Place a 1000 mL plastic beaker on the balance and tare the balance. Weight the amount of sample for analysis and record the weight in the bench sheet.

Add 125 mL of sodium hexametaphosphate solution to each beaker. Stir to mix and soak the sample in this solution for 16 hours

10.4 Sample Partition

Rinse the sample slurry into a dispersion cup using reagent water. Fill the dispersion cup ½ full with reagent water and place the cup on the blender to mix for one minute.

NOTE: Some samples may not be amendable to using the blender examples include but not limited to large gravel, sands, or organic material. If the sample is not amenable, initiate a NCM to notify the PM of the anomaly and proceed to the next step without blending the sample.

Place a #10 sieve on a 1000 mL graduated cylinder. Pour the sample through the sieve. Rinse the dispersion cup with reagent water and pour the rinse through the sieve. Repeat until transfer is complete. Bring the volume in the graduated cylinder to 1000 mL with reagent water. Cover the cylinder with a rubber stopper and equilibrate the sample to ambient temperature in preparation for hydrometer analysis.

Label a medium size aluminum dish with the sample's LAB ID then transfer the sample material that was retained on the #10 sieve to the dish. Place the aluminum dish in the drying oven set at 110 ± 5° C and dry the sample material for at least 16 hours or until constant weight. Set aside for sieve analysis.

10.5 Hydrometer Analysis

Prepare a hydrometer rinse bath by adding 1000 mL of reagent water to a 1000 mL graduated cylinder

Record the hydrometer ID and start time on the worksheet. Set the timer for the elapsed time and perform each task as listed in Table 1: Hydrometer Reading Table.

To shake the cylinder, rotate the flask up and down for one minute approximating at least 60 turns. One turn down and one turn up equals two turns.

To take a hydrometer reading, gently insert the hydrometer into the graduated cylinder and wait ~ 20 seconds. Read the hydrometer from the top of the meniscus to the nearest 0.0005. Enter the reading on the worksheet. After each reading, clean the hydrometer by twisting and dropping the hydrometer into the hydrometer rinse bath.

Insert a temperature probe into the cylinder to the same depth used for the hydrometer reading. Read the temperature to the nearest 0.5°C and enter the temperature measurement on the worksheet. Rinse the temperature probe in the hydrometer rinse bath.

Repeat the above process taking hydrometer readings every 2, 5, 15, 30, 60, 240 and 1440 minutes as per Table 1 then proceed to small sieve analysis.

10.6 Sieve Analysis

Inspect the sample material in the aluminum pan and record a description of the non-soil material (e.g.- sticks, grass, wood, plastic), hardness of material and shape of material in the worksheet.

Hardness qualifiers include hard, soft or brittle. Shape qualifiers include well rounded, rounded, subrounded, subangular, and angular.

<u>Large Sieves</u>

Weigh the 3/4", 3/8", #4 and #10 sieves and enter the weight measurements in the worksheet as the tare weight.

Stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 10 minutes.

Weigh each sieve and record these measurements in the worksheet.

<u>Small Sieves</u>

Transfer the sample from the graduated cylinder to a #200 wet wash sieve. Wash the sample through the #200 sieve until the water runs clear then transfer the material retained on the sieve to a 250 mL glass beaker labeled with the sample's LAB ID.

Place the beaker in the drying oven and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

Gently mix the dried contents of the beaker with a rubber-tipped pestle to break any soil aggregates that may have formed during the drying stage.

Tare the balance and weigh the sieve stack sized between #20 and #200 and record the tare weights.

Transfer the sample to the sieve stack and ensure complete transfer. Use hair or wire brushes to clean the beaker. Place the sieve stack on the Rotap machine and shake for ten minutes.

Weigh each sieve and record these measurements in the worksheet.

11.0 Calculations / Data Reduction

11.1 Calculations

Sample Used (SU): Dry Preparation

$$SU = (pan + dry\ sample\ \text{-}\ pan) - (pan + non\text{-}soil\ material\ \text{-}\ pan) \otimes HMCF$$

Where:
HMCF = Hygroscopic moisture correction factor.

Sieve Analysis (Percent Finer = PF)

Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Viscosity at sample temperature, poises
Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8)+10.5 and 264.5 = (10.5-2.3)/0.031
Time, minutes = Time of hydrometer reading from beginning of sedimentation
Sqrt - square root
SG - Specific Gravity of soil
Viscosity - is the resistance of a liquid to flow
Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

Where:
Constant = (100,000/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample

passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))
Intercept = (low temp. reading -1) - (low temp. * slope)

11.2 Data Reduction

11.2.1 Primary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Upload the batch information into LIMS and complete the batch editor and worksheet. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to 1st level review.

11.2.2 Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review.

11.2.3 Lab Complete

Review the batch, run QC checker as appropriate and set the status to lab complete.

11.2.4 Data Reporting

Sample results are reported from the laboratory's LIMS system using the formatter specified by the Project Manager.

11.2.5 Data Archival

Data are stored in the laboratory's LIMS system.

12.0 <u>Method Performance</u>

Not Applicable

13.0 <u>Pollution Control</u>

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide

by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)
- Liquid Waste- 55 gallon poly drum

15.0 References / Cross-References

- ASTM Standard D 2217 – 85 (Reapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

16.0 Method Modifications

D2217: The laboratory performs sample portioning after soaking the solution in the dispersing agent because the dispersion agent helps break up aggregates associated with clay and sediments.

D422: The laboratory does not always use the recommended amount of sample for analysis because sufficient sample volume is not always received.

17.0 Attachments

- Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)
- Table 2: Percent Solids Table for Weight Determination for D422.

18.0 Revision History

BR-GT-006, Revision 6:

- Title Page: Updated approval signatures
- All Sections: Removed references to dry preparation by ASTM D421; Added procedure for wet preparation.
- Attachments: Inserted Percent Solids Table

Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)

Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)	Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)
0:00	Shake	1		1:01	Read	10	5
0:01	**Place**	**1**		**1:02**	Shake	11	
0:01	**Shake**	**2**		**1:03**	Place	11	
0:02	Place	2		1:04	Read	9	15
0:03	Read	1	2	1:05	Read	11	2
0:04	Read	2	2	1:06	Read	7	31
0:06	Read	1	5	1:07	Read	3	58
0:07	Read	2	5	1:08	Read	11	5
0:08	Shake	3		**1:09**	Shake	12	
0:09	**Place**	**3**		**1:10**	Place	12	
0:09	**Shake**	**4**		1:11	Read	10	15
0:10	Place	4		1:12	Read	12	2
0:11	Read	3	2	1:13	Read	4	63
0:12	Read	4	2	1:14	Read	8	32
0:14	Read	3	5	1:15	Read	12	5
0:15	Read	4	5	1:18	Read	11	15
0:16	Read	1	15	1:19	Read	9	30
0:17	Read	2	15	1:21	Read	5	60
0:20	Shake	5		1:25	Read	12	15
0:21	Place	5		1:26	Read	10	30
0:23	Read	5	2	1:27	Read	6	59
0:24	Read	3	15	1:33	Read	11	30
0:25	Read	4	15	1:34	Read	7	59
0:26	Read	5	5	1:41	Read	12	31
0:27	Shake	6		1:42	Read	8	60
0:28	Place	6		1:52	Read	9	63
0:30	Read	6	2	1:53	Read	10	57
0:31	Read	1	30	2:06	Read	11	63
0:32	Read	2	30	2:07	Read	12	57
0:33	Read	6	5	4:17	Read	1	256
0:34	Shake	7		4:18	Read	2	256
0:35	Place	7		4:19	Read	3	250
0:36	Read	5	15	4:20	Read	4	250
0:37	Read	7	2	4:21	Read	5	240
0:38	Read	3	29	4:22	Read	6	234
0:39	Read	4	29	5:00	Read	7	265
0:40	Read	7	5	5:01	Read	8	259
0:41	Shake	8		5:02	Read	9	253
0:42	Place	8		5:03	Read	10	247
0:43	Read	6	15	5:04	Read	11	241
0:44	Read	8	2	5:05	Read	12	235
0:47	Read	8	5	24:01	Read	1	1440
0:48	Shake	9		24:02	Read	2	1440
0:49	Place	9		24:03	Read	3	1434
0:50	Read	7	15	24:04	Read	4	1434
0:51	Read	9	2	24:05	Read	5	1424
0:52	Read	5	31	24:06	Read	6	1418
0:54	Read	9	5	24:07	Read	7	1412
0:55	Shake	10		24:08	Read	8	1406

0:56	Place	10		24:09	Read	9	1400
0:57	Read	8	15	24:10	Read	10	1394
0:58	Read	10	2	24:11	Read	11	1388
0:59	Read	6	31	24:12	Read	12	1382
1:00	**Read**	**1**	**59**				
1:00	**Read**	**2**	**58**				

Source: Laboratory Prepared Reference Document

Table 2: Percent Solids Table for Weight Determination for D422.

Percent Solid Table

Quantities of sample (in grams) to be utilized in Wet method version of ASTM D854 and D422

% Sol	Spec Grav 25	Hydrometer Slt/Cl 50	Hydrometer Slt/Snd 75	Snd 100	Snd/Gr 200
1	2500	5000	7500	10000	20000
2	1250	2500	3750	5000	10000
3	833	1667	2500	3333	6667
4	625	1250	1875	2500	5000
5	500	1000	1500	2000	4000
6	417	833	1250	1667	3333
7	357	714	1071	1429	2857
8	313	625	938	1250	2500
9	278	556	833	1111	2222
10	250	500	750	1000	2000
11	227	455	682	909	1818
12	208	417	625	833	1667
13	192	385	577	769	1538
14	179	357	536	714	1429
15	167	333	500	667	1333
16	156	313	469	625	1250
17	147	294	441	588	1176
18	139	278	417	556	1111
19	132	263	395	526	1053
20	125	250	375	500	1000
21	119	238	357	476	952
22	114	227	341	455	909
23	109	217	326	435	870
24	104	208	313	417	833
25	100	200	300	400	800
26	96	192	288	385	769
27	93	185	278	370	741
28	89	179	268	357	714
29	86	172	259	345	690
30	83	167	250	333	667
31	81	161	242	323	645
32	78	156	234	313	625
33	76	152	227	303	606
34	74	147	221	294	588
35	71	143	214	286	571
36	69	139	208	278	556
37	68	135	203	270	541
38	66	132	197	263	526
39	64	128	192	256	513
40	63	125	188	250	500
41	61	122	183	244	488
42	60	119	179	238	476
43	58	116	174	233	465
44	57	114	170	227	455
45	56	111	167	222	444
46	54	109	163	217	435
47	53	106	160	213	426
48	52	104	156	208	417
49	51	102	153	204	408
50	50	100	150	200	400

% Sol	Spec Grav 25	Hydrometer Slt/Cl 50	Hydrometer Slt/Snd 75	Snd 100	Snd/Gr 200
51	49	98	147	196	392
52	48	96	144	192	385
53	47	94	142	189	377
54	46	93	139	185	370
55	45	91	136	182	364
56	45	89	134	179	357
57	44	88	132	175	351
58	43	86	129	172	345
59	42	85	127	169	339
60	42	83	125	167	333
61	41	82	123	164	328
62	40	81	121	161	323
63	40	79	119	159	317
64	39	78	117	156	313
65	38	77	115	154	308
66	38	76	114	152	303
67	37	75	112	149	299
68	37	74	110	147	294
69	36	72	109	145	290
70	36	71	107	143	286
71	35	70	106	141	282
72	35	69	104	139	278
73	34	68	103	137	274
74	34	68	101	135	270
75	33	67	100	133	267
76	33	66	99	132	263
77	32	65	97	130	260
78	32	64	96	128	256
79	32	63	95	127	253
80	31	63	94	125	250
81	31	62	93	123	247
82	30	61	91	122	244
83	30	60	90	120	241
84	30	60	89	119	238
85	29	59	88	118	235
86	29	58	87	116	233
87	29	57	86	115	230
88	28	57	85	114	227
89	28	56	84	112	225
90	28	56	83	111	222
91	27	55	82	110	220
92	27	54	82	109	217
93	27	54	81	108	215
94	27	53	80	106	213
95	26	53	79	105	211
96	26	52	78	104	208
97	26	52	77	103	206
98	26	51	77	102	204
99	25	51	76	101	202
100	25	50	75	100	200

Attachment H-4

Macroinvertebrate Tissue Collection

Attachment H-4: Macroinvertebrate Tissue Collection

I. Scope and Application

The following document describes the general methodology that will be used in the field to collect macroinvertebrate tissue samples

II. Personnel Qualifications

Personnel conducting the macroinvertebrate sampling will be familiar with invertebrate sampling procedures.

III. Equipment List

Staff assigned the responsibility of collecting macroinvertebrate samples will be provided with the following information:

- Work documents (field sampling plan, health and safety plan, etc.)
- Water body name and site maps
- Number of samples to be collected
- Collecting and processing procedures
- Special instructions (if any)
- Sampling permits and licenses (if applicable)

The following collection equipment and materials will be available, as required, during benthic macroinvertebrate sampling:

- Personal protective equipment (as required by the health and safety plan)
- 9” by 18” rectangular dip net with 500-micron mesh
- Standard kick-net, -1 meter width 500 micron mesh
- Cooler(s) with ice
- Pre-cleaned small glass sample jars
- Electronic balance
- Bucket
- Forceps and magnifying glass

- Cleaning and decontamination materials, including de-ionized water
- Indelible ink markers
- Camera
- Hand-held GPS
- Field notebook

IV. Cautions

Cautions include typical hazards associated with working around water (e.g., drowning, falling on slippery surfaces, etc.).

V. Health and Safety Considerations

Personnel will take proper precautions when working around water.

Personnel will use appropriate lifting techniques.

Personnel will work using the buddy system for safety.

VI. Procedure

Macroinvertebrate tissue samples will be collected using kick nets or hand picking as described below.

A. Collection

The following procedures describe the use of a kick net to collect invertebrate samples and are based on the protocols found in the US Environmental Protection Agency's (EPA's) Rapid Bioassessment Protocols (Barbour et al., 1999) and the 2000 Aquatic Habitat Assessment for the 1½ Mile Reach (Woodlot, 2000):

1. The field crew will don health and safety equipment (e.g., waders, gloves, etc.).
2. The field crew will identify the proposed sample location using GPS or topographic landmarks.
3. At each of the sample locations selected for benthic community sampling, a composite tissue sample will be collected using a kick net or handpicking in the vicinity of the area sampled for benthic community samples.
4. Samples will be taken with a kick net by holding the net stationary on the substrate of while disturbing (kicking) the area immediately upstream of the net.

5. Periodically, the net will be raised and examined for invertebrates. Larger individuals and those from more prevalent species will be selected in the field and removed from the net using tweezers or forceps. An effort will be made to collect similar types of organisms from each of the locations.

6. If kick nets are not effective, invertebrates for tissue samples may also be collected by hand picking hard substrate such as rocks or woody debris using tweezers.

7. The organisms selected will be placed in a labeled, pre-cleaned glass jar and steps 3 through 6 will be repeated until the required amount of sample mass (10 grams, 30 grams for quality assurance [QA] sample) has been collected.

8. Repeat this process once at each of the remaining sampling transects until the desired number of benthic samples is collected.

9. Each sample will be labeled with sampling date and collection location and put on wet ice prior to shipment to the laboratory.

B. Handling, Packing, and Shipping

The following identifies the procedures that will be used to handle, pack, and ship the macroinvertebrate samples:

1. All samples will be given a sample identification number that will be recorded in the field notebook and that will correspond to the sample analysis, sampling date, and collection location.

2. Samples will be inspected to make sure that labeling is correct and that the sample containers are intact. Sample jars will be tightened and taped, if necessary.

3. Chain-of-custody forms, custody seals, address labels, and air-bill forms will be initiated. A copy of the completed chain-of-custody form and air-bill form will be retained by the sampler.

4. Coolers used for transport will be duct-taped at the drain plug on the outside and inside of the cooler.

5. Samples will be placed upright in the bottom of the coolers with cushioning materials placed on top and in between samples (if applicable).

6. The completed chain-of-custody form will be placed into a plastic bag and duct-taped to the inside of the cooler lid.

7. The cooler will be closed and fastened with duct tape around the seam of the lid to prevent water leakage and with strapping tape around the entire cooler to prevent it from opening during transport.

8. A completed custody seal will be placed across the seam of the cooler lid. A completed address label will be placed on top of the cooler. Both will be taped-over using clear packing tape.

9. Samples will be shipped to the laboratory on ice, and the laboratory will be notified of the shipment and will be contacted immediately following the arrival date to ensure that delivery has occurred.

VII. Waste Management

Project-derived waste will be disposed of in accordance with project requirements.

VIII. Data Recording and Management

Field notes will be recorded during sampling activities, and at a minimum, will include the following:

- Names of field crew and oversight personnel
- Sample location (GPS if specified)
- Date, time, and duration of sampling
- General weather conditions
- Substrate characterization
- General water quality parameters
- Sample information (including matrix, sampling method, sample mass, sample ID, sample date and time)
- Habitat description
- General observations of benthic macroinvertebrate abundance and diversity
- Photograph number when pictures are taken (if necessary)

IX. Quality Assurance

The macroinvertebrate tissue sampling will be conducted consistent with the procedures outlined herein. Deviations from the plan will be discussed with the project manager prior to changing any field procedures.

One QA sample will be collected, from a location to be determined in the field based on sample availability, for in duplicate and for matrix spike/matrix spike duplicate (MS/MSD) analysis. (If this is not feasible, the laboratory will analyze a lab spiked blank and spiked duplicate.)

X. References

Barbour, M.T., J. Gerritsen, B.D. Snyder, J.B. Stribling. 1999. *Rapid Bioassessment Protocols For Use in Streams and Wadeable Rivers: Periphyton, Benthic Macroinvertebrates, and Fish, Second Edition*. EPA 841-B-99-002. Prepared for the U.S. Environmental Protection Agency; Office of Water, Washington, D.C.; 7: 3-5.

Woodlot Alternatives, Inc. (Woodlot). 2000. *Aquatic Habitat Assessment: 1½ Mile Reach – GE-Pittsfield/Housatonic River Site, Pittsfield, MA.* September 2000.

Attachment H-5

Benthic Community Assessment

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AQUATIC MACROINVERTEBRATE ASSESSMENT

Description of Laboratory Qualifications

Lotic Inc. (Lotic) is an environmental consulting firm located in Unity, Maine. As specialists in the identification of freshwater macroinvertebrates and the evaluation of benthic communities, Lotic is recognized as one of the leading firms in the field of macroinvertebrate taxonomy and ecology. Because of this expertise, Lotic is used by various state and federal agencies, as well as private clients. Lotic biologists are trained in the identification of all major groups of benthic macroinvertebrates, including the Chironomidae and Oligochaeta, and are experienced in a wide range of collection and assessment techniques. In addition to benthic macroinvertebrate taxonomy, Lotic has provided Whole Effluent Toxicity (WET) testing for municipal and industrial wastewater treatment facilities for over ten years.

All invertebrate identification work will be performed by a certified taxonomist with rigorous quality assurance/quality control (QA/QC) to ensure the highest level of taxonomic data quality. Lotic has the demonstrated capacity to process over 500 samples a year in a timely and accurate manner.

Laboratory Procedures

Sample Receipt

Samples will be checked against the chain of custody and logged in the sample log book upon receipt. All samples will also be checked for proper preservation and re-preserved in 70% ethanol if found to be less than adequately preserved. Any containers received leaking or broken will be replaced, and also noted in the sample log book. A copy of the chain of custody will be retained by Lotic.

Sorting

Samples will be poured through a 500 micron sieve and rinsed with water to remove as much sediment as possible. Samples will be sorted completely using a stereo microscope, and all organisms placed in 4 dram vials with 70% ethanol. Sorted residue will be safely disposed of after sorting QA/QA is complete.

Organism Identification

Macroinvertebrates will be identified using state-of-the-art stereo microscopes and the most recently published taxonomic references. Chironomidae are cleared by immersion in a 10% solution of room-temperature KOH for 24-48 hours. Once cleared the specimens are slide mounted in a CMC-10. Once the mountant has dried, the coverslips are ringed with CMC-10. Oligochaeta are mounted in polyvinyl lactophenol. All slide material is identified with a compound microscope. Slides are labeled with the site location, date and sample number.

Organisms will be identified to genus or species unless the condition of the organism or lack of workable keys prevents it. All identified organisms will be retained in the original sample containers, with the exception of the voucher collection.

Quality Assurance/Quality Control

Sorting

10% of all samples will be randomly chosen and the residue checked for sorting QA/QC by another laboratory technician. If any sample meets less than 95% sorting efficiency, all of the samples sorted by that technician will be resorted.

Internal Taxonomic Quality Assurance

All sample specimens will be identified to genus or species as allowable by specimen condition and maturity. 10% of the samples identified by each taxonomist will be set aside for re-identification by the other project taxonomist If taxonomic agreement (as determined with the Bray-Curtis Index of Similarity) is less than 95%, the taxonomists will discuss the differences, identify where errors were made, and take corrective action.

As a routine component of Lotic QA/QC protocols, a voucher collection is assembled of at least three specimens (when possible) of every taxon identified for each project. This collection will be retained by Lotic until requested by the client or permanently archived to resolve any taxonomic issues. Each vial in the voucher collection will be labeled with the taxon name, sample ID, sample date, taxonomist, and any other relevant sample information.

External Taxonomic Quality Assurance

Lotic and its associates maintain professional contacts with numerous research taxonomists and systematists for taxonomic verification of unusual or rare specimens. Any uncertain unusual taxa will be sent to one or more of these outside experts for verification. All samples subject to QA/QC procedures, along with the results of those procedures, will be recorded in the QA/QC log book.

Data Entry and Reporting Quality Assurance

After data entry in Microsoft Excel format, a qualified taxonomist will check 25% of the completed data sheets against the original bench sheets. If no errors are found, the check is complete. If any errors are found, then all data sheets are checked against the database and all errors are rectified. Any necessary corrections will be noted in the QA/QC log book.

Analysis

If required, the following metrics can be calculated to obtain a water quality evaluation:

- Total richness
- EPT richness (collectively and as individual orders)
- Taxa ratio (E/T x P)
- Indicator taxa richness
- % dominant taxon
- Site index (based on the Hilsenhoff Index)
- Functional feeding group ratios

These metrics are based on the Maine Department of Environmental Protection model, and can be adjusted as needed.

Attachment H-6

Caged Bivalve Bioaccumulation Study Procedures

Attachment H-6: Caged Bivalve Bioaccumulation Study Procedures

I. Introduction

The following *in-situ* caged bivalve study protocol – adapted from the methods presented in the *Draft Standard Guide for Conducting Field Bioassays with Marine, Estuarine and Freshwater Bivalves* (Salazar and Salazar, 1997) – provides the general sampling procedures to be used to conduct a caged bivalve (mussel) trend monitoring program. Caged bivalve monitoring will employ in-river exposure vessels (e.g., cages) and native populations of freshwater mussels to monitor PCB bioavailability in a river. Study procedures are presented below.

II. Sampling Procedures

The mussel cages will be constructed as a modified version of that described in Salazar and Salazar (1997) and USEPA, (1995a). Specifically, a two-cage array of Gee Model 40K holding cages will be suspended in the water column approximately half way up from the River bottom. These galvanized steel mesh cages will be employed as flow-through chambers that allow food particles to flow into the cage while retaining the study population of mussels without injury.

<u>Materials</u>

The following materials will be available for use, as required, during a caged bivalve bioaccumulation study:

- Health and safety equipment (as required by the Health and Safety Plan);
- River maps;
- 5-foot sections of steel rebar and clamps;
- Gee Model 40K holding cages;
- Appropriate numbers of native mussels;
- Transport container (live well, cooler) and ice;
- Clam rake;
- Hammer;
- Depth gauge;
- Thermometer;
- Appropriate packaging materials and forms;

- Field notebook;
- Waterproof labels; and
- Preservative (formaldehyde or alcohol).

Procedures

General procedures that will be followed to deploy and sample the mussel cages include the following:

Step 1 - Don appropriate health and safety equipment (e.g., personal flotation device).

Step 2 - Collect (by hand) an appropriate number of native mussels (*Elliptio Complenata*) from a river basin background location (e.g., Connecticut River).

Step 3 - Select 10 individuals from the study population and process them (following steps in Section III below) into two field blank screening samples for pre-study analysis of PCBs and lipids to determine background concentrations.

Step 4 - Select a second sample of 3 to 5 individuals and preserve them following standard preservation techniques. Retain for laboratory identification/confirmation of species type.

Step 5 - At the appropriate locations, secure mussel cages at mid-depth in the water column with steel rebar, and place a minimum of 20 mussels in each cage, label each cage with cage number and location, (see Figure H-3A for schematic of in-situ shallow water caged mussel setup).

Step 6 - Deploy 2 two-cage mussel arrays at each of the three study locations (four cages per location). Position arrays with a north and south river bank orientation.

Step 7 - Secure mussel arrays on the river bottom with steel rebar.

Step 8 - Determine the exact location of each array in the field and record location in the field notebook. Where possible, the cages will be at least one meter off of the river bottom and one foot below the water surface, and located in a position where fluctuations in the water level will not be expected to expose the cages during low flow.

Step 9 - Sample mussels after an appropriate exposure period, removing the setups from the water after the final sampling event.

Step 10 - As mussel samples are collected, record the following in the field notebook:

- date and time of sampling;
- cage number and location;
- contact duration;
- sampling personnel;
- river water temperature;
- water depth and cage location in the water column; and
- notes on mussel mortality and condition.

Step 11 - Retain sufficient mass of mussels for required chemical analyses (e.g., 5 to 10 individuals or a minimum of 10 grams of tissue per sample).

Step 12 - During and after collection, hold samples on ice in an insulated cooler until processing for shipment to the analytical laboratory.

Step 13 - Repeat Steps 9 through 12 until appropriate quantities of mussels are obtained from each array included in the sampling event.

III. Sample Processing and Packaging

Procedures for field processing, wrapping, and labeling biota samples are listed below:

A. During and after field collection, all mussel samples will be held on ice in an insulated cooler.

B. All mussel samples will remain whole and unshucked.

C. Number each composite sample and record the following information for each sample in the field notebook:

- weight (total weight of unshucked composite sample);
- number of individuals comprising sample;
- species;

- sample location; and
- sample identification number.

D. Rinse samples in distilled water, then wrap in aluminum foil, followed by freezer paper, and tape securely so that the package does not open during shipment.

IV. Shipping

For shipment to the analytical laboratory, all mussel samples will be packaged in accordance with the following procedures:

A. Place sample packages in an insulated cooler lined with two bags of ice on the bottom of the cooler. Fill cooler with biota samples, leaving sufficient room for two bags of ice on top of the samples. If needed, fill remaining space in cooler with additional ice.

B. Fill out appropriate chain-of-custody forms with instructions for sample processing and chemical analyses. Put chain-of-custody forms in a sealable plastic bag and tape to the inside of the cooler lid.

C. Close cooler and seal with shipping tape; place a signed custody seal label across closure at front of cooler.

D. Affix airbill (if appropriate) with shipper's and consignee's addresses to top of cooler.

E. Ship samples to arrive at the laboratory within 24 hours of sample collection. In accordance with USEPA guidance, preservation with ice as described above is appropriate for mussel samples when the maximum storage/shipping time from collection to delivery at the processing laboratory is less than 24 hours (USEPA, 1995b).

V. Equipment Decontamination

Reusable equipment that comes into contact with biota tissues will be cleaned prior to use and between samples using the following procedure:

A. Potable water rinse, and wash with a detergent solution (i.e., Alconox).

B. Potable water rinse.

Solid materials (e.g., disposable gloves and other disposable equipment) from sampling activities will be placed in plastic bags. These bags will be transferred into larger containers and disposed of properly.

VI. Laboratory Processing

Once the caged mussel samples have been delivered to the analytical laboratory, the following processing procedures apply:

Step 1 - Storage

A. Samples should be stored on site on freezer shelves. Samples should be frozen within 24 hours from the time of receipt by the laboratory. Keeping the biota samples on ice in the field and during transportation, followed by freezing at the laboratory, should minimize any tissue deterioration.

Step 2 - Preparing to grind

A. Samples shall be thawed at room temperature for 24 hours prior to processing.

B. Prepare sample bottles – label year and sequence number on tape and wrap around each sample bottle. Consult the sample tags and/or chain-of-custody (COC) forms for specific parameters to be run.

Step 3 - Processing the specimen samples

A. Mussel samples will be prepared as whole-body composite samples minus the shell. Mussels will be shucked using a sterile tool and weighed (without the shell) to provide a total sample weight for each sample. Prior to shucking, mussel samples will be thoroughly rinsed with deionized water. All shells will be discarded.

B. A new sheet of aluminum foil will be used for each sample. Processing tools will be decontaminated between each sample. Non-phosphate soap and distilled/deionized water will be used for cleaning, followed by a triple rinse sequence of hexane and distilled/deionized water.

C. Grinding

- The samples shall be homogenized thoroughly using a tissue miser homogenizer, then packaged in appropriate sample bottles.

- Square pieces of foil should be placed over the top of all ground samples before being sealed with the lid.

- Sample grinders will be disassembled and rinsed at the beginning of use and between each sample. Non-phosphate soap and distilled/deionized water will be used for cleaning, followed by a triple rinse sequence of hexane and distilled/deionized water. The grinding equipment should be allowed to thoroughly dry before grinding the next sample.

VII. Chain-of-Custody Procedures

All samples will be collected and handled in accordance with the chain-of-custody procedures summarized below:

A. Prior to relinquishing samples for packaging and shipment, one member of the sampling team will record relevant information on a chain-of-custody form.

B. The samples will be packaged for shipment as described in Sections III and IV above.

C. If samples are stored temporarily prior to shipment, they will be kept cold and placed in a secured storage area. Coolers will be sealed and custody seals affixed just prior to shipment.

References:

Salazar, M., and S. Salazar. DRAFT Guide for Conducting Field Bioassays with Marine, Estuarine & Freshwater Bivalves. EVS Consultants. April 13, 1997.

United States Environmental Protection Agency (USEPA). AED Laboratory Operating Procedure Caged Bivalve Deployment. Revision I. 1995a.

USEPA. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1: Fish Sampling and Analysis. Second Edition, Office of Water, EPA 823-R-95-007. September 1995b.

Attachment H-7

Tissue and Preparation and Homogenization for Biota and Plant Matrices

STANDARD OPERATING PROCEDURE

TISSUE AND PREPARATION & HOMOGENIZATION

FOR BIOTA AND PLANT MATRICES

Reference Methods: US EPA SW-846 Test Methods for Evaluating Solid Waste

LOCAL SOP NUMBER:	NE132_07
EFFECTIVE DATE:	03/29/2011
SUPERSEDES:	NE132_06
SOP TEMPLATE NUMBER:	SOT-ALL-Q-006-rev.03

APPROVALS

[signature]	03/29/2011
Dan Pfalzer Assistant General Manager	Date
[signature]	03/29/2011
Christina L. Braidwood Quality Manager	Date

PERIODIC REVIEW

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

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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STANDARD OPERATING PROCEDURE

LABORATORY PROCEDURE NE132_07.DOC

REVISION 7 (03/29/11)

1.0 IDENTIFICATION OF TEST METHOD

1.1 Standard Operating Procedure for tissue preparation, processing and homogenization prior to extraction/digestion and analysis.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 This method is applicable to the preparation and homogenization of animal and plant matrixes; including but not limited to: fish (whole body and fillets), mollusks (mussels, clams, etc.), crustaceans (lobster or shrimp, etc.), mammals (mice, mink, muskrat, shrew etc.), reptiles and amphibians (frogs or turtles, etc.), macro invertebrates (benthic worms, eels, insects and other biota), and vegetation (coastal and wetland grasses/plants).

3.0 DETECTION LIMIT

3.1 Not applicable

4.0 SCOPE AND APPLICATION

4.1 This method is intended to describe the preparation and homogenization procedures prior to the extraction, digestion and/or clean up of sample extracts. This procedure uses a variety of cutting, grinding and scaling equipment for size reduction, composting, and homogenization. Client and/or project may dictate additional specific requirements than stated below. Samples are best processed when partially frozen. Samples may be re-frozen after processing pending extraction or digestion.

5.0 SUMMARY OF TEST METHOD

5.1 Fish

5.1.1 Samples are weighed, measured, and gender determined if possible. The fish may be processed whole body or as fillets, and with the skin on or off. If fillets are to be removed and processed separately, this is generally done after the removal of the skin. If compositing is required, the identified samples for composite are filleted or skinned prior to homogenization. The carcass of the fish (after removal of the fillet) may be maintained for separate homogenization and analysis if requested.

5.2 Mollusks, crustaceans and other like invertebrates

5.2.1 Samples are measured and weighed prior to processing. Mollusks must be removed from their shells before processing. Due to the low weight of a single mollusk, crustacean, or invertebrate, these sample types are generally composited with others of the same species and/or sampling area prior to homogenization. Gender determination may need to be performed, i.e. lobsters. This is done prior to any processing and recorded. Additionally, lobsters are usually dissected, and the edible

meat (tail and claw) is removed for homogenization. Certain internal organs such as the hepatopancreas may need to be processed separately. If crabs are being processed, the legs, claws and body cavity are generally homogenized together.

5.3 Mammals

5.3.1 Mammals such as mink, mice, shrew or other rodents, must be prepared in a glove box or bio-hazard hood with the use of a HEPA biological respirator due to the potential health hazards associated with mammal tissue. All project specific sample preparation (weighing, skinning, compositing and homogenization) is performed in the glove box. Waste from the process must be treated with bleach before disposal. The outside surfaces of the sample containers must be disinfected before removal from the glove box.

5.4 Reptiles and Amphibians

5.4.1 Samples are generally processed as whole body samples. Depending upon the size, the specimen may need to be cut into small pieces and processed in part, then re-combined as a single sample. Due to the thickness of the skin of most reptiles, such as frogs, it is recommended that these be processed without the skin. If the skin must be processed, ensure that the grinder or processor blades are sharpened before use. The blades may need to be re-sharpened between every few samples as needed. Turtles must be removed from the shell prior to processing by digging out the head and legs, and as much of the body as feasible.

5.5 Macro invertebrates

5.5.1 Macro invertebrates such as worms, eels, insects or benthic biota are generally processed as whole body samples. Depending upon the size, the specimen may need to be cut into small pieces and processed in part, then recombined as a single sample. Due to the low weight of a single invertebrate, these sample types are generally composited with others of the same species and/or sampling area prior to homogenization.

5.6 Plants

5.6.1 Samples are rinsed prior to processing to remove soil, silt, small insects or other debris. Depending upon the size of the plant and the leaves, the sample may be processed mechanically, or may have to be cut into small pieces by hand. Plants can be processed either wet or dry, depending upon project specifications

6.0 DEFINITIONS

6.1 Abdomen- the posterior section of the body behind the thorax in an arthropod.

6.2 **Abductor-** to draw or spread away (as a limb or the fingers) from a position near or parallel the median axis of the body or from the axis of a limb.

6.3 **Arthropod-** any of a phylum (Arthropoda) of invertebrate animals (as insects, arachnids, and crustaceans) that have a segmented body and jointed appendages, a usually chitinous exoskeleton molted at intervals, and a dorsal anterior brain connected to a ventral chain of ganglia.

6.4 **Biota-** the flora or fauna of a region.

6.5 **Bivalve-** being or having a shell composed of two valves (shells).

6.6 **Caudal-** directed toward or situated in or near the tail or posterior part of the body.

6.7 **Carapace-** bony or chitinous case or shield covering the back or part of the back of an animal (as a turtle or crab).

6.8 **Composite-** combining the typical or essential characteristics of individuals making up a group.

6.9 **Crustacean-** any of a large class (Crustacea) of mostly aquatic mandibular arthropods that have a chitinous or calcareous and chitinous exoskeleton, a pair of often much modified appendages on each segment, and two pairs of antennae and that include the lobsters, shrimps, crabs, wood lice, water fleas, and barnacles.

6.10 **Digestate-** product of digesting.

6.11 **Fillet-** to cut, a boneless cut of fish.

6.12 **Head-** the upper or anterior division of the animal body that contains the brain, the chief sense organs, and the mouth.

6.13 **Hepatopancreas-** a glandular structure (as of a crustacean) that combines the digestive functions of the vertebrate liver and pancreas.

6.14 **Homogenize-** to reduce the particles of so that they are uniformly small and evenly distributed.

6.15 **Mantle-** a fold or lobe or pair of lobes of the body wall of a mollusk or brachiopod that in shell-bearing forms, lines the shell and bears shell-secreting glands.

6.16 **Pectoral muscle-** any of the muscles which connect the ventral walls of the chest with the bones of the upper arm and shoulder and of which there are two on each side of the human body.

6.17 **Swimmerets-** one of a series of small unspecialized appendages under the abdomen of many crustaceans that are best developed in some decapods (as a

lobster) and usually function in locomotion or reproduction

6.18 **Telson-** the terminal segment of the body of an arthropod or segmented worm.

6.19 **Thorax- 1)** the middle of the three chief divisions of the body of an insect also, the corresponding part of a crustacean or an arachnid. **2)** the part of the mammalian body between the neck and the abdomen also, its cavity in which the heart and lungs lie.

7.0 INTERFERENCES

7.1 Samples being tested for metals must be processed with a ceramic knife and/or ground with a plastic blade to prevent contamination from metals such as steel or tin.

7.2 Samples being tested for organics must be processed with metal, Teflon, PTFE and or glass utensils. The use of plastics may cause interferences with the analysis of samples.

8.0 SAFETY

8.1 The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. Approved PPE, which includes safety glasses, gloves, must be worn at all times in the lab. Lab coats are provided and may be worn. All Personal Protective Equipment (PPE) must be removed before leaving the laboratory area and before entering the employee lounge or eating area. Always wash your hands before leaving the laboratory.

8.2 All standards, reagents and solvents shall be handled under a hood using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, make sure to use a secure transporting devise and/or secondary outer container.

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

8.4 Extreme caution must be taken when using or handling knives, descalers, and grinders to homogenize the biota samples.

8.5 Re-useable cotton mesh glove liners may be worn under latex or PVC gloves as an additional measure when using sharp tools or knives, or when dealing with samples that have sharp teeth, spines, fins, or thorns. The mesh lining can help prevent piercing of the skin in case a tool or sample slips, during dissection or other preparation steps.

8.6 Polychlorinated biphenyls should be treated with extreme caution; as a class of chemical compounds they possess both toxic and suspected carcinogenic

properties.

8.7 All additional company safety practices shall be followed at all times as written in the Pace Analytical Chemical Hygiene Plan.

9.0 EQUIPMENT AND SUPPLIES

9.1 Cutting board-made of either glass or polyethylene.

9.2 Food processor with titanium cutting blade (small), or blender with stainless steel blades (large).

9.2.1 2- Retsch Grindomix (model GM200) with glass and or plastic mixing bowls

9.2.2 1-Kitchen Aid Little Ultra Power

9.2.3 1-Tor Rey (model M22) Large Food Processor

9.3 Knives: ceramic stainless steel, or titanium. (See Section 7.0 for interferences and/or contamination associated with different material knives and blades).

9.3.1 Gerber Stainless Steel Boning knives

9.3.2 Dexter Russel Chopping knives

9.3.3 Oneida Stainless Steel fillet knives

9.3.4 URI Eagle Ceramic Knife

9.4 Necropsy dissection kits

9.5 Analytical balance with precision to 0.01g.

9.6 Labconco multi-hazard glove box.

9.7 Advantage 200 LS Respirator Facepiece

9.8 Bench liner material (Lab Mat) and scissors.

9.9 Aluminum foil.

9.10 Plastic wrap or wax paper.

9.11 Titanium fork.

9.12 Teflon-coated spatula.

9.13 Teflon or stainless steel tweezers and dissection scissors.

9.14 PVC or Latex gloves.

9.15 Ruler.

9.16 Mallet.

9.17 Stainless steel or plastic strainer.

9.18 Salad spinner.

9.19 Pre-cleaned glass sample jars with Teflon or PTFE-lined caps.

9.20 Kim wipes.

9.21 Nylon bristled brushes for cleaning.

10.0 REAGENTS AND STANDARDS

10.1 **Deionized (DI) water**- Deionized (DI) water or reagent water is ASTM Type II laboratory reagent grade water or better (Type I).The Millpore NANO-pure system provides Type I water used in the metals laboratory for rinsing lab glass and plastic ware. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination.

10.2 **Hexane** - Pesticide grade

10.3 **Acetone** - HPLC grade

10.4 **Nitric acid 25%** - Add 250mL concentrated $HNO3$ to 400mL of reagent water and dilute to 1L in an appropriate flask. (See metals lab for this prepared solution).

10.5 **10% Bleach solution** - Add 100mL of commercial bleach to 500mL of reagent water and dilute to 1 liter in an appropriate beaker or flask.

10.6 **Alconox** - cleaning solution.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT and STORAGE

11.1 Sample collection is not applicable to the Pace laboratory operation.

11.2 Please see the Pace SOP (NE227) that describes the responsibilities of sample custody including all proper documentation, verification, and tracking procedures following Chain of Custody (COC) protocols, sample receipt procedures, and Internal COC procedures for sample tracking include the use of sample tracking logbooks.

11.3 All samples should remain frozen at all times unless being tested. Fish usually arrive whole bodied or already filleted. Once received the sample must be ground and homogenized so that it may be analyzed. The homogenized fish tissue can be held for 6 to 12 months. The fish solvent extracts can be held for 3 months. Some

clients may request that the body and/or head of fish be saved once the fillets are cut out. Other biota material may have other specifications stated specifically for that project.

11.4 If samples are not shipped frozen, they will be stored in freezers at Pace Analytical upon arrival, and until processing. The samples must remain frozen and maintained at < -20˚C. Sample processing and extraction/digestion hold times are suspended by freezing the sample. Hold time monitoring is resumed when samples are removed from freezers for processing and then returned to freezers pending extraction or digestion. The organic hold time is 14 days from sample collection to extraction, and 40 days from extraction to analysis. The metals hold time is six months from sample collection to digestion and analysis. If mercury is to be determined, the hold time is 28 days from sample collection to digestion and analysis.

11.5 Tissue samples: As guidance, a minimum of 50 grams of sample must be collected for organic analyses, and 5 grams for metals analyses, in a glass jar with a Teflon or PTFE lined screw cap. The amount of sample needed, will depend upon the project management plan such as reporting limits and the need for MS/MSD and/or duplicate analyses. Extra sample must be collected, if possible, to allow the laboratory adequate sample volume in case of re-extract and reanalysis is needed. Large whole individual fillets or vegetation may be wrapped in plastic or aluminum foil depending upon the requested analyses. Large crustaceans, reptiles or amphibians may be individually packed in well-labeled Styrofoam coolers.

12.0 QUALITY CONTROL

12.1 Contamination Prevention

12.1.1 If the purity of a reagent is in question, analyze for contamination.

12.1.2 Blades for dissection may need to be re-sharpened between every few samples as needed.

12.1.3 Certain project specific sample preparation (weighing, skinning, compositing and homogenization) is performed in the glove box. Waste from the process must be treated with bleach before disposal. The outside surfaces of the sample containers being processed must be containerized, treated and disinfected before removal from the glove box.

12.2 The procedures described below are general cleaning and pre-processing procedures that are to be followed regardless of the type of tissue being processed. Samples are prioritized by the Laboratory Supervisor or Lab Manager based on hold time and client due date. All weights, measurements and other project required observations are recorded in LIMS.

12.2.1 Wash all utensils, sample processors (blades, blade post, cup and lid) and cutting boards with an Alconox solution and a sponge. Rinse thoroughly with tap water, then with DI water.

12.2.2 If the samples are going to be processed for organic analyses only, rinse

all washed utensils, processor parts and surfaces with hexane followed by rinsing with acetone.

12.2.3 If samples are going to be processed for metal analyses only, rinse all plastic and ceramic utensils with DI water and then Nitric acid 25% solution and then DI water again.

12.2.4 If requested by the client, the equipment or processing blank should be collected at this time by pouring DI water into and out of the processor, over the surfaces of the utensils and over the cutting board. The blank is collected in the appropriate container, at the project specification frequency, for the determinative analysis.

12.2.5 Gloves must be worn when handling tissue samples. Latex gloves may be worn. All gloves must be talc or dust free.

12.2.6 Tissue samples should be partially thawed before starting, to the point where it becomes possible to make an incision in, or cut through, the flesh. When samples are completely thawed they become soft and difficult to cut or fillet. NOTE: If whole bodies are not being processed, and the tissue is partially frozen during dissection, there is less of a chance of puncturing the gut cavity and any internal organs. Inadvertent puncture of the internal organs may contaminate the part(s) of the animal that have been selected for analysis. Also, internal organs may rupture during freezing. If this is observed during dissection, it must be noted in the processing records. Note any morphological abnormalities on the processing records.

12.3 Hold times: The homogenized fish tissue can be held for 6 to 12 months. The fish solvent extracts can be held for 3 months.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Not Applicable

14.0 PROCEDURES

14.1 Fish Tissue Preparation:

14.1.1 Determine the wet weight for each individual fish using a calibrated balance and record in LIMS. The balance should be covered with aluminum foil if aluminum is not a metal of concern. If aluminum is a metal of concern and the sample will not be analyzed for organic compounds the balance should be covered with plastic wrap. If the sample is for both metal and organic compounds, wax paper may be used. Catch any excess fluid coming from the thawing specimen into the wax paper, foil or plastic wrap. All liquid from thawed whole fish must be kept as part of the sample. The technician must remember to zero the balance with the aluminum foil, plastic wrap, or wax paper on it before weighing the specimen. The foil, plastic wrap, or wax paper must be changed after each weighing.

14.1.2 Determine the length of each fish using a ruler, and record in LIMS. Some measurements may, or may not be, a part of the project specifications.

14.1.3 If gender identification is needed this must be done prior to the scaling and filleting processes.

14.1.4 Removal of Scales or Skin: If required by project specifications, the scales and/or skin of the fish will be removed prior to filleting.

14.1.5 Lay the fish on the cleaned, and/or lined, cutting board.

14.1.6 Scrape the fish from tail to head using the electric, automated descaler with ceramic claws to remove the scales. Note: If performing metals analysis, titanium or ceramic must be used.

14.1.7 Rinse the cutting board between fish with DI water and Alconox. If plastic, wax paper, or foil is used, change between fish.

14.1.8 Rinse the outside of the fish with DI water and pat dry with paper towel Place the fish on its side, on a clean cutting board, for filleting or skinning.

14.1.9 To skin the fish, loosen the skin behind the gill cover and pull the skin off toward the tail with a Catfish skinning tool, cutting lightly along the inside of the skin, Slowly separate the skin from the muscle tissue of the body or the fillet.

14.2 Filleting the Fish

14.2.1 Using fresh gloves and the specified knife, make a cut behind the entire length of the gill cover, making sure to cut through the skin, if still attached, flesh, and as close to the bone as possible. Note: If the fish samples are small, and it appears difficult to fillet, or if the amount of the fillet appears to be insufficient for the analysis, consult the Project Manager prior to filleting. In some cases it may be necessary to homogenize the whole body.

14.2.2 Make a cut across the base of the tail fin keeping as close to the caudal fin (tail) as possible. Continue cutting along the underbelly of the fish moving from the head to the tail.

14.2.3 Go back to the cut made at the beginning at the gill cover and slice down the entire length of the fish following along the backbone until reaching the cut previously made across the tail.

14.2.4 Remove the fillet from the fish. Be sure to include the belly flap in each fillet and do not remove the dark muscle tissue in the vicinity of the lateral line from the light muscle tissue that makes up the rest of the muscle tissue mass.

14.2.5 Remove any bones that may be left attached to the fillet. Repeat the fillet

steps for the second side of the specimen.

14.2.6 The general procedure recommended for filleting fish is illustrated in Apendix 1.

14.2.7 Note in the sample processing records in LIMS if the internal organs were ruptured during freezing or if inadvertent puncture of the internal organs occurred during the filleting process, rinse the fillet(s) tissue with DI water.

14.2.8 Cover the balance with the appropriate clean lining, and weigh the fillet(s). Record the fillet(s) weight(s) in the processing records.

14.2.9 If the fillet(s) and/or the carcass are to be homogenized immediately, proceed to Section 14.3. If not, rinse all fish parts with DI water and store in the appropriate container; see Section 9.0 for allowable materials. Note that it may be necessary to chop the fillet(s) or carcass into smaller pieces, with the appropriately cleaned knife, before storage, and before homogenization, so the entire sample will fit into the storage container or the homogenization vessel. If the samples will not be homogenized immediately, the samples must be placed back into the freezer, until homogenization.

14.3 Homogenization

14.3.1 Allow the fillet(s), carcass or whole body to partially thaw. Retain all fluids as part of the sample.

14.3.2 Homogenize whole fish bodies, carcasses, or fish fillets by placing them into the small or large food processor fitted with the appropriate blades. The sample may need to be cut into smaller pieces for processing. Process the sample until it appears to be fully and consistently homogenous. Continue to grind the sample until there are no chunks present in the homogenate. The homogenous nature of the sample is vitally important for reproducible results. Sample should be homogenized fully and thoroughly.

14.3.3 Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. Composite homogenates must be prepared from equal weights of individual homogenates. All individual weights that make up one composite must be recorded, if required, or one composite weight may be recorded. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-homogenized by hand mixing prior to being extracted or digested.

14.3.4 Place the individual or composite homogenized samples into the appropriate glass jars to be frozen pending future extraction/digestion. If the samples will not be extracted/digested immediately, the samples must be returned to the freezer until extraction/digestion.

14.3.5 All utensils and equipment must be washed in between samples

according to the procedures described previously in Section 12.2.

14.4 Mollusk (Bivalves) Preparation (Mussels, Clams)

14.4.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 12.2. Obtain samples from freezer.

14.4.2 If required by the project specifications, measure and record the length of the sample shell. Cover the balance with the proper material as described in Section 9.0, and weigh and record the sample weight in LIMS.

14.4.3 Wearing the proper gloves, place the sample on a clean, cutting board. Samples should be partially thawed. If the sample is frozen, it will be difficult to break open the shell. If the sample is excessively thawed, the internal tissue will become soupy and difficult to remove.

14.4.4 If preparing bivalve specimens, use the titanium knife to cut the abductor muscle by sliding the knife through the crevice where the two shells meet. Once the abductor muscle is cut the two shell pieces should come apart easily.

14.4.5 Carefully remove the top shell, and using the Teflon coated spatula, scoop out the internal tissue that is resting on the mantle.

14.4.6 Cover the balance with the proper material and weigh the amount of tissue obtained from the sample. Record the weight along with the information previously recorded on the processing records. The sample may now be stored pending homogenization in the appropriate jar.

14.4.7 Since the amount of tissue obtained from one bivalve is generally small, several specimens are frequently combined to make one sample. Utensils do not need to be rinsed between the individual samples that comprise one composite, but utensils must always be rinsed in between each composite sample.

14.4.8 After the tissue has been removed from all of the specimen shells for one composite or individual sample, place the tissue in the clean small processor with the titanium blade to be homogenized. Grind the sample until it appears to be fully and consistently homogenized and there are no large chunks.

14.4.9 If tissue is being processed for volatile organic carbon (VOC) analysis the homogenization must be done by hand.

14.4.10 Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. Composite homogenates must be prepared from equal weights of individual homogenates. All individual weights that make up one composite must be recorded, if required, or one composite weight may be recorded. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-

homogenized by hand mixing prior to being extracted or digested.

14.4.11 Place the processed samples into the appropriate glass jars to be frozen for future extraction/digestion, and place back into the freezer.

14.4.12 All utensils and equipment must be washed in between samples according to the procedures described previously in Section 12.2.

14.5 Crustaceans (Lobsters, Crabs, Shrimp)

14.5.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 12.2. Obtain samples from the freezer.

14.5.2 If project specifications require gender determination of lobsters, this must be done prior to dissecting. To determine the gender, hold the lobster by the thorax, and flip it over to examine the underneath abdomen, just below the legs and where the abdomen division begins, there is a first pair of swimmerets. The first pair of swimmerets is what is used to distinguish the lobster's gender. If the first pair is soft, has small hairs, and the swimmerets are crossed, it is female. On a male lobster, the first pair of swimmerets is hard and stiff, and generally do not touch.

14.5.3 If the hepatopancreas of the lobster samples is to be analyzed, the lobster samples must be received alive. If the samples are frozen prior to dissection, the hepatopancreas will burst upon thawing making it impossible to remove. To remove the hepatopancreas, the live lobster should be placed on a cleaned cutting board. Wearing the proper gloves, one analyst holds claws out in front of the lobster, while also holding down the lower abdomen and tail. The second analyst takes a titanium-coated knife, and places it on the grove in the outer shell, just behind the head region. Keeping the knife at an angle, the second analyst must push down and forward, to remove the head. Once the head is removed, the hepatopancreas can be seen lying just under the carapace and running the length of the thorax. The hepatopancreas is generally a greenish-yellow color, but there may be some variation. Using the Teflon coated spoon, scoop the hepatopancreas out gently trying not to break it into pieces. Cover the tray of the balance with the proper material, and weigh and record the weight of the hepatopancreas in the processing record, and place it into an appropriate sample jar for freezing and future extraction/digestion.

14.5.4 To remove the edible meat, remove the two claws from the body of the lobster at the joint. Place a piece of lab mat or paper towel over the claw and pound with a mallet. Once the shell is crushed, remove the meat, using the appropriately cleaned tweezers or other tool, making sure to get all the meat in the joints and arms. Cover the balance tray with the appropriate material and record the total tissue weight arms. Record this weight with the previously recorded information from the two claws and sample processing record.

14.5.5 Remove the abdomen and telson from the rest of the outer shell by pulling the lobster apart. Using the titanium coated knife, cut through the

center underside tissue of the lobster and laterally along the exoskeleton of the tail. Once the abdomen and tail have been cut open, separate the shell from the edible meat using cleaned utensils. Any eggs found in the female lobsters will have to be removed and discarded or sampled separately. Cover the balance tray with the appropriate material, and record the weight of the tissue obtained from the abdomen and telson on the processing record. The sample may now be stored pending homogenization in the appropriate jar.

14.5.6 If removing tissue from crabs, break off all legs and claws. Squeeze, pull, or pick all the tissue out of the legs and claws. Pull apart the outer shell. Scoop out the tissue using a Teflon coated spatula. Cover the balance tray with the appropriate material, and record the weight of the tissue obtained from the abdomen and telson on the processing record. The sample may now be stored pending homogenization in the appropriate jar.

14.6 Mammals (Mice, Mink, Muskrat, Shrew)

14.6.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 12.2. Obtain samples from the freezer.

14.6.2 Place the first specimen partially thawed to be processed, and all equipment needed into the glove box/Bio-hood on a freshly laid out lab mat (Blue diaper).

14.6.3 Once all materials are in the glove box and set up for use, seal the transfer box and ensure the motor blower is on. Over tightening of the outer or inner door knobs is not necessary to achieve a good seal. Place your hands into the gloves attached to the glove ports and place Latex gloves over the glove port gloves for use. The outer Latex gloves will need to be changed in between each sample.

14.6.4 If the gender of the mouse or shrew needs to be determined, turn the animal over and note the length of the anus and the distance of the anus from the tail. If the anus is elongated in shape and does not touch the base of the tail, testicles and a large genital papilla are visible, and there are no nipples, the animal is male. If the anus is round in shape and almost touches the base of the tail and/or there are nipples (up to five sets), the animal is female. If the animal is very small, young or immature and a gender determination cannot be made, note that the gender is non determinable. Record the gender observations on the processing records.

14.7 Organ Dissection/Processing

14.7.1 If the mammal is being dissected for Brain, Liver, Kidney, Heart, Lung, or Adipose (Fat) tissue, each organ will need to be harvested.

14.7.2 Place the animal on its back with forceps. Pinch the skin at the base of anus and carefully make an incision at the tail end, and cut just below the skin along the abdomen and past the chest cavity. Cutting the skin flap

at the abdomen cavity carefully separate the adipose tissue from the muscle tissue. Below it should be a white/yellow material. Take this material out.

14.7.3 Identify each organ and remove them from the abdomen cavity.

14.7.4 Weigh and record the weight of the mammal organs and place into the appropriate container.

14.7.5 The rib cage will need to be cut with scissors. Once chest cavity is open, remove the heart and lungs.

14.7.6 Weigh and record the weight of the mammal organs and place into the appropriate container.

14.7.7 Since the amount of tissue obtained from one animal may be small, manually grinding of the organs may need to be done at the time of extraction.

14.7.8 Place the processed samples into the appropriate glass jars to be frozen for future extraction/digestion into the freezer.

14.7.9 Before removing any equipment all utensils and equipment must be washed with DI water and 10% bleach solution.

14.7.10 All disposable materials must be double bagged for disposal.

14.8 <u>**Whole Animal Processing:**</u>

14.8.1 If skinning of the mammal is required, carefully make an incision at the tail end and cut just below the skin along the back, from one hind leg to the other. Make another cut from one hind leg to one front leg and repeat the cut on the other side of the animal. Starting from the tail, lift the skin flap, and carefully separate the skin from the muscle tissue below. Pull the skin forward from the tail to the head to expose the back tissue of the animal. Repeat the procedure on the stomach side of the animal. Note: it may be very difficult to remove the skin from the legs, head, and tail. If some skin cannot be removed, note this on the processing records.

14.8.2 Weigh and record the weight of the mammal on the processing records. Depending upon the size of the mammal, it may need to be chopped into small pieces before being ground. Generally, mice and shrew can be quartered before homogenization if needed.

14.8.3 Put the whole body or chopped sample into the cup of the grinding unit. Turn the grinding unit on low speed and gradually increase the speed to homogenize the sample being careful to minimize any splatter or outside contamination. Homogenize until a uniform consistency is achieved.

14.8.4 Transfer the homogenized sample from the cup to the pre-labeled sample jar using the appropriate utensil. Clean the outside of the sample

jar with the 10% bleach soaked Kim wipe.

14.8.5 To clean the grinding unit in between samples, remove as much residual tissue on the blade as possible by operating the unit at low or medium speed, using DI water and 10% bleach. Rinse unit with DI water if metals are being done and/or hexane or acetone for organics.

14.8.6 Repeat steps 14.9.2 through 14.9.5 until the samples are complete.

14.8.7 Since the amount of tissue obtained from one mouse or shrew may be small, several specimens may be combined to make one sample, as required by project specifications. Utensils do not need to be rinsed between the individual samples that comprise one composite, but utensils must always be cleaned in between each composite sample.

14.8.8 If several specimens will be composited to make one sample, follow the applicable Sections of 14.9.2 through 14.9.5, for each of the specimens. The tissue obtained from each specimen may be weighed and recorded individually, then totaled for the composite weight. If only one composite weight is sufficient for the project specifications, weigh the entire composite and record that weight in LIMS.

14.8.9 Place the processed samples into the appropriate glass jars to be frozen for future extraction/digestion, placed back into the freezer.

14.8.10 Before removing any equipment all utensils and equipment must be washed with DI water and 10% bleach.

14.8.11 All disposable materials must be double bagged for disposal.

14.9 Reptiles and Amphibians (Frogs and Turtles)

14.9.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 12.2. Obtain samples from the freezer

14.9.2 Wearing the proper gloves, place the turtle sample on the cleaned cutting board. The turtle should be partially thawed. If the turtle is frozen, it will be difficult to remove the muscle. If the sample is excessively thawed, the internal tissue will become soupy and difficult to remove.

14.9.3 Take all project required measurements. The distance between the anterior and posterior edge of a turtle carapace (top of shell) should be measured with a ruler and recorded on the processing records. If the entire mass of the turtle, including the shell, needs to be recorded, cover the balance with the proper material and weigh and record this weight in LIMS.

14.9.4 Since the bottom of shell and carapace are extremely dense and difficult to cut through with normal dissecting tools, the muscle tissue of the turtle must be removed by cutting the body of the turtle away from the shell. Insert a knife, made of the proper material, into the skin of the turtle, close to the shell on the lower half of the body. Slowly, cut along the

entire circumference of the shell. Repeat the procedure on the upper half of the body, on both sides of the shell.

14.9.5 With dissection scissors or a ceramic or titanium paring knife of the proper material, remove the skin from the hind limbs, tail, and fore limbs and neck. Remove any visible muscle tissue within the carapace. Most of this tissue will be found in the upper portion of the carapace around the pectoral area.

14.9.6 Using the appropriate utensils, remove the muscle tissue from the tail, neck, hind limbs, and fore limbs, including the feet, leaving bone and claws behind.

14.9.7 Cover the balance with the proper material and weigh the amount of tissue of the turtle sample. Record the weight along with the information previously recorded on the processing records. The sample may now be stored pending homogenization in the appropriate jar.

14.9.8 If processing frogs, allow the frogs to partially thaw, take the project specific measurements, and record them in LIMS. The number of frogs required to make up one sample, and the weight and length of the individual frogs, must be taken and recorded, if specified. In all cases, the skin must be removed from the frog prior to processing and chopped into smaller pieces, due to its thickness. It will then be added to the processor with the whole body of the frog, or it may be discarded depending upon the project specifications.

14.9.9 To skin the frog, make an incision, using the proper utensils, and cut into an area where there is an excess of skin, most likely around the neck. Slowly, pull the skin off of the frog using dissecting scissors, or a ceramic or titanium paring knife, as needed. Once skin is removed, chop it up into tiny pieces using the appropriate knife and set it aside to be processed with the whole frog body.

14.9.10 Cover the balance with the proper material and weigh the amount of tissue obtained from the frog samples if the tissue and the whole body will not be processed. Record the weight along with the information previously recorded on the processing records. The sample may now be stored pending homogenization in the appropriate jar.

14.9.11 Since the amount of tissue obtained from one small turtle or frog may be insignificant, several specimens may be combined to make up one sample. Utensils do not need to be rinsed between the individual samples that comprise one composite, but utensils must always be rinsed in between each composite sample.

14.9.12 If several specimens will be composited to make up one sample, the tissue obtained from each specimen may be weighed and recorded individually, then totaled for the composite weight. If only the composite weight is sufficient for the project specifications, weigh the entire composite and record that weight in LIMS.

14.9.13 After the tissue has been removed from all of the specimens, homogenize the muscle tissue, and skin if required, by placing it into the small or large food processor fitted with the appropriate blades. The sample may need to be cut into smaller pieces for processing. Grind the sample until it appears to be fully and consistently homogenous. Continue to grind the sample until there are no chunks present in the homogenate.

14.9.14 Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. Composite homogenates must be prepared from equal weights of individual homogenates. All individual weights that make up one composite must be recorded, if required, or one composite weight may be recorded. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-homogenized by hand mixing prior to being extracted or digested.

14.9.15 Place the processed samples into the freezer to be frozen for future extraction/digestion.

14.9.16 All utensils and equipment must be washed in between samples according to the procedures described previously in Section 12.2.

14.10 Macro Invertebrates (Benthic Worms, Eels, Insects and other Biota)

14.10.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 12.2. Obtain samples from the freezer.

14.10.2 Cover the balance tray with the appropriate material and record the weight of the invertebrate sample. Since the weight obtained from one invertebrate (benthic worm, insect, biota) may be small, several invertebrates may be combined to make one sample. In many cases, several invertebrates of the same species and sample location are delivered to the laboratory in one sample jar. Each specimen from this jar must be weighed, if requested, and composited to form one homogenized and unique sample. If only one composite weight is sufficient for the project specifications, weigh the entire composite and record that weight. Utensils do not need to be rinsed between the individual samples or specimens that comprise one composite, but utensils must always be rinsed between each composite sample.

14.10.3 Invertebrates such as eels must be chopped into smaller pieces before homogenization. This is generally due to the length of the specimen and the thickness of the skin.

14.10.4 Place the weighed specimen into the clean small processor with the titanium blade to be homogenized. Process the sample until it appears to be fully and consistently homogenized and there are no large chunks.

14.10.5 Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. Composite homogenates must be prepared from equal weights of individual

homogenates. All individual weights that make up one composite must be recorded, if required, or one composite weight may be recorded. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-homogenized by hand mixing prior to being extracted or digested.

14.10.6 Place the processed samples into the appropriate glass jars to be frozen for future extraction/digestion, into the freezer.

14.10.7 All utensils and equipment must be washed in between samples according to the procedures described previously in Section 12.2.

14.11 Vegetation (Coastal and Wetland Grasses/Plants)

14.11.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 12.2. Obtain samples from the freezer.

14.11.2 Wearing the appropriate gloves, plants must be rinsed with DI water to remove soil, silt, small insects, and other debris. Place the plants in a stainless steel or plastic strainer, depending on the determinative sample analysis, and rinse thoroughly with DI water. If analyzing the sample for both metals and organic compounds, rinse the plants carefully over a sink, being sure not to touch the sides of the sink with the plant sample.

14.11.3 Depending on the size and texture of the plants, some may be homogenized in the small food processor with the titanium blade. Samples such as long grass will have to be chopped into small pieces (approximately ½ inch) using titanium or ceramic knives. Leaves can generally be homogenized in the small food processor without pre-cutting.

14.11.4 Cover the balance tray with the appropriate material and record the weight of the plant sample. Since the weight obtained from one plant may be small, several plants may be combined to make one sample. Utensils do not need to be rinsed between the individual samples that comprise one composite, but utensils must always be rinsed in between each composite sample.

14.11.5 If several plants will be composited to make one sample, the weight of each specimen may be recorded individually, and then totaled for the composite weight. If only one composite weight is sufficient for the project specifications, weigh the entire composite and record that weight in LIMS.

14.11.6 After the plant weight for one composite or individual sample has been recorded, place the plant(s) in the clean small processor with the titanium blade to be homogenized, or place them onto the cleaned cutting board to be chopped. Grind or chop the plants until they appear to be fully homogenized.

14.11.7 Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. Composite

homogenates must be prepared from equal weights of individual homogenates. If required, all individual weights that make up one composite must be recorded, otherwise one weight may be recorded for the composite. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-homogenized by hand mixing prior to being extracted or digested.

14.11.8 Place the homogenized plants back into the freezer to be frozen for future extraction/digestion.

14.11.9 All utensils and equipment must be washed between samples according to the procedures described previously in section 12.2.

15.0 CALCULATIONS

15.1 Not Applicable

16.0 METHOD PERFORMANCE

16.1 Not Applicable

17.0 POLLUTION PREVENTION

17.1 Refer to SOP Pace054 and Pace089 for instructions on the disposal of waste generated during the procedures previously mentioned.

18.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

18.1 Not Applicable

19.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

19.1 Not Applicable

20.0 CONTINGINCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1 Not Applicable

21.0 WASTE MANAGEMENT

21.1 Refer to SOP Pace054 and Pace089 for instructions on the disposal of waste generated during the procedures previously mentioned.

22.0 REFERENCES

22.1 NELAP “Quality Systems” Manual, 2005.

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

22.3 EPA/6OOIR-961027, Guidance for the Preparation of Standard Operating Procedures (SOPS) for Quality Related Documents, 1996.

22.4 US EPA 823-R-95-007, "Guidance for Assessing Chemical Contaminated Data for Use in Fish Advisories", Volume 1: Fish Sampling and Analysis 2nd Edition, Office of Science and Technology, Office of Water, 1995.

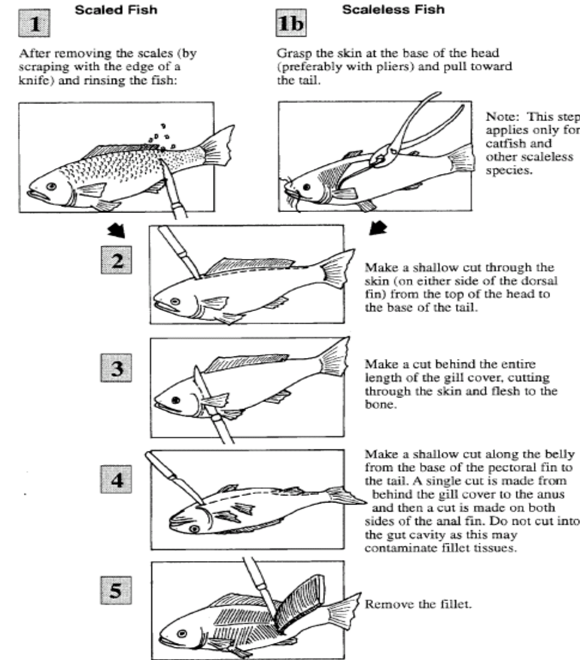
22.5 U.S. EPA, 1991d

23.0 ATTACHMENTS

23.1 Fish Filleting Diagram

23.2 Fish External & Internal Anatomy

Fish Filleting Diagram



23.2 FISH EXTERNAL/INTERNAL ANATOMY

EXTERNAL ANATOMY

1. Remove one fish from the storage tank, place in dissecting pan. Make sure fish is euthanized prior to any dissection.

2. Locate all fins (Figures 1a and 1b):

Paired:	pectoral (caudal to head, located ventrolaterally)
	pelvic (cranial to anus, located ventrolaterally)
Single:	dorsal (caudal to head on dorsal midline)
	adipose (caudal to dorsal fin on dorsal midline; salmonids)
Anal:	(Caudal to anus on ventral midline)

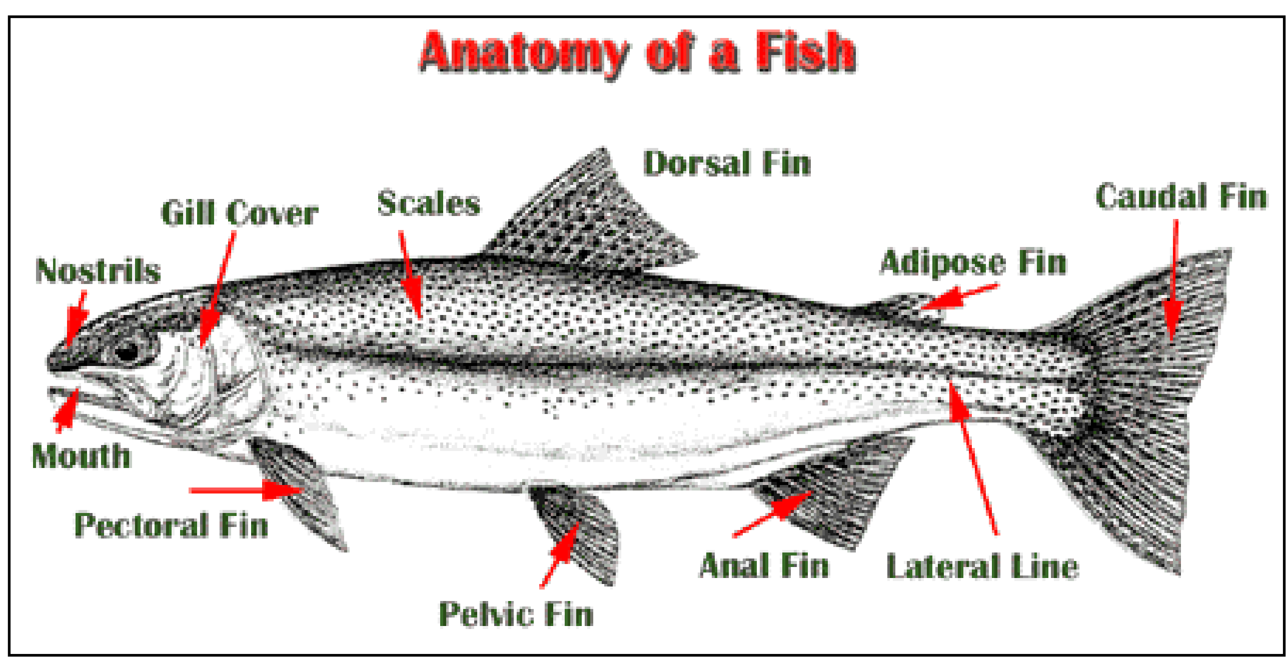


Figure 1a. Anatomy of a Fish (typical salmonid)

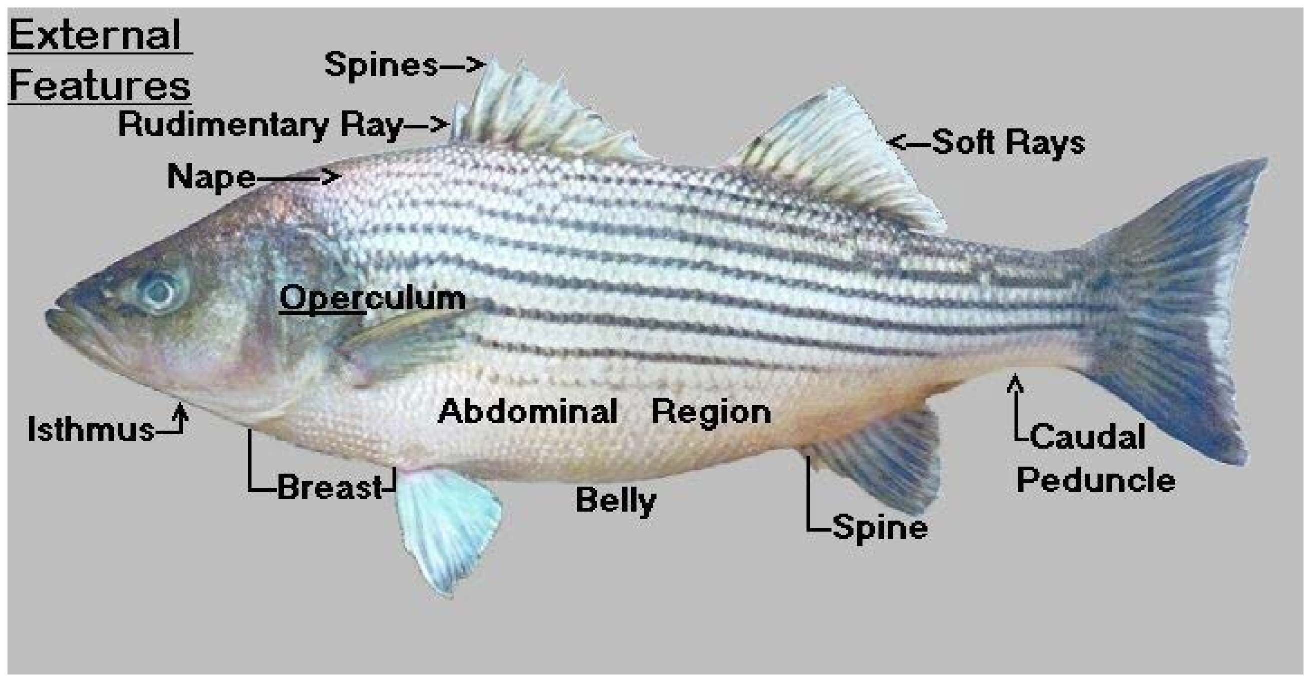


Figure 1b. External Anatomy of Striped Bass

3. Find the lateral line located laterally at mid-body running from head to tail. It arches dorsally over the operculum.

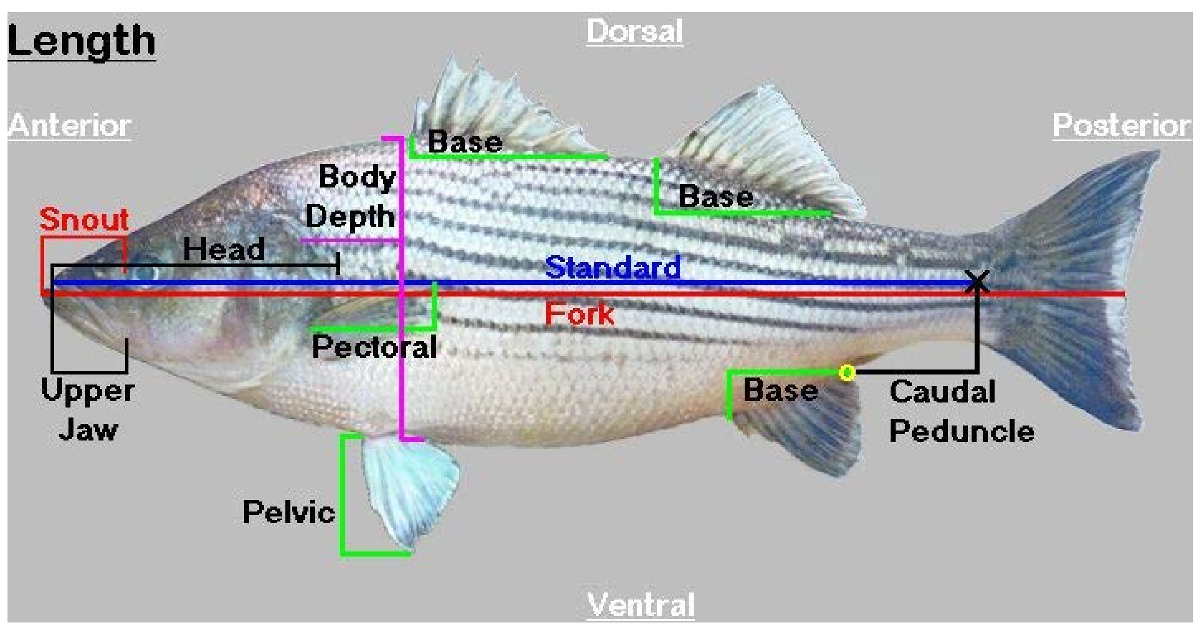


Figure 1c. Typical Measurements Locations

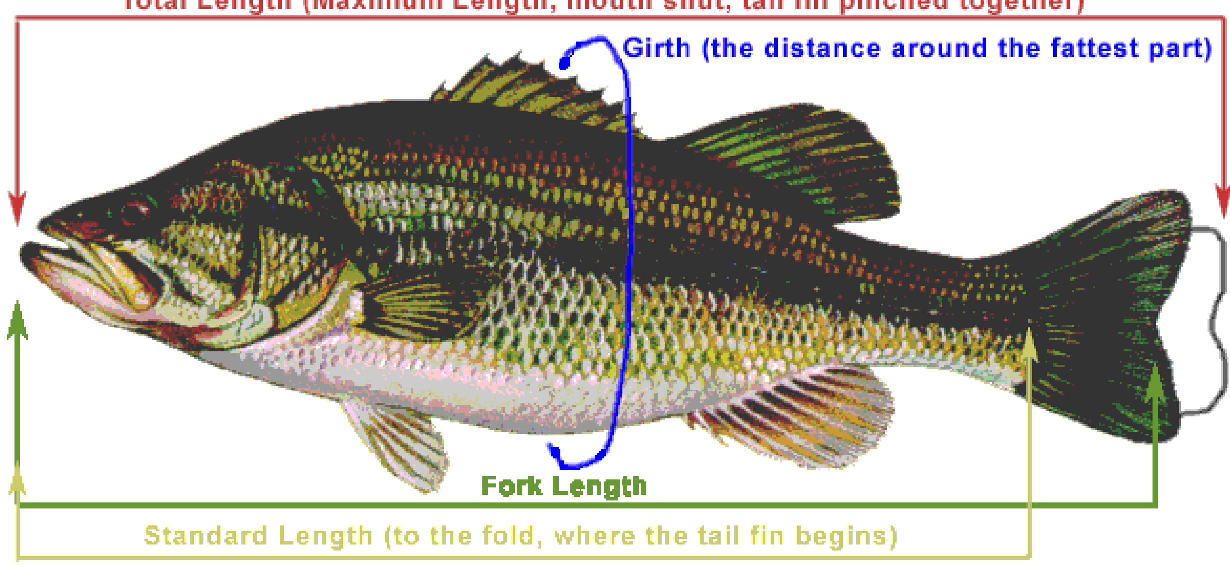
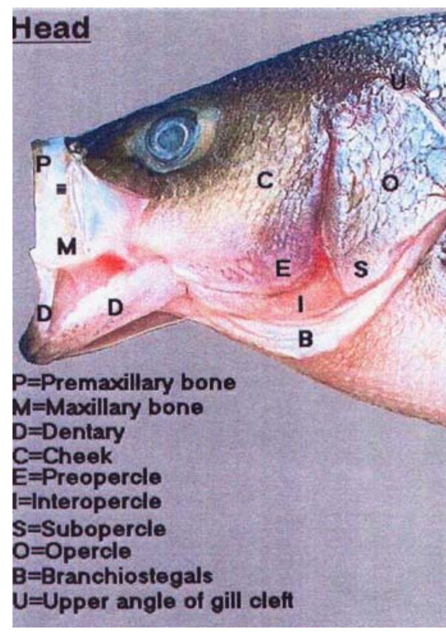
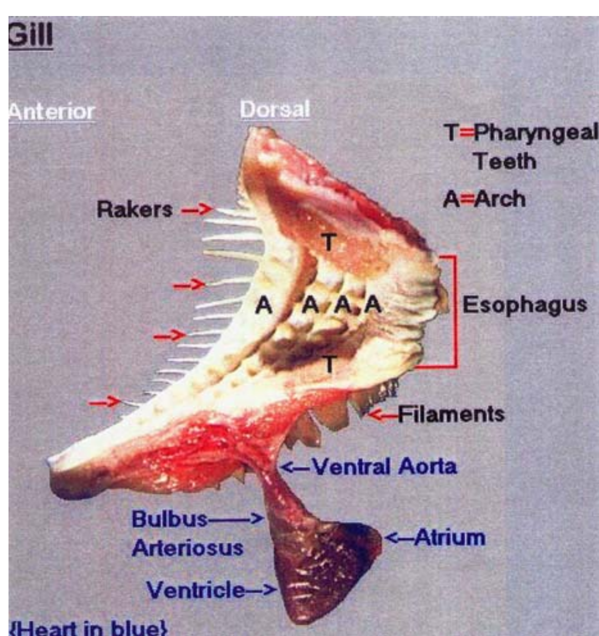


Figure 1d. Typical Measurements of Large Mouth Bass

4. The operculum covers the gills. Lift the opercular flap and identify the bony gill arches, cartilagenous gill filaments, and primary lamellae projecting off the gill filaments (Figures 1f, 1g)





5. Lay the fish on its right side with the head to your left. Open the body cavity with three cuts (Figure 1h). The first cut should originate just craniad to the anus and run cranial to a point ventral to the operculum. The second cut originates from the same point as the first and runs craniad along the dorsum of the body cavity to a point just dorsal to the operculum. The third cut connects the first two. All cuts should be made carefully with the blunt tip of the scissors in the body cavity while applying slight upward pressure to avoid damaging internal organs. Lift off the body wall.

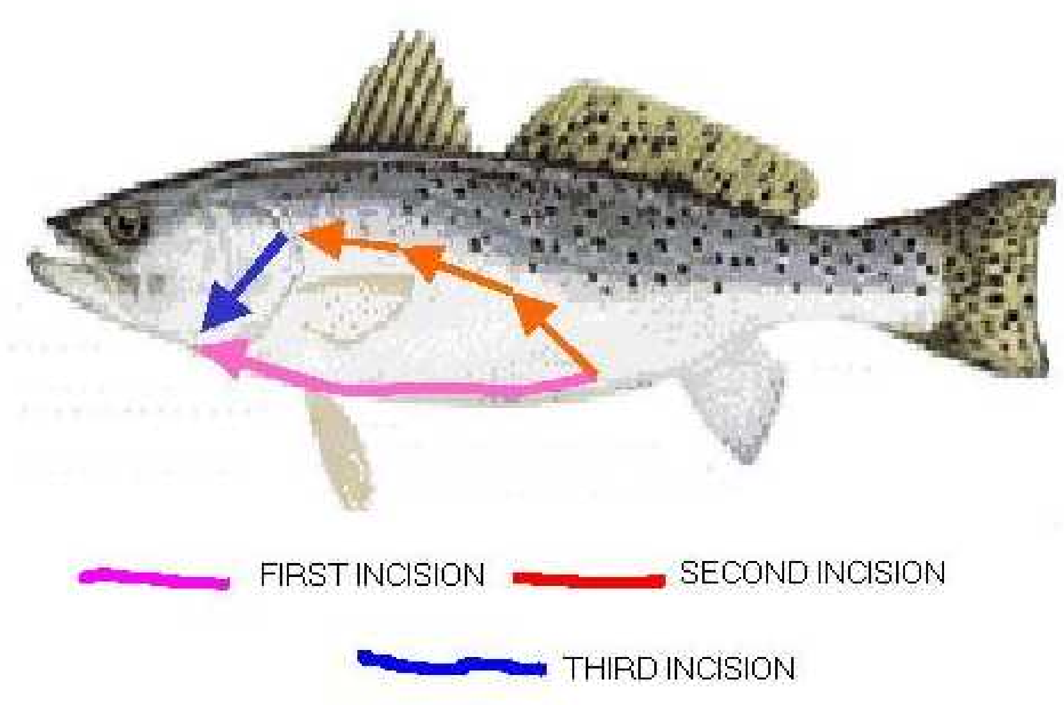
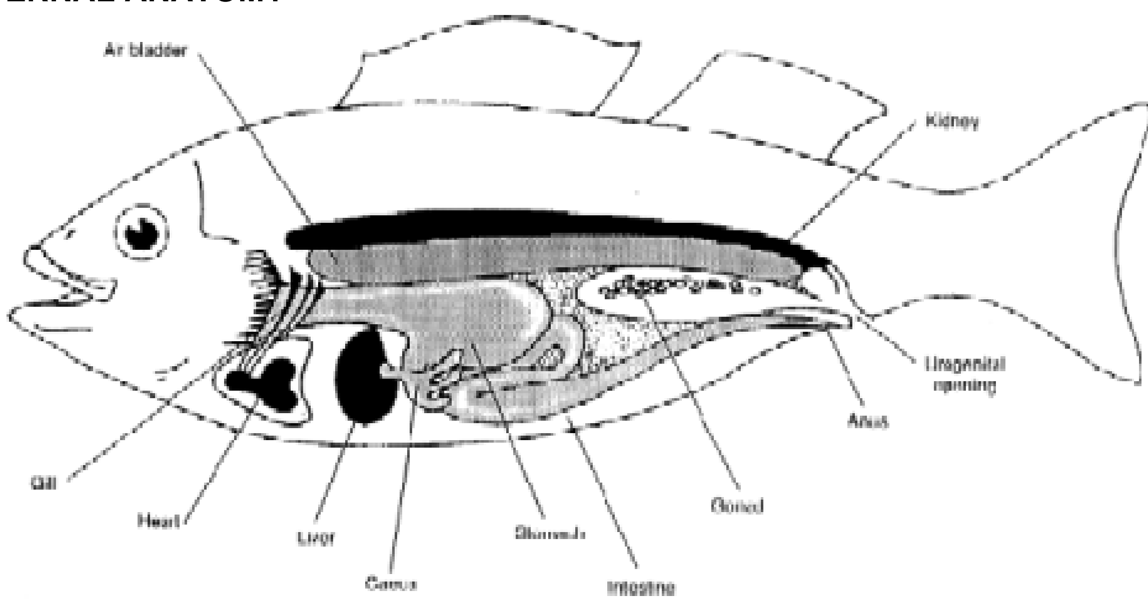
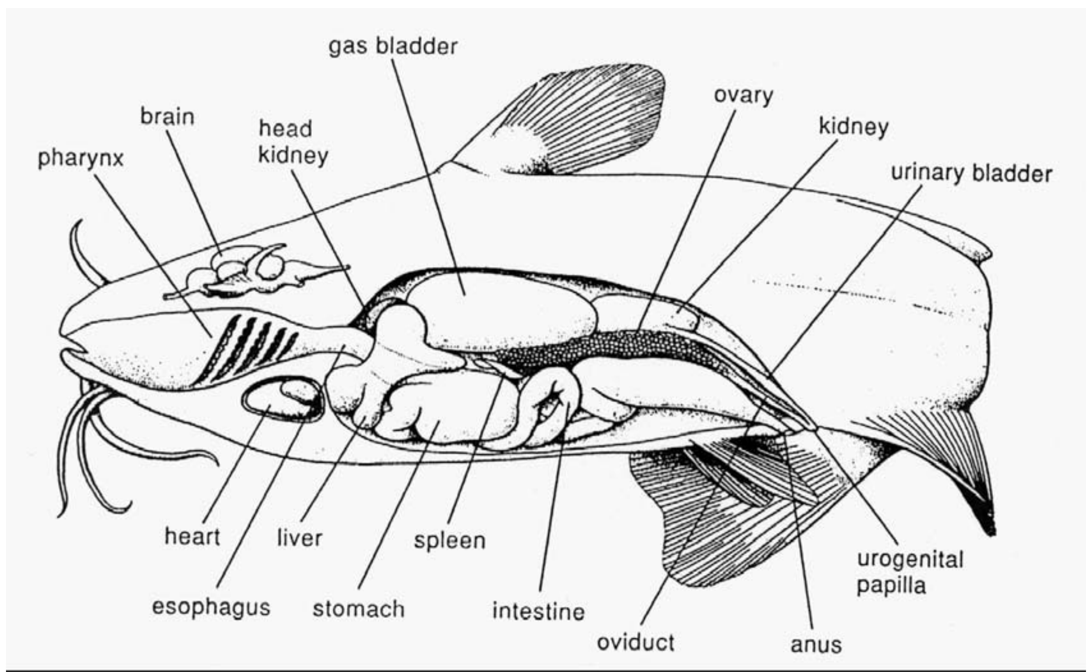


Figure 1h. Incisions to Expose Abdominal Cavity

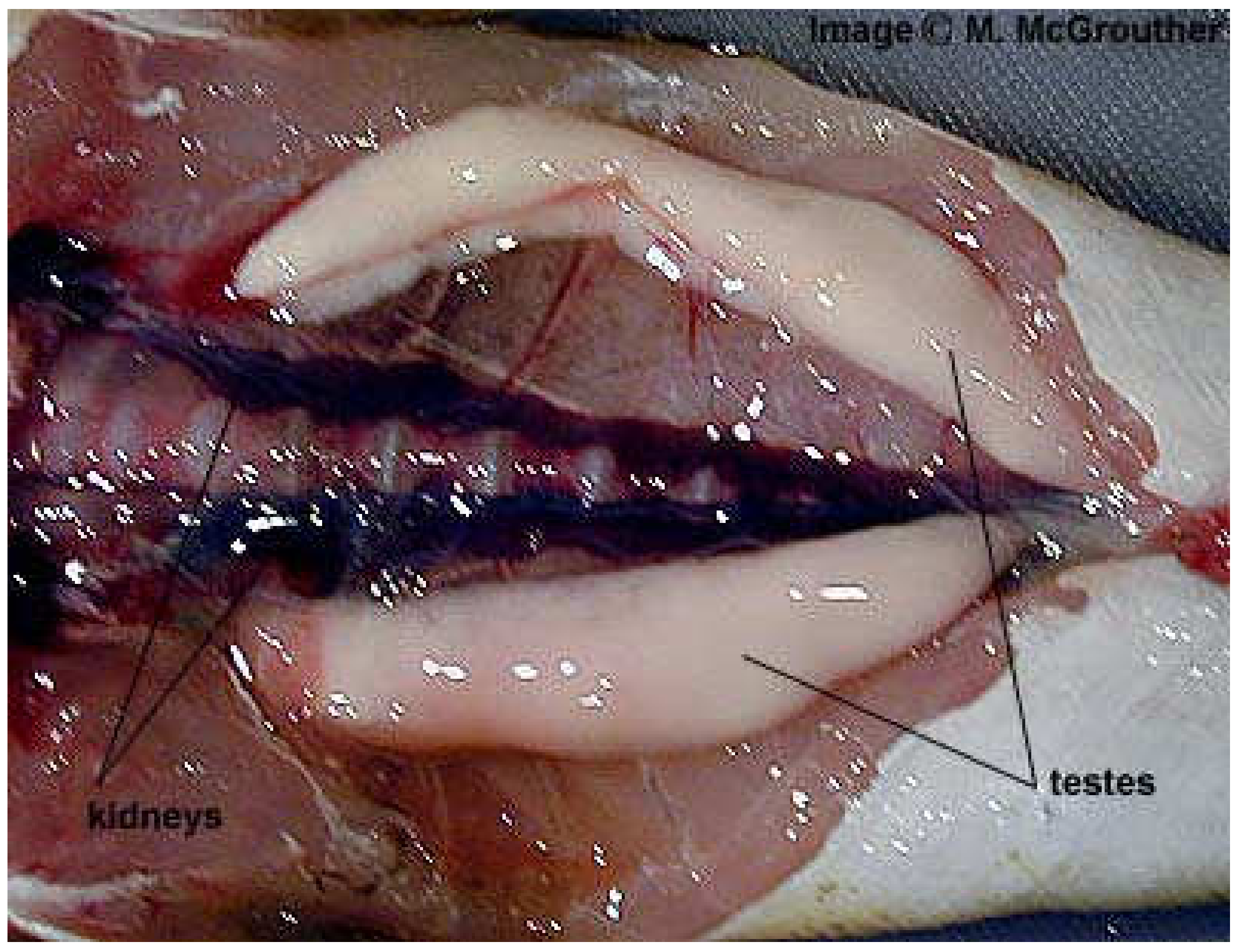
INTERNAL ANATOMY



Identify the gastrointestinal tract (Figures 1i-1,2). Pass a blunt probe through the oral cavity, pharynx, esophagus and into the stomach. Many fish species have pyloric cecae, which are blind sacs projecting from the aborad portion of the stomach. The stomach empties into the intestine, a long tubular structure supported by thin membranes called mesenteries. The intestine terminates at the anus. In fish the intestine is not divided into three distinct regions. The length and complexity of the intestine is directly proportional to the amount of plant matter consumed (herbivorous species have longer intestines). Open the stomach and intestines and note the normal texture and appearance of the lining, or mucosa. The intestinal mucosa will often exhibit lesions when enteric of systemic disease is present. The spleen is a small dark red organ attached to the mesenteries just caudal to the stomach. There may be more than one spleen. The main auxiliary digestive organs are the liver and pancreas. The liver is a large, tan, often leaf-shaped organ just caudal to the heart. The liver is a good site to see many lesions and is also a good site from which to isolate bacterial and viral pathogens. The location and size of the pancreas varies by species. The most common location is interspersed within the liver parenchyma. It may or may not be grossly visible. Cut the intestine near the anus, cut the esophagus and remove the gastrointestinal tract.



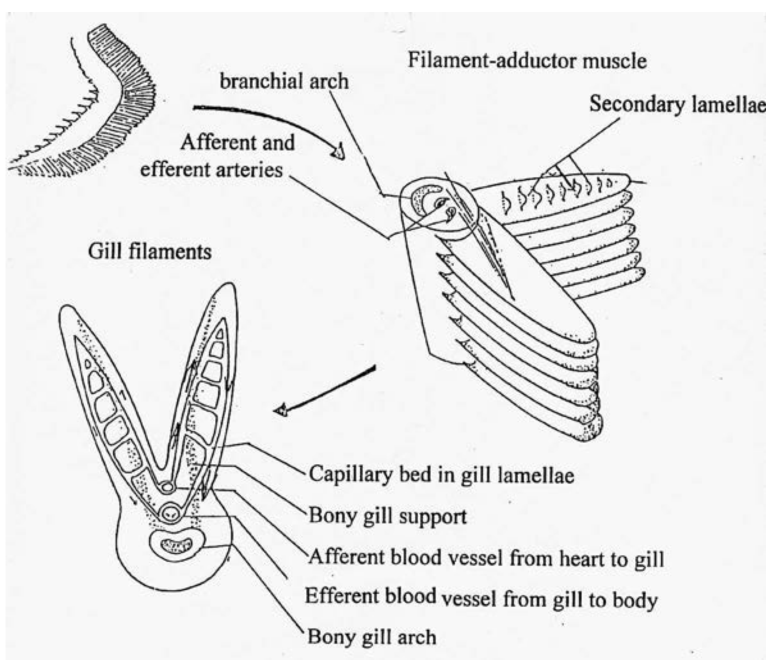
7. Locate the gonads, either ovaries or testes. Ovaries will appear as numerous spherical structures that may comprise up to 70% of body weight. Testes may comprise up to12% of body weight. In mature animals they will appear as a soft white organ suspended from the dorsal body wall. Also, if you don't see either of these organs, you might be working with an immature specimen. Note body length and compare to literature on the species/specimen you are working with.



The gonads and kidneys of an Eastern Blue-spotted Flathead. The gonads (testes) are the large, pale organs and the kidneys are the red tissue either side of the backbone.

8. Along the dorsum of the body cavity lies the swim bladder. It is a thick-walled white organ. Occasionally you may see hemorrhages in the swim bladder.

9. The kidneys also lie in the dorsum of the body cavity. The head kidney and trunk kidney are roughly divided by the swim bladder. In some species (e.g., salmonids) the kidneys are almost fused. The kidneys often exhibit lesions, and the trunk kidney is usually the preferred site for obtaining bacterial and viral cultures. In most fish we work with in this lab, the head kidney and trunk kidney appear fused.



10. The heart lies just caudal to the gills (return to previous figure, Figure 1i). The heart is enclosed in a thin-walled sac, the pericardium. Open the pericardium and examine the heart in situ. Blood returns from the body wall to the sinus venosus, a thin-walled chamber which empties into the atrium. The sinus venosus might be difficult to identify. The atrium pumps blood to the ventricle. The atrium lies cranial and dorsal to the ventricle. The ventricle is the main pump and largest part of the heart. Blood flows from the ventricle craniad to the bulbus arteriosus. The thick-walled elastic bulbus helps regulate blood pressure as blood leaves the heart. As the bulbus passes through the pericardium en route to the gills it becomes the ventral aorta.

Attachment H-8

Extraction and Cleanup of PCBs from Fish and Biota Materials

STANDARD OPERATING PROCEDURE

EXTRACTION AND CLEANUP OF FISH AND BIOTA MATERIALS

Reference Methods: EPA Method 3540C & EPA Method 8082

LOCAL SOP NUMBER:	NE017_09
EFFECTIVE DATE:	03/25/2011
SUPERSEDES:	NE017_08
SOP TEMPLATE NUMBER:	SOT-ALL-Q-006-rev.03

APPROVALS

Dan Pfalzer
Assistant General Manager

03/25/2011
Date

Christina L. Braidwood
Quality Manager

03/25/2011
Date

PERIODIC REVIEW

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

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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STANDARD OPERATING PROCEDURE

LABORATORY PROCEDURE NE017_09.DOC

REVISION 9 (03/25/2011)

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1.0 IDENTIFICATION OF TEST METHOD

1.1 This is a Standard Operating Procedure for the extraction and cleanup of polychlorinated biphenyls from fish and biota material by SW-846 Method 3540 (Soxhlet Extraction).

2.0 APPLICABLE MATRIX AND MATRICES

2.1 This test method is appropriate for all fish and biota material. The extraction technician must have an understanding of the methods and requirements of USEPA-SW- 846A "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition. Methods 3540, 3500, 2500A.

2.2 An approved instructor must also certify the extraction technician for performing the procedure. The extraction technician should have completed an acceptable demonstration of precision and accuracy before performing this method without supervision. This method may be restricted to use by or under the supervision of an extraction technician knowledgeable in the area of PCB extraction and clean-up. The extraction technician should further be aware of the proper care and handling of PCBs as well. Refer to MSDS for details.

3.0 DETECTION LIMIT

3.1 See determinative method (SOP NE148) for details.

4.0 SCOPE AND APPLICATION

4.1 The following procedure is used by Pace Analytical for the extraction of fish tissue for PCB analysis, although it may be adapted for other biota extractions.

5.0 SUMMARY OF TEST METHOD

5.1 Samples are initially dried utilizing 1:1 magnesium:sodium sulfate.

5.2 The samples are then loaded into pre rinsed cellulose extraction thimbles. These loaded thimbles are then placed into soxhlet extractor apparatus where they are spiked and surrogated.

5.3 After an 18 ± 2 hour extraction the derived solvent is exchanged to pure hexane via a TurboVap evaporator system.

5.4 The extract is set to volume and put through a clean up. This cleanup is composed of several steps including acid, TBA, Florisil, and mercury.

5.5 The extract is then properly diluted and submitted for GC analysis.

6.0 DEFINITIONS

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

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

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

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

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

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

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

7.0 INTERFERENCES

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

8.0 SAFETY

8.1 Polychlorinated biphenyls should be treated with extreme caution; as a class of chemical

compounds they possess both toxic and suspected carcinogenic properties. Refer to MSDS for further details.

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

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

9.0 EQUIPMENT AND SUPPLIES

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

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

9.3 Soxhlet Repetitive Flushing (reflux) Unit: 45/50 Pyrex #3740-M (or equivalent)

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

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

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

9.7 Cellulose Extraction Thimble: Contains sample during Soxhlet extraction.

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

9.9 TurboVap Evaporator: Zymark #ZW640-3.

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

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

9.12 Vials: glass, 40mL & 4 dram (with polyseal sealed cap) (20 mL & 10 mL) capacity, for sample extracts.

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

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

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

9.16 Pipettes: S/P Disposable Serological Borosilicate Pipettes.

1. 1mL X 1/10
2. 5mL X 1/10
3. 10mL X 1/10
4. Fisher Pasteur Borosilicate glass pipette 9" #72050 (or equivalent)

9.17 Aluminum weigh dishes: Eagle Thermo Plastics, #64-D70-100-PK (or equivalent)

9.18 4oz. Jars: Industrial Glassware

10.0 REAGENTS AND STANDARDS

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

10.2 Boiling Chips: Chemware PTFE Boiling Stones, P#0919120 (or equivalent)

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

10.4 Acetone: High Purity Solvent Baxter (Burdick/Jackson) #UN1090. (or equivalent)

10.5 1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.

10.6 Florisil: deactivated, Tested and Approved, see NE283.

10.7 TBA Reagent: Tetrabutylammonium Hydrogen-Sulfite Reagent (prepared in the laboratory)

10.8 Mercury: Triple distilled Mercury Waste Solutions, Inc. (or equivalent)

10.9 Sulfuric Acid: Na_2SO_4 (concentrated) Malinkrodt #2468 #UN1830. (or equivalent)

10.10 1:1 Magnesium Sulfate/Sodium Sulfate: 1:1 by volume, mixture prepared in the lab.

10.11 Sodium Sulfite: Sigma – Aldrich, #23932-1 (or equivalent)

10.12 Propanol: JT Baker, #11-9095-03 (or equivalent)

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT and STORAGE

11.1 All samples should remain frozen at all times unless being tested. Fish usually arrive whole bodied or already filleted. Once received, the sample must be ground and homogenized so that it may be analyzed. The homogenized fish tissue should be held for 6 to 12 months.

11.2 The fish solvent extracts should be held for 3 months. Some clients may request that the body and/or head of fish be saved once the fillets are cut out. Other biota material may have other specifications stated specifically for that project.

12.0 QUALITY CONTROL

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

12.2 A method blank sample and lab control spike must be prepared per each extraction batch or 1 per 20 site samples whichever is more frequent. A matrix spike/lab duplicate should be prepared for every 20 site samples or as per client specified quality assurance project plan (QAPP). The spike default for LCS, MS is 1.0mL of A1242 @ 12.5ppm. Client and/or project specifications may dictate alternate amount or Aroclor.

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

13.0 CALIBRATION AND STANDARDIZATION

13.1 The analytical balance should be calibrated daily to ensure accurate measurements are made when weighing out samples for extraction.

13.2 Please see determinative method (SOP NE148) for details.

14.0 PROCEDURES

14.1 Sample Preparation

14.1.1 Throughout the entire process it should be noted that if the extraction technician encounters any problems or difficulties with any samples or steps involved, all work should <u>STOP!</u> Any problems should be brought to the attention of the supervisor and documented in LIMS.

14.1.2 The extraction technician should match each sample container label to the chain of custody identification number which is in the job folder.

14.1.3 The fish samples are usually received as fillets and must be processed to produce a homogenous material prior to extraction. Once the sample has been logged into LIMS, the sample is processed according to SOP NE132.

14.1.4 The sample is then placed into the freezer (R-4) for storage until the extraction process is started.

14.2 Procedure: Sample Extraction

14.2.1 Rinse enough cellulose extraction thimbles for one per sample and QC sample with Hexane. Allow them to dry out in a 4oz. jar in a fume hood.

14.2.2 Fill a Pyrex pan with ice cubes and cold water to about a 1/2 inch deep.

14.2.3 Into a pre-cleaned and tared 4oz. jar, accurately weigh to the nearest 0.001g, using an analytical balance, between 10-11 grams of homogenized tissue with a metal spatula. Record this weight in LIMS. Place the jar containing the sample into the Pyrex pan to chill. Repeat for remaining samples.

Note: All sample containers are to be returned to the appropriate refrigerator / freezer. For all empty containers, see the Chemical Hygiene Plan for proper disposal.

14.2.4 After the sample has been sufficiently chilled, add enough 1:1 mix of magnesium sulfate/sodium sulfate to sufficiently cover the sample and mix well with a metal spatula. If the sample has not dried after a few minutes, more 1:1 magnesium sulfate/sodium sulfate may be added. Once the sample is well-dried and free flowing, transfer the sample to an extraction thimble using the same metal spatula.

Note: Be careful not to add too much drying agent to the sample, if too much is added, the sample may not fit completely into the thimble. In this case the sample will have to be split into two separate Soxhlet apparatus set-ups and re-combined following extraction.

14.2.5 Add several boiling chips to a 250mL round bottom flask. Then add 200mL of a 1:1 Hexane/Acetone mixture. Place a Soxhlet extractor onto the round bottom flask, checking for cracks or chips that would cause solvent to leak out.

14.2.6 Label the round bottom flask with the sample number and place the corresponding thimble into the Soxhlet extractor using long tweezers. Record the round bottom and soxhlet number in LIMS. Repeat for each sample, rinsing tweezers between samples.

14.2.7 Spike surrogate and spike compound solutions directly into the thimble in the soxhlet. The addition of spiking material to a sample, blank, or QC must be witnessed by another extraction technician. Record the names of the technicians spiking and witnessing, surrogate and spike concentration, the amount spiked, and the spike solution reference code in LIMS.

14.2.8 Rinse the inside and the outside connecting joints of the condenser units that will be used with hexane. Then, turn on chiller to cool the condensers. Chiller should be set to approximately 12°C.

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

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

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

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

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

14.2.14 Rinse the soxhlet with several pipette volumes of Hexane and tip again to drain into the round bottom. Set the soxhlet aside at this time.

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

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

14.2.17 All glassware must be rinsed with technical grade (tech)-Acetone or a "for rinsing-only" labeled solvent, and dried in the hood before other cleaning steps.

14.3 Solvent Reduction: TurboVap Evaporator System

14.3.1 The TurboVap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The TurboVap evaporator system is used to reduce the sample volume. The TurboVap uses a heated water bath and positive pressure nitrogen flow / vortex action. The unit maintains a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract, which allows fractional reduction of the solvents without loss of higher boiling point analytes.

14.3.2 Turn the unit on and allow to heat up to 40°C ± 2°C.

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

14.3.4 Wipe down inside of TurboVap with a Hexane wetted paper towel including top lid and pins. Place TurboTubes containing the sample extracts into the TurboVap and close lid. Slowly open the pressure regulator. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces, maintaining uniform flow throughout the volume reduction.

14.3.5 The process for solvent (Hexane/Acetone) reduction takes approximately 20-30minutes. Do not leave the unit unattended as extracts may be blown to dryness and PCB loss may occur. Immediately notify a supervisor if an extract is blown to dryness.

14.3.6 Concentrate the solvent to approximately 10 mL. Remove the samples from the TurboVap and place in the rack. The remaining solvent will consist largely of Hexane since the Acetone component is fractionally removed at a faster rate than Hexane. However, a solvent exchange with Hexane should be completed to ensure the Acetone has been entirely removed.

14.3.7 Fill the turbo tube back up to approximately 100mLs with hexane. Concentrate the solvent back to 10mls. Then remove the turbo tube and place in a rack.

NOTE: Not all samples will evaporate at the same rate; sample extracts containing large amounts of petroleum or other non-volatile liquids may stop reducing before the 10.0 mL point is achieved. Samples, which stop reducing, should be removed as soon as possible.

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

14.3.9 All dirty glassware must be rinsed with Tech-grade Acetone or a "For Rinsing-Only" labeled solvent and dried in the fume hood before being washed.

14.4 % Lipid Analysis

14.4.1 Label one aluminum weigh dish for each sample. Using an analytical balance accurately weigh each aluminum dish to the nearest 0.0001g and record in LIMS.

14.4.2 Using a pipette, remove 10mLs of the 25x sample completed in step 14.3.8, and place in the aluminum dish.

14.4.3 Set the dishes aside in the chemical fume hood for solvent evaporation. These need to be set in an area where they will not be disturbed. Be careful not to work over the aluminum dishes. Doing so may get particles into the dishes and affect the results of the % total lipids.

14.4.4 Once dry, place the dish in a dessicator designated specifically for % lipids. The dishes need to stay in the dessicator for a minimum of 4 hours.

14.4.5 After the four hours, weigh the dish a final time using the same analytical balance, and enter the weight into LIMS.

14.4.6 See Section 15.1 for instructions on calculating % lipids.

14.5 Sample Extract Cleanup

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

14.5.2 Not all clean-up procedures need to be performed on every sample and several are sample matrix specific. The experience of the analyst combined with the sampling site history should guide the selection of which clean-up procedures are necessary. The sample preparation chemist records the sequence and number of repeats of cleanup steps performed. Sample extract cleanups are performed on set volume extracts. The default set volume is 25mL for biota samples.

14.5.3 Sample extract cleanups are performed on set volume extracts. The default set volume is 25 mL for biota samples. Biota samples are normally cleaned up in this order: Sulfuric Acid, Florisil slurry, and then Hg shake.

14.5.4 Sulfuric Acid Wash

14.5.4.1 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds, which are co-extracted with the PCB residues.

14.5.4.2 Chill the sample in the freezer for 10-20 minutes. Add 10mL concentrated H_2S0_4 and slowly rotate back and forth by hand. Sulfuric acid reacts strongly with the lipids so these samples should **NOT** be vigorously shaken. Centrifuge for approximately 2 minute on a speed setting of ¾.

14.5.4.3 Transfer the Hexane layer (upper layer) to a new, labeled 40mL vial.

14.5.4.4 Repeat section 14.5.4.2 if the sample extract appears to be heavily loaded (opaque) with colored material. Two to three acid washes may be required.

Note: It is entirely possible that all colored material will not be removed from the extract.

14.5.5 Florisil Adsorption (Slurry)

14.5.5.1 The Florisil slurry removes co-extracted polar compounds, residual water, and residual acid and is recommended as the final cleanup step before the extract is submitted for GC analysis.

14.5.5.2 Add approximately 3 grams of tested and approved 10% deactivated Florisil® to each vial containing the sample extract.

14.5.5.3 Vigorously shake the vial for approximately 1 min by hand or on the wrist shaker. Swirl to get any Florisil off the walls of the vial, and then allow to settle.

14.5.5.4 Transfer the hexane layer to a clean 40mL vial.

14.5.6 Elemental Sulfur Clean-up

14.5.6.1 Elemental sulfur is soluble in the extract solvents used for biota samples. It is commonly found in many sediment/soil samples, decaying organic material, and some industrial wastes. Large amounts of sulfur can cause the electron capture detector (ECD) to signal saturate for long periods during the elution envelope of PCBs. Even small amounts of sulfur can interfere with PCB measurement as a co-eluting chromatographic peak.

14.5.6.2 Two techniques exist for the elimination of elemental sulfur in PCB extracts. Mercuric precipitation (Mercury Shake) and the Tetrabutylammonium (TBA) sulfite procedure. Tetrabutylammonium sulfite causes the least amount of degradation of a broad range of pesticides and organics compounds, while mercury may degrade organophosphorus and some organochlorine pesticides. The TBA procedure also has a higher capacity for samples containing high concentrations of elemental sulfur.

14.5.7 Removal of Sulfur Using Mercury

14.5.7.1 Add 1-3 drops of mercury to the sample extracts, cap vial, and place on the wrist shaker for 30 min. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental sulfur.

14.5.7.2 Transfer the sample extract to a clean and properly labeled 40mL vial.

14.5.7.3 The precipitated sulfur can be removed from the extract by performing a sulfuric acid clean-up or Florisil slurry (**Section 14.5.4 and 14.5.5**).

14.5.8 Removal of Sulfur using TBA Sulfite

14.5.8.1 The TBA procedure removes elemental sulfur by converting it to the thiosulfate ion, which is water soluble.

14.5.8.2 Add 2.0mL TBA Sulfite Reagent, 1.0 mL 2-propanol, and approximately 0.65g of sodium sulfite crystals to the extract and shake for at least 5 minutes on the wrist shaker and observe. An excess of sodium sulfite must remain in the sample extract during the procedure. If the sodium sulfite crystals are entirely consumed add one or two more aliquots (approximately 0.65g) to the extract and observe.

14.5.8.3 Place the samples on the wrist shaker for 45 minutes observing at 15 minute intervals to make sure that the sodium sulfite is not consumed.

14.5.8.4 After 45 minutes, add 5mL organic free water (DI water) and shake for ten additional minutes on the wrist shaker.

14.5.8.5 Place the samples into the centrifuge and spin for approximately 2 minutes on a speed setting of ¾.

14.5.8.6 Transfer the Hexane (top) layer to a new and properly labeled 40mL vial and cap.

14.6 Extract Screening and Dilution:

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

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

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

14.6.4 Transfer 1mL of the extract to a labeled 1.5mL GC autosampler vial. Record the

sample data in LIMS and submit with the sample extracts to the GC analyst.

15.0 CALCULATIONS

15.1 All fish samples require a % lipid analysis, see section 14.4. The lipid content is calculated utilizing the below equation:

$$\% \text{ Lipids} = \frac{(\text{Final Weight} - \text{Initial Weight})}{\text{Sample Weight}} \times \frac{\text{Whole Volume (25ml)}}{\text{Extracted Volume (10ml)}} \times 100$$

15.2 Please see determinative method (SOP NE148) for details.

16.0 METHOD PERFORMANCE

16.1 Please see determinative method (SOP NE148) for details.

17.0 POLLUTION PREVENTION

17.1 See NEA168 for proper pollution prevention procedures.

18.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

18.1 Please see determinative method (SOP NE148) for details.

19.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

19.1 Please see determinative method (SOP NE148) for details.

20.0 CONTINGINCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1 Please see determinative method (SOP NE148) for details.

21.0 WASTE MANAGEMENT

21.1 See NE054, NE083, and NE089.

22.0 REFERENCES

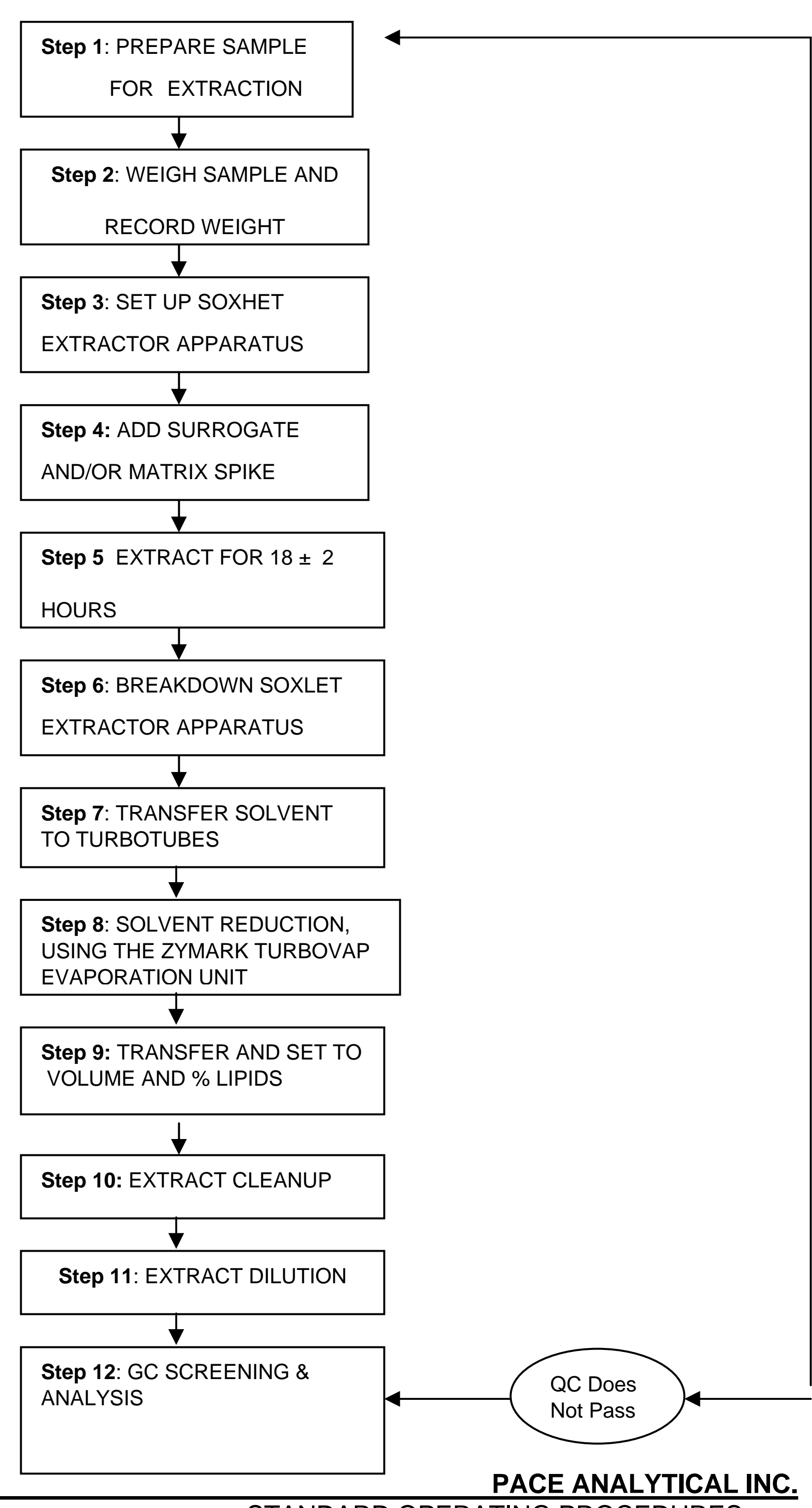
22.1 SW-846 methods 3500A & 3600A; United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Vol.1B, Cincinnati, OH 45268.

22.2 Guide to Environmental Analytical Methods, Genium Publishing Corporation, Schenectady, NY 12304.

23.0 TABLES, DIAGRAMS, FLOWCHARTS and VALIDATION DATA

23.1 Attachment A: Flowchart for the Extraction and Clean-up of Fish and Biota Materials for PCB Analysis

ATTACHMENT A: FLOWCHART FOR THE EXTRACTION AND CLEAN-UP OF FISH AND BIOTA MATERIALS FOR PCB ANALYSIS



STANDARD OPERATING PROCEDURE REVIEW

SOP Name	Review Number	Reviewers	Title	QAO Approval	Effective Date
NE017_08	00	Carrie Barss Christina L. Braidwood Robert E. Wagner	Extractions Supervisor QAO Lab Director	Christina Braidwood	11/25/08
NE017_09	00	Jill Grygas Christina L. Braidwood Robert E. Wagner	Extractions Supervisor QAO Lab Director	Christina Braidwood	03/25/11

Attachment H-9

Congener Specific PCB Analysis

STANDARD OPERATING PROCEDURE

CONGENER SPECIFIC POLYCHLORINATED BIPHENYL (PCB) ANALYSIS

Reference Methods: GREEN BAY MASS BALANCE METHOD

LOCAL SOP NUMBER:	NE013_10
EFFECTIVE DATE:	03/24/2011
SUPERSEDES:	NE013_09
SOP TEMPLATE NUMBER:	SOT-ALL-Q-006-rev.03

APPROVALS

Dan Pfalzer — 03/24/2011

Dan Pfalzer
Assistant General Manager — Date

Christina L. Braidwood — 03/24/2011

Christina L. Braidwood
Quality Manager — Date

PERIODIC REVIEW

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

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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STANDARD OPERATING PROCEDURE

LABORATORY PROCEDURE NE013_10.DOC

REVISION 10 (03/24/2011)

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1.0 Identification of Test Method

1.1 Congener Specific Method for Congener-Specific Polychlorinated Biphenyl (PCB) Quantification and Identification by Capillary Column/Gas Chromatography with Electron Capture Detection.

2.0 Applicable Matrix

2.1 This method is applicable for the determination of PCB Congeners in soils, sediments, biota, oils, water and other solid and liquid matrices.

3.0 Detection Limit

3.1 Detection Limit: Reporting Limits (RLs) and Method Detection Limits (MDLs) vary for each matrix and are based on total PCB concentration.

3.2 MDL and RL are the following:

3.2.1 Aqueous matrix, 1L final volume 5mL, MDL = 9.34ng/L Total PCB ; RL 32.3ng/L Total PCB.

3.2.2 Solid Matrix: 10g final volume to 25mL, MDL = 0.0135 ug/g Total PCB ; RL 0.312ug/g Total PCB.

3.3 Individual peak MDLs and RLs are determined every two years with matrix specific MDL studies or as required when a major equipment change occurs. See Appendix E for MDL values.

4.0 Scope and application, including components to be analyzed

4.1 This method is applicable in the determination and quantification of Polychlorinated Biphenyls (PCB) in sediments, soils, biota, oils, water, and other solid and liquid matrices. This method is a congener-specific determination, employing a high resolution fused-silica capillary chromatographic column. The method has been, in part, developed from the following documents:

4.2 "Quality Assurance Plan, Green Bay Mass Balance Study, 1. PCBs and Dieldrin, US EPA Great Lakes National Program Office", prepared by Deborah L. Swackhamer, Quality Assurance Coordinator, Field and Analytical Methods Committees, University of Minnesota, December 11, 1987. This document outlines quality assurance and quality control procedures to be followed by laboratories participating in the Green Bay Mass Balance Study. Where applicable, Northeast Analytical, Inc., will incorporate and utilize this information in quality control of data generated. Instrumental analysis and conditions (Mullin, M.D., 1985, PCB Workshop, US EPA Large Lakes Research Station, Gross Ile, MI, June.) cited in the Green Bay Mass Balance Study document will be refined to be applicable to an in-house data management software package.

4.3 "Comprehensive, Quantitative, Congener-Specific Analyses of Eight Aroclors and Complete PCB Congener Assignments on DB-1 Capillary GC Columns", George M. Frame, Robert E. Wagner, James C. Carnahan, John F. Brown, Jr., Ralph J. May, Lynn A. Smullen, and Donna L. Bedard, Chemosphere, Vol. 33, No. 4, pp. 603-623, 1996. This journal publication provides complete assignment of all 209 PCB congeners to the GC peaks separable on a DB-1 capillary column. It also provides weight percent information for PCB congeners in Aroclor formulations used in labeling protocols for reporting purposes.

4.4 "Standard Operating Procedure for the Gas Chromatographic Analysis of Hydrophobic Organic Contaminant Extracts from Great Lakes Water Samples", US EPA Great Lakes National Program Office, 77 West Jackson Boulevard, Chicago, IL 60604-2590, GLNPO Organics SOP – 10, 6/1/94:Revision 2. This USEPA SOP summarizes M. Mullin's congener composition of the mixed Aroclor standard used for calibration.

4.5 "Biphenyls and Halogenated Pesticides by High Resolution Gas Chromatography", M.D. Mullin, Large Lakes Research Station, LLRS-SOP-ORG-013, revision 2, August 3, 1990, p 1-10. This LLRS SOP written by M. Mullin summarizes the

calibration composition of the mixed Aroclor standard reported in "Mullin, M.D., PCB Workshop, U.S. EPA Large Lakes Research Station, Grosse Ile, MI, June 1985.

4.6 This gas chromatographic capillary column method, utilizing an electron capture detector, will effectively separate 112 or more peaks representing 209 PCB congeners.

5.0 Summary of Test Method

5.1 This method provides detailed instructions for gas chromatographic conditions for the analysis of PCBs by capillary gas chromatography.

5.2 This method utilizes a mixed Aroclor standard (Aroclor 1232/1248/1262 in the ratio of 25:18:18) for calibration. Method detection limit and practical quantitation limit will be established experimentally using the procedure in USEPA 40 CFR, Part 136, App.B.

5.3 In general, samples are first extracted with a pesticide-grade solvent. The extracts are further processed through a series of clean-up techniques. The sample is then analyzed by direct liquid injection onto the gas chromatographic column and detected by an electron capture detector. This method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.

5.4 A key component of this method is the importance placed on the chromatographic separation that must be achieved for this congener specific technique. A total of 112 chromatographic peaks are detected, containing 209 PCB congeners in various ratios. This allows an almost complete profile of environmentally occurring PCBs.

6.0 Definitions

6.1 Accuracy – The nearness of a result or the mean of a set to the true value. Accuracy is assessed by analysis of reference samples and measurement of percent recoveries.

6.2 Analytical Batch –The basic unit for analytical quality control is the analytical batch, which is defined as samples which are analyzed together with the sample method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar matrices (e.g. water, sediment, soil, etc.).

6.3 Aroclor – Polychlorinated Biphenyls (PCBs) were commercially produced for a variety of uses including, transformers, capacitors, inks, paints, dust control agents, and pesticides. Monsanto Corporation was a major producer and sold PCBs under the trade name Aroclor.

6.4 Blank – A blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix, however, a universal blank matrix does not exist for solid samples, but sometimes sodium sulfate is used as a blank matrix. The blank is taken through the appropriate steps of the process. A reagent blank is an aliquot of analyte-free water or solvent analyzed with the analytical batch. Field blanks are aliquots of analyte-free water or solvents brought to the field is sealed containers and transported back to the laboratory with the sample containers. Trip blanks and equipment blanks are two specific types of field blanks. Trip blanks are not opened in the field. They are a check on sample contamination originating from sample transport, shipping and from site conditions. Equipment blanks are opened in the field and the contents are poured appropriately over or through the sample collection device, collected in a sample container, returned to the laboratory as a sample. Equipment blanks are a check on sampling device cleanliness.

6.5 Calibration Standard (ICAL)– A series of known standard solutions used by the analyst for instrument calibration. Calibration standards are prepared from primary standard and/or stock standard solutions.

6.6 Congener – Any of the 209 Chlorinated Biphenyl compounds of varying degree of chlorination, mono through decachlorobiphenyl.

6.7 Continuing Calibration Check Standard(CC) –The continuing calibration check standard contains all the target analytes found in the calibration standards and is used to verify that the initial calibration is prepared correctly and that the

instrument system is correctly calibrated. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards (not independent for CQCS-PCB).

6.8 CAS Number – An assigned number used to identify a chemical. CAS stands for Chemical Abstracts Service, an organization that indexes information published in Chemical Abstracts by the American Chemical Society and that provides index guides by which information about particular substances may be located in the abstracts. Sequentially assigned CAS numbers identify specific chemicals, except when followed by an asterisk (*) which signifies a compound (often naturally occurring) of variable composition. The numbers have no chemical significance. The CAS number is a concise, unique means of material identification. (Chemical Abstracts Service, Division of American Chemical Society, Box 3012, Columbus, OH 43210: [614] 447-3600).

6.9 Duplicate– A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

6.10 Environmental Sample – An environmental sample or field sample is a representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination as requested or required. Environmental samples are normally classified as follows:

6.10.1 Drinking water -delivered (treated or untreated) water designated as potable water.

6.10.2 Water/Wastewater -raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluent.

6.10.3 Sludge -municipal sludge and industrial sludge.

6.10.4 Waste –aqueous and non-aqueous liquid wastes, chemical solids, contaminated soils, and industrial liquid and solid wastes.

6.11 Homolog – Any of the ten sets of PCB congeners of the same degree of chlorination.

6.12 Initial Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the analytical detector or method.

6.13 Instrument Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

6.14 Laboratory Control Sample (LCS) – Also known as the Quality Control (QC) Check Standard or Quality Control (QC) Check Sample. The LCS consists of an aliquot or reagent water or other blank matrix to which known quantities of the method analytes are added. The LCS is extracted and analyzed exactly like a field sample, and its purpose is to determine whether the analysis is in control and whether the laboratory is capable of making accurate and precise measurements.

6.15 Laboratory Method Blank – An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

6.16 Matrix – The predominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

6.17 Matrix Spike – Aliquot of sample (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

6.18 Matrix Spike Duplicate – A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

6.19 Method Detection Limit (MDL) – The minimum constituent concentration that can be measured and reported with 99% confidence that the signal produced is different from the blank in a given matrix. The MDL is determined from a minimum

of seven replicate samples, taken through the entire preparation and analysis procedure. The standard deviation, *s*, of those replicates is multiplied by a student's t factor in order to calculate the MDL.

6.20 MSDS – Material Safety Data Sheet. OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs.

6.21 PCB – Polychlorinated Biphenyl (PCBs) are a class of 209 individual chemical compounds (congeners), in which one to ten chlorine atoms are attached to biphenyl. Use of PCBs has made them a ubiquitous environmental pollutant.

6.22 Practical Quantitation Limit (PQL) – The minimum constituent concentration that can be determined with 95% confidence to be at the value stated. It is calculated by multiplying three to five times the MDL.

6.23 Precision – The agreement between a set of replicate measurements without assumption of knowledge of the true value. Precision is assessed by means of duplicate/replicate sample analysis.

6.24 Primary Standard Solution – A solution of several analytes prepared from stock solutions that can be diluted as needed to prepare calibration solutions and to prepare other standard solutions.

6.25 Quality Control – Set of measures within a sample analysis methodology to assure that the process is in control.

6.26 Sample Matrix Spike/Sample Matrix Spike Duplicate (MS/MSD) – An aliquot of a field sample that is fortified with known quantities of the method analytes and subjected to the entire analytical procedure. Its purpose is to assess the appropriateness of the method for the matrix by measuring recovery.

6.27 Standard Curve – A standard curve is a curve which plots concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by diluting the stock analyte solution in graduated amounts which cover the expected range of the samples being analyzed. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

6.28 Stock Solution – Standard solution which can be diluted to derive the other standards.

6.29 Surrogate – Organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, calibration and check standards, samples (including duplicates and QC reference sample) and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

6.30 Surrogate Standard – A pure compound added to a sample in the laboratory just before processing so that the overall efficiency of a method can be determined.

7.0 Interferences

7.1 One of the major sources of interference in the analysis of PCBs is co-extracted organochlorine pesticides. Several of the commonly found pesticides and associated pesticide degradation products (e.g. PP-DDT, PP-DDE, PP-DDD) overlap the PCB profile envelope and co-elute with several PCB peaks causing inaccurate measurement. The analyst must be careful to review the chromatographic pattern and peak retention times and flag peaks that are suspected of containing interference so that they are not included in the total PCB values generated. Rigorous cleanup of sample extracts (i.e. sulfuric acid cleanup and florisil cleanup) removes many non-target pesticides.

7.2 Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters exhibit response on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Laboratory method blanks must be thoroughly reviewed for presence of non-target peaks and comparison of samples with blank chromatographic patterns.

7.3 The sample matrix itself is also a potential source for method analyte interference. Sample preparation, extraction procedures, and extract clean-up protocols are covered in separate SOPs that deal exclusively with sample extraction.

8.0 Safety

8.1 Safety glasses and disposable gloves must be worn when handling samples and extracts.

8.2 All manipulations of sample extracts should be conducted inside a chemical fume hood. Manipulation of sample extracts outside of a fume hood should be minimized by the analyst.

8.3 Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize themselves with the precautions for handling solvents and chemicals used to process samples. The analyst should refer to the laboratory chemical hygiene plan for further safety information.

8.4 Samples remaining after analysis should either be returned to the customer for disposal or disposed of through the laboratory's disposal plan. Refer to the sample custodian for assistance and also standard operating procedure NEO54, disposal of laboratory waste.

9.0 Equipment and Supplies

9.1 Gas Chromatograph: Complete system for high resolution, capillary column capability and all required accessories. Northeast Analytical, Inc. will use an Agilent Model 6890 gas chromatograph (or equivalent), equipped with capillary split/splitless injector (or equivalent), temperature programmable oven, Model 7683 automatic sampler (or equivalent), and micro-electron capture detector (or equivalent). A data system and integration of detector signal is interfaced to the gas chromatograph.

9.2 Chromatographic Data System: A data system for measuring peak height and peak area. An Empower computer network based workstation (Waters Corporation), will be employed to capture detector response and digitally store the chromatographic, electronic peak integration for precise calculations, database structuring of the analytical information, and archival capabilities.

9.3 GC Column: The gas chromatograph column to be used for analysis will be a DB-1 (J&W Company) or a ZB-1 (Phenomenex), bonded polydimethylsilicone, 30 meter fused silica capillary column with an internal diameter of 0.25mm and phase coating thickness of 0.25 microns. This column is capable of resolving 112 chromatographic peaks from the full spectrum of all PCB congeners that could be expected in an environmental sample. Refer to Appendix A and Appendix B for a complete description of PCB congeners identified in each GC chromatographic peak and achievable chromatographic separations.

9.4 Volumetric Flasks – 10, 50 and 100mL, ground-glass stopper. For standard preparation.

9.5 Micro syringe – 10uL for internal standard addition.

9.6 Hamilton Syringes – 50, 100, 250, 500, 1000 and 2000uL for standard preparation.

9.7 Class A volumetric pipettes – 1mL, 5mL, 10mL

9.8 Pasteur pipettes.

9.9 250ml and 100ml beakers, glass.

9.10 Disposable 1.0, 5.0, and 10.0 ml pipettes.

9.11 Vials – Glass, 10 and 20mL capacity for sample extracts.

9.12 Bottles – Glass , 120mL capacity for standard storage.

9.11 Autosampler Vials – 2ml capacity vials with PTFE septa.

9.12 Ferrules: 0.4mm graphite/vespel, Agilent or equivalent.

9.13 Injector septa – Restek Ice Blue Septa or equivalent.

9.14 Injector liner – Agilent Split/Splitless Liner w/ glass wool or equivalent.

9.15 Viton Fluorocarbon O-ring – Agilent or equivalent.

9.16 Autosampler Vials – 2ml capacity vials with PTFE septa.

10.0 Reagents and Standards

10.1 Solvents – Pesticide grade quality. Hexane, Acetone, Toluene, Methylene chloride. Obtained form Burdick & Jackson or equivalent.

10.2 Octachloronaphthalene (Internal Standard): Obtained from Ultra Scientific (Hope, RI) or AccuStandard with purity greater than 95%.

10.3 Polychlorinated Biphenyls: Neat commercial material or solutions for standard preparation. These materials are multi-component mixtures of PCB congeners and are the actual materials that were used in products such as transformers and capacitors. Monsanto was the largest producer of PCB formulations and sold them under the trade name Aroclor.

10.4 PCB Congeners: A complete set of all 209 PCB congeners to individually verify the exact elution order on the chosen chromatographic system. A subset of congeners are also used as a secondary supplemental calibration standard for those congeners that do not exist at a high enough level in the Aroclor based calibration standard. Congener Set obtained from AccuStandard or Ultra Scientific.

10.5 Stock Standard Solutions

10.5.1 Stock standards are prepared from individual neat Aroclor formulations by weighing approximately 0.1000g to the nearest 0.2 mg and dissolving and diluting to volume in a 100mL volumetric flask with hexane. This will give a stock concentration of 1,000ug/mL.

10.5.2 The stock standard is transferred into screw-cap 120mL boston bottles and stored in a freezer (≤0°C), protected from light. Stock standards should be checked at frequent intervals for signs of evaporation, especially just prior to preparing calibration standards.

10.5.3 Stock PCB standards must be replaced after one year or sooner if comparison with continuing calibration check standards indicate a problem.

10.5.4 Stock standards for the following are prepared by the above procedure:

Aroclor 1232
Aroclor 1248
Aroclor 1262

10.6 Mixed Aroclor Stock Standard at 62.7ug/mL: A primary stock standard is prepared at 62.7ug/mL that is used for preparing secondary stock standards and calibration standards. This stock standard is prepared by combining Aroclor 1232, Aroclor 1248, and Aroclor 1262 in a 25:18:18 ratio with a final mixture concentration of 25.7ug/mL, 18.6ug/mL, and 18.4ug/mL respectively (total=62.7ug/mL). These ratios are strictly maintained so that the percent composition data remains applicable, since it was developed for use under these fixed mixture parameters. The final concentration of the mixed standard may vary to accommodate instrument sensitivity or more closely represent sample concentrations, but the same ratio values must be maintained. Using a 5.0mL Class A pipette, accurately add 2.49mL of stock Aroclor 1232 standard (1,033ug/mL) to a 100mL volumetric flask. Using a 2.0mL Class A pipette, accurately add 1.82mL of stock Aroclor 1248

standard (1,019ug/mL) and 1.80mL of stock Aroclor 1262 standard (1,024ug/mL) to the same 100mL volumetric flask. Make volume to the 100mL mark with hexane.

10.6.1 Store the Mixed Aroclor Stock Standard at 62.7ug/mL in a refrigerator (4 ± 2°C) or freezer (≤0°C) in a tightly capped bottle. This standard must be replaced after one year, or sooner, if comparison with the continuing check standard indicates a problem.

10.6.2 Linearity Study Standards: The Linearity Standards are all prepared from the 62.7ug/mL mixed Aroclor stock standard. The following standard concentrations are needed. 31.35ug/mL w/OCN; 6.27ug/mL w/o OCN, 6.27ug/mL w/OCN; 1.25ug/ml w/o OCN; 1.25ug/ml w/OCN and 0.125ug/mL w/OCN.

10.6.2.1 Linearity Study Standard at 31.35ug/mL: Using a 5.0mL Class A pipette, accurately add 5.0mL of the 62.7 stock standard into a 10mL volumetric flask. Using a 10 microliter syringe add 9.0uL of 202ug/mL Octachloronaphthalene into the same 10mL volumetric flask and set to volume with hexane. The Octachloronaphthalene is used as an internal standard for instrument calibration. Transfer the standard solution to a 10ml vial and store in a refrigerator (4 ± 2°C) or freezer (≤0°C).

10.6.2.2 Linearity Study Standard at 6.27ug/mL w/o OCN: Using a 1.0mL Class A pipette, accurately add 1.0mL of the 62.7 stock standard into a 10mL volumetric flask and set to volume with hexane. Transfer the standard solution to a 10ml vial and store in a refrigerator (4 ± 2°C) or freezer (≤0°C).

10.6.2.3 Linearity Study Standard at 6.27ug/mL w/OCN: Using a 1.0mL Class A pipette, accurately add 1.0mL of the 62.7 stock standard into a 10mL volumetric flask. Using a 10 microliter syringe add 9uL of 202ug/mL Octachloronaphthalene into the same 10mL volumetric flask and set to volume with hexane. The Octachloronaphthalene is used as an internal standard for instrument calibration. Transfer the standard solution to a 10ml vial and store in a refrigerator (4 ± 2°C) or freezer (≤0°C).

10.6.2.4 Linearity Study Standard at 1.25ug/mL w/OCN: Using a 5.0mL Class A pipette, accurately add 2.0mL of the 6.27ug/mL standard w/o OCN (10.6.2.2) into a 10mL volumetric flask. Using a 10 microliter syringe add 9uL of 202ug/mL Octachloronaphthalene into the same 10mL volumetric flask and set to volume with hexane. The Octachloronaphthalene is used as an internal standard for instrument calibration. Transfer the standard solution to a 10ml vial and store in a refrigerator (4 ± 2°C) or freezer (≤0°C).

10.6.2.5 Linearity Study Standard at 1.25ug/mL w/o OCN: Using a 5.0mL Class A pipette, accurately add 2.0mL of the 6.27ug/mL standard w/o OCN (10.6.2.2) into a 10mL volumetric flask and set to volume with hexane. Transfer the standard solution to a 10ml vial and store in a refrigerator (4 ± 2°C) or freezer (≤0°C).

10.6.2.6 Linearity Study Standard at 0.125ug/mL w/ OCN: Using a 1.0mL Class A pipette, accurately add 1.0mL of the 1.27ug/mL standard w/o OCN (10.6.2.5) into a 10mL volumetric flask. Using a 10 microliter syringe add 9uL of 202ug/mL Octachloronaphthalene into the same 10mL volumetric flask and set to volume with hexane. The Octachloronaphthalene is used as an internal standard for instrument calibration. Transfer the standard solution to a 10ml vial and store in a refrigerator (4 ± 2°C) or freezer (≤0°C).

10.6.3 All Linearity Study standards must be replaced after 6 month

10.7 Mixed Aroclor Calibration Standard at 6.27ug/mL: The calibration standard is prepared from the 62.7ug/mL mixed Aroclor stock standard (10.6). Using a 10.0mL Class A pipette, accurately add 10.0mL of the 62.7 stock standard into a 100mL volumetric flask. Using a 100 microliter syringe add 90.0uL of 202ug/mL Octachloronaphthalene into the same 100mL volumetric flask and set to volume with hexane. The Octachloronaphthalene is used as an internal standard for instrument calibration. Transfer the standard solution to a 120-ml boston bottle and store in a freezer (≤0°C).

10.7.1 The 6.27ug/mL Mixed Aroclor Calibration Standard must be replaced after 6 month, or sooner, if comparison with continuing check standards indicates a problem.

10.8 Supplemental Congener Standard: A Supplemental Congener Standard is analyzed along with the 6.27ug/mL Mixed Aroclor Calibration Standard. This standard contains congeners that exist at low levels in the mixed Aroclor standard and

comprises congeners that are not typically found in Aroclor formulations, but could become important in by-product PCB analysis or the study of model experiments that use unusual PCB congeners. This standard is analyzed to supply accurate retention time information and response factors for quantification.

10.8.1 Supplemental Congener Stock Standard at 2.00ug/mL: All stock standards are purchased as solutions at 100ug/mL from Ultra Scientific or equivalent. All supplemental congeners are diluted together (except 3-Chlorobiphenyl) to 2.00ug/mL. For each congener, using a 2000uL syringe, transfer 2.0mL of the 100ug/mL stock standard into the same 100mL volumetric flask and set to volume with hexane. The 3-Chlorobiphenyl, due to its low ECD response, will be added to the secondary stock standard. Transfer the standard solution to a 120mL boston bottle and store in a refrigerator (4 ± 2°C) or freezer (≤0°C). This stock standard must be replaced after one year.

10.8.1.1 This standard can also be purchased as a custom mix with a concentration of 100ug/mL per congener. The preparation remains the same as in 10.8.1.

10.8.1.2 Supplemental Congener Secondary Stock Standard at 0.0500ug/mL: Into a 50mL volumetric flask combine, using a 2000uL syringe, 1.25mL of the Supplemental Congener Stock Standard, using a 1000uL syringe, 1.0mL of the 3-Chlorobiphenyl purchased stock standard at 100ug/mL and using a 50uL syringe, 45uL of the Octachloronaphthalene Stock Standard at 202ug/mL. Set to volume with hexane and transfer the standard solution to a 120mL boston bottle and store in a refrigerator (4 ± 2°C) or freezer (≤0°C). The Octachloronaphthalene is used as an internal standard for instrument calibration and is at a concentration of 0.1818ug/mL in the standard. The standard concentration is 2.00ug/mL for 3-Chlorobiphneyl and 0.050ug/mL for all other congeners in the standard.

Supplemental Congener Standard

DB-1 Peak Number	**IUPAC Congener Number**	**(IUPAC #) PCB Congener Analyzed**	**Conc ug/mL**
3	2	(2) 3-Chlorobiphenyl	2.000
9	14	(14) 3,5-Dichlorobiphenyl	0.050
11	30	(30) 2,4,6-Trichlorobiphenyl	0.050
12	11	(11) 3,3'-Dichlorobiphenyl	0.050
19	23,34,54	(34) 2',3,5-Trichlorobiphenyl	0.050
28	36	(36) 3,3',5-Trichlorobiphenyl	0.050
30	39	(39) 3,4',5-Trichlorobiphenyl	0.050
35	62,65	(65) 2,3,5,6-Tetrachlorobiphenyl	0.050
36	35	(35) 3,3',4-Trichlorobiphenyl	0.050
41	68,96	(96) 2,2',3,6,6'-Pentachlorobiphenyl	0.050
43	57,103	(103) 2,2',4,5',6-Pentachlorobiphenyl	0.050
62	154	(154) 2,2'4,4',5,6'-Hexachlorobiphenyl	0.050
68	123	(123) 2',3,4,4',5-Pentachlorobiphenyl	0.050
70	140	(140) 2,2',3,4,4',6'-Hexachlorobiphenyl	0.050
76	127,168,184	(127) 3,3',4,5,5'-Pentachlorobiphenyl	0.050

10.9 Internal Standard Stock Standard at 202ug/mL: The internal standard used for capillary gas chromatography of PCBs will be Octachloronaphthalene (OCN). Weigh, 10.1mg of solid Octachloronaphthalene (OCN) into a 5mL vial. Quantitatively transfer the OCN using six successive 2-mL washings of toluene to a 50mL volumetric flask. Be sure to rinse the 5mL vial walls carefully so that all OCN is completely transferred to the 50mL volumetric flask. Make the solution to volume

using toluene and mix the internal standard solution by shaking the flask several times. This will give a concentration of OCN of 202ug/mL. Carefully transfer the internal standard solution to 25mL vials, tightly cap, and store in a refrigerator (4 ± 2°C) or freezer (≤0°C). A portion of the internal standard is transferred to a 5mL reacti-vial with a Teflon syringe-valve cap to use on a daily basis. The react-vial minimizes evaporation since the cap does not have to be removed.

10.9.1 The internal standard is added to all calibration standards, continuing check standards, blanks, samples, and QC samples at the same amount. In most cases this will be achieved by spiking 9.0uL of OCN internal standard solution to 10mL of standard or sample extract to give a solution concentration of 0.1818ug/mL.

10.9.2 The internal standard will be added to calibration standards, sample extracts, blanks, and QC samples prior to gas chromatographic analysis. Thus, the internal standard is used as a quantification spiking standard and will eliminate sample injection volume variations, but will not correct for analytical losses during sample preparation.

10.9.3 OCN internal stock standard must be replaced after one year. Working standards are replaced every 6 month.

10.10 Surrogate Stock Standard (2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl) at 100ug/mL:The surrogate stock standard is prepared from a solid standard. Weigh 5.0mg of the neat Standard material into a solvent rinsed 10mL vial. Quantitatively transfer the surrogate standard using six successive 2-mL washings of hexane to a 50mL volumetric flask. Be sure to rinse the 10mL vial walls carefully so that the entire surrogate standard is completely transferred to the 50mL volumetric flask. Make to volume with hexane and mix the surrogate standard solution by shaking the flask several times. This will give a concentration of surrogate standard of 100ug/mL. Carefully transfer the surrogate standard solution to a 125mL boston bottle, tightly cap, and store in a freezer (≤0°C) or refrigerator (4 ± 2°C). The surrogate standard must be replaced after one year.

10.10.1 Surrogate Calibration Standard (2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl) at 50.0ng/mL with Internal Standard at 181.8ng/mL: The Surrogate Calibration Standard is prepared from the 100ug/mL Surrogate Stock Standard and the 202ug/mL Internal Standard Stock Standard. Into a 100mL volumetric flask, using a 50-microliter syringe, transfer 50uL of the Surrogate Stock Standard at 100ug/mL. Into the same 100mL volumetric flask transfer, using a 100-microliter syringe, 90 microliters of the 202ug/mL Internal Standard Stock Standard. Make to volume with hexane and transfer the standard solution to a 125mL boston bottle, tightly cap, and store in a freezer (≤0°C) or refrigerator (4 ± 2°C). This will give a concentration of Surrogate Calibration Standard of 50ng/mL and Internal Standard (OCN) of 181.8ng/mL. This Standard must be replaced after six months.

10.10.2 Surrogate Calibration Standard (2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl) at 20.0ng/mL with Internal Standard at 181.8ng/mL: The Surrogate Calibration Standard is prepared from the 100ug/mL Surrogate Stock Standard and the 202ug/mL Internal Standard Stock Standard. Into a 100mL volumetric flask, using a 50-microliter syringe, transfer 20-miroliter of the Surrogate Stock Standard at 100ug/mL. Into the same 100mL volumetric flask transfer, using a 100-microliter syringe, add 90 microliters of the 202ug/mL Internal Standard Stock Standard. Make to volume with hexane and transfer the standard solution to a 125mL boston bottle, tightly cap, and store in a refrigerator (4 ± 2°C). This will give a concentration of Surrogate Calibration Standard of 20ng/mL and Internal Standard (OCN) of 181.8ng/mL. This Standard must be replaced after six months.

10.11 Continuing Calibration Check Standards (CCCS): Continuing calibration check standards at 1.27ug/mL and 0.127ug/mL are prepared from Aroclor solutions obtained from a different source (ULTRA Scientific or equivalent) than the calibration standard. The continuing calibration check standard is a mixed Aroclor 1232, 1248, and 1262 in the fixed ratio used to prepare the calibration standard and must be strictly adhered to.

10.11.1 50.0ug/mL Continuing Calibration Check Stock Standards: Aroclor 1232, Aroclor 1248, and Aroclor 1262 are obtained from ULTRA Scientific or equivalent at 1000ug/mL in isooctane. Using a 500-microliter syringe, 500-microliter of 1000ug/mL Aroclor 1232 is transferred to a 10.0mL volumetric flask and made to volume with hexane. This procedure is repeated for Aroclor 1248 and Aroclor 1262. The 50ug/mL stock standards are each transferred to an individual 10mL vial, tightly capped and stored in a freezer (≤0°C) or refrigerator (4 ± 2°C). These stock standards must be replaced after one year.

10.11.2 1.27ug/mL Continuing Calibration Check Standard with OCN: Using a 1.0mL syringe transfer 1.0mL of 50.0ug/mL Aroclor 1232, 0.720mL of 50.0ug/mL Aroclor 1248, and 0.720mL of 50.0ug/mL Aroclor 1262 into a 100mL volumetric flask. Using a 100-mircoliter syringe, add 90.0-microliter of OCN internal standard (final

concentration of 0.1818ug/mL). Set to volume with hexane and mix well by shaking and inverting flask several times. The prepared continuing calibration check solution will contain a total of 1.27ug/mL PCB (0.500ug/mL Aroclor 1232, 0.360ug/mL Aroclor 1248, and 0.360ug/mL Aroclor 1262).

10.11.3 Transfer the 1.27ug/mL Continuing Calibration Check Standard with OCN to a 120mL boston bottle, cap tightly, and store in a freezer (≤0°C) or refrigerator (4 ± 2°C). A new continuing check standard must be prepared every six months.

10.11.4 1.27ug/mL Continuing Calibration Check Standard without OCN: Using a 1.0mL syringe transfer 1.0mL of 50.0ug/mL Aroclor 1232, 0.720mL of 50.0ug/mL Aroclor 1248, and 0.720mL of 50.0ug/mL Aroclor 1262 into a 100mL volumetric flask. Make to volume with hexane and mix well by shaking and inverting flask several times. The prepared continuing check solution will contain a total of 1.27ug/mL PCB (0.500ug/mL Aroclor 1232, 0.360ug/mL Aroclor 1248, and 0.360ug/mL Aroclor 1262).

10.11.5 Transfer the 1.27ug/mL Continuing Calibration Check Standard without OCN to a 120mL boston bottle, cap tightly, and store in a freezer (≤0°C). A new continuing calibration check standard must be prepared every six months. This continuing check standard without OCN is used to prepare the 0.127ug/mL Continuing Calibration Check Standard with OCN.

10.11.6 0.127ug/mL Continuing Calibration Check Standard with OCN: Using a 10.0mL Class A pipette transfer 10.0mL of 1.27ug/mL Continuing Calibration Check Standard without OCN to a 100mL volumetric flask. Using a 100-microliter syringe, add 90.0uL of OCN internal standard (final concentration of 0.1818ug/mL). Make to volume with hexane and mix well by shaking and inverting flask several times. The prepared continuing calibration check solution will contain a total of 0.127ug/mL PCB (0.050ug/mL Aroclor 1232, 0.036ug/mL Aroclor 1248, and 0.036ug/mL Aroclor 1262).

10.11.7 Transfer the 0.127ug/mL Continuing Calibration Check Standard with OCN to a 120mL boston bottle, cap tightly, and store in a freezer (≤0°C) or refrigerator (4 ± 2°C). A new Continuing Calibration Check Standard must be prepared every six months.

11.0 Sample Collection, Preservation, Shipment and Storage

11.1 Sample Collection and Preservation:

11.1.1 Routine soil, sediment, sludge, solid and concentrated liquid samples should be collected in 8 oz clear glass wide-mouth jars, fitted with a Teflon-lined cap. Aqueous samples should be collected in 1 liter amber glass bottles with a Teflon-lined cap. Project specific, the jars maybe required to be pre-cleaned to EPA specification protocol A – recommended for extractable organic, semivolatile and pesticide analysis. Protect samples from light.

11.1.2 All samples must be placed on ice or refrigerated at 4°C (± 2°C) from the time they are collected until delivery to the lab. Samples collected and delivered to the laboratory on the same day may not reach 4± 2°C. Sample cooling is considered adequate if samples are received on ice.

11.2 Sample Shipment:

11.2.1 Sample Shipment is accomplished through a carrier such as Federal Express or United Postal Service for overnight 1-day delivery to the lab. Shipment is normally handled by the field personnel collecting the samples and coordinated with sample receiving department at the lab. Samples can also be picked up by the lab courier service if samples are collected within driving distance to the lab.

11.3 Sample Storage:

11.3.1 The samples must be protected from light and refrigerated at 4°C (± 2°C) from time of receipt until they are removed from storage for extraction. Remaining sample material will be stored protected from light and refrigerated at 4°C (± 2°C). Sample will be disposed of or stored / archived according to project specifications.

11.3.2 Routine soil, sediment, sludge, solid, liquid and concentrated liquid samples are stored in a refrigerator (4 ± 2°C) dedicated for this type of sample.

11.4 Sample Extract Storage:

11.4.1 Sample extracts must be protected from light and stored refrigerated at 4°C (± 2°C) during the analysis. After analysis is complete, sample extracts will be discarded after 60 days or can be archived in a freezer (≤0°C) and at less than -20°C for longer periods of time depending on the project requirements.

11.4.2 Field samples, sample extracts, and calibration standards must be stored separately.

11.5 Required Hold Time:

11.5.1 Extraction of solid samples by appropriate technique must be completed within fourteen days from sample collection.

11.5.2 Extraction of aqueous samples by appropriate technique must be completed within seven days from sample collection.

11.5.3 Sample extracts must be analyzed within forty days of sample extraction.

12.0 Quality Control

12.1 This section outlines the necessary quality control samples that need to be generated at the time of sample extraction. The results of the quality control measurement samples document the quality of the data generated. The following table lists the Quality Control samples required for capillary gas chromatography analysis of PCBs.

Quality Control Requirements

QC Sample	Frequency
Method Blank	With each sample batch (up to 20 samples)
Lab Control Spike	With each sample batch (up to 20 samples)
Cont Cal Check Std	Analyzed prior to each sample batch and at a Frequency or one per ten injections. Each analytical sequence must close with a Continuing Calibration Check Standard (CCCS).
Duplicate Analysis	Field generated sample – analyzed at discretion of client.
Matrix Spike	One matrix spike per 20 field samples or designated sample batch may be performed as specified in the client site plan.
Matrix Spike Duplicate	One matrix spike duplicate per 20 field samples or designated sample batch may be performed as specified in the client site plan.

12.2 Method Blank:

12.2.1 With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples and organic-free reagent water blank is processed. For soil, sediment, solid, fish, and other tissue sample a laboratory clean sodium sulfate is processed. For oil samples, a PCB-free blank control oil is processed. The

method blank must exhibit PCB levels less than the matrix defined reporting limit (RL). If the method blank exhibits PCB contamination above the reporting limit, the samples associated with the contaminated blank should be re-extracted and analysis repeated. If there is no original sample available for re-extraction, then the results should be flagged with a “B” indicating blank contamination. The value measured in the blank is reported for those samples associated with the particular blank out of criteria.

12.3 Laboratory Control Spike:

12.3.1 A Laboratory Control Spike (LCS), also referred to as a QC reference check standard, is extracted with each batch of samples at a rate of one per 20 samples. For water sample, spike one liter of laboratory organic free water, extract and analyze. For solid and tissue samples spike 10 grams of sodium sulfate/ hydromatrix, extract and analyze. For oil samples spike 0.50 gram of PCB free oil, extract and analyze. An Aroclor is chosen for the LCS analyte, typically based on program requirements or expected sample contamination. Calculate the percent recovery for the PCB spike. If the percent recovery for the LCS is out of criteria, (60%-140%) the analysis is out of the control for that analyte and the problem should be immediately corrected. If there is sufficient sample, the samples associated with the Laboratory Control Spike that failed must be re-extracted and re-analyzed. If no more sample material is available, the data must be flagged to indicate low or high Control Spike recovery.

12.4 Duplicate Analysis:

12.4.1 Duplicate analysis of the same sample is performed to assess method precision. A duplicate can also be performed as a blind duplicate, so that identification with original sample is withheld. The analysis of a duplicate sample precludes that PCBs are to be found at appreciable levels in samples. If this is not known the analysis of matrix spike/matrix spike duplicates provide more consistent quality control information. The relative percent difference of the two measurements on the sample is calculated on total PCB concentration by the following equation:

RPD = (DUP1-DUP2)/AVG x 100

Where: RPD = Relative Percent Difference
DUP1 = The greater of the measured values
DUP2 = The lesser of the measured values
AVG = Average of the two analysis

The relative percent difference must be less than or equal to 30%.

12.5 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

12.5.1 Spiked sample matrix data are analyzed to assess analytical accuracy and recovery of analytes of interest. Thus the sample is spiked and carried through sample analytical procedures including extraction, clean up, and GC analysis. Depending on the specific project plans and at the discretion of the client a matrix spike or matrix spike and matrix spike duplicate can be analyzed.

12.5.2 There must be sufficient sample for analysis of matrix spike/matrix spike duplicate samples and the sample must be homogeneous in PCB distribution for valid data to be produced. Spike MS and/or MSD samples with the Aroclor matrix spike standard at a concentration approximately two to five times the sample concentration. Extract and analyze the two spiked samples following procedures used for actual sample analysis. Calculate the percent recovery of the matrix spike/matrix spike duplicate by the following equation:

P = A-B/T x 100

Where: P = Percent recovery, %
A = concentration of analyte in the spike sample aliquot
T = Know true values of the spike concentration
B = Background concentration of PCB in the unspiked sample aliquot

12.5.3 Matrix spike recovery information is used to assess the long-term precision and accuracy of the method for each encountered matrix. Matrix spike/matrix spike duplicate results are not used alone to qualify an extraction batch. Generally, percent recovery for MS/MSD samples should be greater than or equal to 60% and less than or equal to 140% based on the total PCB concentration. If the percent recovery is outside the limits, all calculations should be checked and the data should be narrated to describe possible matrix interference.

12.6 Surrogate:

12.6.1 Surrogate-spiking compounds monitor the extraction efficiency and sample processing procedures for each sample. Surrogate compounds are chosen which do not chromatographically interfere with the PCB target congeners and which behave similarly to the target PCB congeners during extraction and sample processing.

12.6.2 A surrogate compound is added to each sample, matrix spike, matrix spike duplicate, duplicate, method blank, and laboratory control spike at the time of extraction. The surrogate compound chosen for this method is 2,2',3,3',4,4',5,6,6' – Nonachlorobiphenyl, Accu Standard Cat. No. C-206S-TP (this congener is not present or found at trace amounts in Aroclor formulations). The following are typical surrogate amounts added to normally encountered matrices. These amounts can be adjusted by the analyst, if PCB background levels are high and surrogates are being diluted out of analysis range.

12.6.2.1 Water: 0.50ml of 0.20ppm 2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl

12.6.2.2 Soil/Sediment/Solid/Fish: 1.00ml of 1.25ppm 2,2',3,3',4,4',5,6,6'-Nonachlorobipenyl

12.6.3 Surrogate recovery measurements are used for advisory purposes, if surrogate recovery is not within laboratory establish limits, the following steps are required. The laboratory established limits for surrogate recovery are 60%-140%.

12.6.3.1 Review calculations that were used to generated surrogate percent recovery values to make certain there are no errors.

12.6.3.2 Check by GC analysis surrogate solutions used during sample extraction steps to ensure that no problems exist with spiking solutions.

12.6.3.3 Review data for chromatographic interferences.

12.6.3.4 Re-extraction and/or re-analysis of samples may be indicated if problems persist with surrogate recoveries. If the surrogate percent recovery is out of limits on the re-extracted samples, low or high surrogate recovery is due to matrix affects and the data can be reported as estimated.

12.7 Continuing Calibration Check Standard (CCCS):

12.7.1 As outlined in Section 12.9.1, a CCCS will be analyzed on each working day prior to sample analysis and at an interval of one CCCS per 10 samples. The CCCS must meet the acceptance criteria established in Section 13.3. If the CCCS Standard fails to meet the acceptance criteria, the following guidance must be followed.

12.7.2 If samples are being run using an autoanalyzer (i.e., the instrument is unattended) and a CCCS that fails to meet the acceptance criteria is present in the analytical sequence but acceptable CCCSs are observed later in the analytical sequence, samples bracketed by acceptable CCCSs will be reported.

12.7.3 If the reason for the failure of the CCCS appears to be a poor injection (or a degraded standard solution), the CCCS will be re-injected (or re-prepared and re-injected) immediately following the failed CCCS. This can only occur if the instrument is being attended by an analyst. If upon re-injection, the CCCS meets all the acceptance criteria established in Section 13.3. and there is no

apparent impact on the sample data (i.e., acceptable internal standard areas and surrogate recoveries are observed), the analytical sequence will continue and samples will not be reanalyzed. The associated sample data will be reported.

12.7.4 If the CCCS fails to meet the acceptance criteria, the initial calibration standards must be re-analyzed and new response factors generated. After re-calibration, the CCCS must be analyzed again and compared to the acceptance criteria. If the CCCS fails to meet the acceptance criteria after re-calibration, sample analysis must not proceed until the problem is corrected.

12.7.5 All samples that were analyzed directly before or after the CCCS exceeded established criteria must be re-analyzed.

12.8 Retention Time Windows:

12.8.1 Refer to the table entitled "Quality Control Acceptance Criteria and Corrective Action Plan" in Section 19.0 for retention time window and retention time shift acceptance criteria and corrective action.

12.8.2 The Initial CCCS of each analytical sequence is used to establish the retention time window for each analyte. The retention time window equals the absolute retention time of the Initial CCCS for a given batch of samples ±0.07 minutes.

12.8.3 Besides using the retention time window to assign peaks for quantification, the analyst should also rely on their experience in pattern recognition of multi-response sample analysis.

12.9 Analytical Sequence Queue:

12.9.1 A typical analytical sequence is as follows:

Injections	Material Injected
1-2	Hexane Instrument Blanks
3	Initial Calibration Standard 6.27 ppm
4	Hexane Blank
5	Supplemental Congener Standard
6	Hexane Blank
7-8	Surrogate Calibration Standards
9	Hexane Blank
10	Continuing Calibration Check Standard (CCCS)
11	Method Blank
12	Lab Control Spike
13-19	Samples (including Duplicates, MS/MSD)
20	Continuing Calibration Check Standard (CCCS)
21-29	Samples (including Duplicates, MS/MSD)
30	Continuing Calibration Check Standard (CCCS); repeat 21 to 30

13.0 Calibration and Standardization

13.1 Calibration:

13.1.1 Gas chromatographic operation parameters:

13.1.1.1 GC Column: DB-1 or ZB-1 (J&W or Phenomenex, bonded polydimethylsilicone), 30 meters, 0.25 mm internal diameter, 0.25 micron phase coating.

13.1.1.2 Oven Temperature Program: 50°C for 2.5 min hold time, 50°C to 150°C at 15.0°C/min, hold 0.01minutes, 150°C to 220°C at 4.3°C/minute, hold at 220°C for 35.1 minutes.

13.1.1.3 GC Column Velocity: Approximately 30 cm/sec Helium. Column flow adjusted to elute OCN Internal Standard between 42.0 and 48.0 minutes.

13.1.1.4 Detector: Electron Capture Detector (micro-ECD), attenuation 0, range 3. (adjustable based on signal sensitivity).

13.1.1.5 Detector Temperature: 300°C.

13.1.1.6 Injector Temperature Program: 250°C , pulsed split injection, initial pressure at 25.0PSI, pulsed pressure at 40PSI, pulsed time 0.75 minutes, split ratio 25 (adjustable based on signal sensitivity).

13.1.1.7 Detector Make-up Gas: Approximately 65mL/min Nitrogen. Adjusted for signal sensitivity.

13.1.1.8 Autosampler: 1.0uL sample volume (adjustable based on signal sensitivity). Sample pumps 4, viscosity delay 3, sample wash 2, solvent A 2 washes, solvent B 2 washes, slow plunger OFF, sampling offset OFF, solvent A pre-wash 1, solvent B pre-wash 1.

13.2 Initial GC Calibration:

13.2.1 Prior to running samples the system must be calibrated and the Initial Continuing Calibration Check Standard must be verified.

13.2.2 Establish the gas chromatographic operation parameters outlined in Section 13.1.1 and prepare the appropriate calibration standards composed of a mixture of Aroclor 1232, 1248, and 1262 as outlined in Sections 10.6 through 10.11.

13.2.3 Chromatographic Resolution Criteria: Chromatographic resolution is measured by peak height to valley height for two pairs of closely eluting peaks. The peak valley height formed between DB-1/ZB-1 peaks 14 and 15 must be equal to or less than the half height of peak 15. The peak valley height formed between DB-1/ZB-1 peaks 74 and 75 must be equal to or less than one-third the height of peak 74. This peak resolution must be established initially and maintained throughout sample analysis.

13.2.4 Initial High Level Linearity Verification: The gas chromatograph must undergo a linearity study. A high-level three-point initial calibration check for linearity is analyzed and calculated relative response factors must meet a limit for relative standard deviation for each GC peak of less than 20%. This high level linearity verification is associated with samples with medium to high levels of PCBs for sample matrices such as biota, sediment, soil, oil, and other solid or liquid samples that contain appreciable levels of PCBs. The high level initial linearity is comprised of the following standards: 31.35ug/mL w/OCN Standard (10.6.2.1) the 6.27ug/mL w/OCN Standard (10.6.2.3) and the 1.27ug/mL w/OCN Standard (10.6.2.4). See Appendix C.

13.2.5 Initial Low Level Linearity Verification: The gas chromatograph must undergo a linearity study. A low-level three-point initial calibration check for linearity is analyzed and calculated relative response factors must meet a limit for relative standard deviation for each GC peak of less than 20%. This low level linearity verification is associated with samples with low levels of PCBs for sample matrices such as water and other solid or liquid samples that contain low levels of PCBs. The low-level initial linearity is comprised of the following standards: 6.27ug/mL w/OCN Standard (10.6.2.3), the 1.27ug/mL w/OCN Standard (10.6.2.4), and the 0.127ug/mL w/OCN Standard (10.6.2.6). See Appendix C.

13.2.6 Initial 72-Hour Retention Time Window Measurement: An initial retention time study must be performed to establish retention time windows to assist in PCB peak assignment. Three high level Continuing Calibration Check standards (prepared at a concentration of 1.27 ug/ml as described in Section 10.11, must be analyzed over at least a 72-hour period. In addition, the Supplemental Congener

Standard is analyzed three times over at least a 72-hour period. The mean retention time is calculated from these standards along with the standard deviation for each GC peak. The retention time window is established as ±3 times the determined standard deviation. If the 3 times the standard deviation value is less than 0.07 minutes a default of 0.07 minutes is applied to that GC peak. The default 0.07 minutes retention time window is the default window used in Contract Laboratory Program, Statement of Work for Organic Analysis for PCB/Pesticide analysis. See Appendix C.

13.2.7 Our laboratory uses a computer based chromatography software module (Water Corporation, Empower software) interfaced to the gas chromatograph and a In House LIMS System for data handling, reporting and packaging. The Empower workstation acquires and processes the analog detector signal, which is then digitally converted. Empower then stores the digitized chromatograms in a database on a designated Server. All phases of integration of peak areas, quantitation and production of chromatograms and raw data reports is performed within the Empower software. This data then can be captured by the LIMS system for further data handling, scoring, reporting, packaging and data distribution as requested by the client.

13.2.8 After the above criteria are met system calibration for sample analysis can be performed. Appendix A identifies which congener and or congeners compose each resolvable GC peak in the calibration standard, along with the amount that each congener or co-eluting group of congeners are represented in the calibration standard. Throughout this document the IUPAC PCB numbering system will be used for congener identification. Appendix B is an example of an acceptable chromatogram of the calibration standard, along with peak congener labels for cross-reference to data in Appendix A. Analyze the 6.27ug/mL Calibration Standard to initiate calibration of the GC system. Also analyze the Supplemental Congener Standard to calculate relative response factors for congeners that do not exist in the 6.27ug/mL Calibration Standard. Response factors are calculated relative to the internal standard by the following equation:

$$RRF = (Ax/Ais)x(Cis/Cx)$$

Where:	RRF	=	Relative response factor of congener(s).
	Ax	=	Area of peak for the congener(s).
	Ais	=	Area of peak for the internal standard.
	Cx	=	Concentration of the congener(s).
	Cis	=	Concentration of the internal standard.

13.3 Continuing Calibration:

13.3.1 Chromatographic Resolution: Chromatographic resolution is measured by peak height to valley height for two pairs of closely eluting peaks. The peak valley height formed between DB-1/ZB-1 peaks 14 and 15 must be equal to or less than the half height of peak 15. The peak valley height formed between DB-1/ZB-1 peaks 74 and 75 must be equal to or less than one-third the height of peak 74. This peak resolution must be established initially and maintained throughout sample analysis.

13.4 Response Factor Verification:

13.4.1 The relative response factors calculated from the calibration standard will be verified on each working day by analyzing a CCCS, calculating the selected congener concentrations and comparing to their known concentration. The CCCS concentration is either 1.27ug/mL or 0.127ug/mL depending on the expected concentrations of PCB in the sample. A subset of six congeners and Total PCBs will be used to verify the relative response factors before samples are processed. The Percent Difference for Total PCBs must be <15%. The six congeners include:

DB-1 PEAK NUMBER	IUPAC CONGENER NUMBER	RELATIVE PEAK LEVEL IN CALIBRATION STANDARD	PEAK CONC 1.27ug/mL or 0.127ug/mL CONTINUING CALIBRATION STD (ng/mL)	PERCENT DIFFERENCE LIMITS
7	6	Low level peak in	14.10 / 1.41	<30
116	205	Low level peak in	0.820 / 0.082	<30
47	70	Medium level peak in	25.22 / 2.52	<15
93	174, 181	Medium level peak in	23.74 / 2.37	<15
37	104, 44	high level peak in	31.90 / 3.19	<15
102	180	high level peak in	45.26 / 4.53	<15

13.4.2 After the Continuing Calibration Check Standard is analyzed, calculate the amount for these six congeners and Total PCBs and compare those values to the known concentrations by the following equation:

Percent Difference = [Amt(1)-Amt(2)]/Amt(2) x 100

Where: Amt(1) = Amount calculated for congener or Total PCBs.
Amt(2) = Known amount for congener or Total PCBs.

13.4.3 A percent difference greater than ± 30% for the two low-level peaks (7 and 116) indicate an instrument problem or unacceptable relative response factors. A percent difference greater than ± 15% for the medium level (47 and 93) and high level (37 and 102) peaks also indicates an instrument problem or unacceptable relative response factors. If any of the evaluation congeners or Total PCBs fails to meet the percent difference acceptance criteria, the guidance provided in Section 18.3. must be followed.

13.4.4 The percent recovery for the internal standard Octachloronaphthalene (OCN) in the CCCS must be within 50-150% of the OCN area in the associated calibration standard. If the OCN area fails to meet the acceptance criteria, the guidance provided in Section 18.3. must be followed.

13.4.5 If re-calibration is performed, the CCCS must be analyzed again and values calculated using the new relative response factors. If the CCCS fails to meet the percent difference criteria after re-calibration, sample analysis must not proceed until the problem is found and corrected (i.e., GC gas leak, autosampler lines plugged, broken injector liner).

14.0 Procedure

14.1 Sample Extraction and Preparation:

14.1.1 The following SOPs detail sample extraction procedures that are utilized in preparing samples for analysis by this analytical method:

SOP NAME	TITLE
NE005	SOXHLET EXTRACTION : SOLIDS
NE006	WATER EXTRACTION
NE017	FISH & BIOTA EXTRACTION
NE088	WIPE EXTRACTION FOR PCB
NE111	WASTE DILUTION FOR PCB
NE124	CLLE PCB WATER EXTRACTION
NE132	FISH/BIOTA GRINDING PROCEDURES
NE140	PCB SCREENING BY GC
NE143	ASE EXTRACTION FOR PCB: SOLIDS
NE144	ASE EXTRACTION FOR WIPE: PCB
NE158	% LIPID DETERMINATION: FISH & BIOTA

14.2 Gas Chromatographic Procedures:

14.2.1 Pre-secreening of sample extracts: See standard operating procedure NE140 for details on the PCB screening procedures used prior to final analysis by this method. The GC will be standardized by using Aroclor 1221, Aroclor 1242 and Aroclor 1260. These three Aroclor formulations incorporate most environmental PCBs found in sample extracts and provide a good estimate of PCB amount for final dilution for capillary analysis. A three level calibration curve is utilized (0.50ug/mL, 2.5ug/mL and 5.0 ug/mL standards). The concentration of each Aroclor (grouped as Aroclor 1221, Aroclor 1242 and Aroclor 1260 only) in a sample will be calculated based on the extract volume (not the sample weight or volume) to supply solution concentration values that show if the extract needs to be diluted for final capillary GC analysis. If a dilution is necessary, sample extracts are diluted to a solution concentration between 0.60ug/ml to 1.0 ug/mL depending of the Aroclor pattern from the pre-screening results. Preferable solution set volumes are 5mL and 10mL as the internal standard (OCN) is added at this time to give a solution concentration of 0.1818ug/mL in the final dilution.

Dilution Scheme Examples

Dilution Factor based on 25x final extract volume	Amount of original Sample Transferred (mL)	Amount of Hexane added (ml)	Final Volume of diluted Sample (ml)	Amount of 202 ppm OCN Added for final analysis
e31.3 x	4	1	5	4.5 uL
41.7 x	3	2	5	4.5 uL
50.0 x	2.5	2.5	5	4.5 uL
62.5 x	2	3	5	4.5 uL
83.3 x	1.5	3.5	5	4.5 uL
125 x	1.0	4.0	5	4.5 uL
167 x	1.5	8.5	10	9.0 uL
250 x	1	9	10	9.0 uL
500 x	0.5	9.5	10	9.0 uL

Should higher dilutions be needed, a dilution sequence is performed, utilizing the dilution examples above without the addition of OCN. The OCN is only added to the final dilution used for analysis.

14.2.2 Approximately 1.0mL of the final dilution extract is then transferred into a labeled autosampler vial.

14.2.3 The sequence of the analytical queue is set up in the NEA LIMS as a unique batch file. This file contains all the sample extraction information such as sample name, client ID, sample volume extracted, set volume and the exact order in which standards, instrument blanks, and samples will be analyzed. Once the sample set is uploaded into the Empower acquisition/run screen and saved, the sample set is printed and the samples are loaded into the GC autosampler tray in the order specified by the sample set queue.

14.2.4 The following labeling will be used on the autosampler vial and for the sample set file created for the analytical queue.

14.2.4.1 The initial Calibration Standard will be labeled as ICAL0301. Substitute the actual date of analysis in the file name.

14.2.4.2 The Supplemental Congener Calibration Standard will be labeled as SC0301. Substitute the actual date of analysis in the file name.

14.2.4.3 The Surrogate Standard will be labeled SS0301. Substitute the actual date of analysis in the file name.

14.2.4.4 The Instrument blanks will be labeled 070301B01, B02, B03, etc. Substitute the actual date of analysis in the file name.

14.2.4.5 The Continuing Calibration Check Standards will be labeled CCCS0301A, CCCS0301B, etc. Substitute the actual date of analysis in the file name.

14.2.4.6 Samples are labeled with the laboratory identification number on the autosampler vial. In the sample set file the laboratory identification number along with the client identification, sample weight, set volume and dilution are entered.

14.2.5 At this point the chromatography software can be initiated to start data collection. The gas chromatograph is placed into run mode and sample analysis is performed until the analytical queue is complete.

14.3 Peak Integration and Analyte Identification:

14.3.1 Due to the complex nature of the PCB patterns encountered and the range of peak intensities that can occur in the sample chromatograms, manual peak integration is performed to accurately integrate the samples. Manual peak integration is also performed on standards to best address the changing signal intensities. Manual integration provides for better peak start and peak end positioning, better control of peak baselines and more accurate data.

14.3.2 Analytes are identified by matching retention time to the calibrated peak in the initial calibration standard that are within the retention time window of ±0.07 minutes. The analyst must also use judgment in pattern recognition.

14.3.3 The PCB congener composition of DB-1/ ZB-1 peaks is identified in Appendix A. Peaks may include one or more co-eluting PCB congeners.

14.3.3.1 In the case of some peaks, the congeners assigned to the peak consist of co-eluting congeners and a congener that is resolved or is just slightly out of the normal retention time window of ± 0.07 minutes. With exception of congeners IUPAC 77 and 122, the resolved peaks are found at trace levels in Aroclor (and, therefore, not present in the calibration standards) and are addressed in this method in the event they are detected. If these congeners are detected in a sample, the retention time window of the assigned peak is set to ensure the congener is quantitated. A standard comment is included on the Congener Weight and Mole Report (see Appendix C) identifying this issue. If detection of one of the resolved congeners occurs, a comment will be included in the report narrative indicating that the assigned DB-1/ZB-1 peak includes the presence of the resolved congener. The peaks consisting of co-eluting congeners and a congener that is resolved are as follows:

DB-1/ZB-1 Peak[1]	Resolved Congener (IUPAC #)
37 (**44**, *104*)	*104*
48 (**66**, *76*, *98*, *80*, *93*, **95**, **102**, *88*)	*80*, *88*, *93*
56 (*78*, **83**, *112*, *108*)	*108*
61 (**77**, **110**, 148)	**77**
72 (**122**, 131, 133, 142)	**122**

89 (**128**, *162*)	*162*
105 (**200**, *169*)	*169*

1 - IUPAC congener numbers listed in boldface font were found to be present in at least one of the Aroclor at or above 0.05 weight percent. These congeners should be considered the primary congeners existing in a peak composed of co-eluting congeners. IUPAC congener numbers listed in italic font were absent or present below 0.05 weight percent.

14.3.4 If uncertainty in analyte identification exists, analysis by secondary methodology such as NEA Comprehensive Quantitative Congener Specific Method or GC/MS Method 680.

15.0 Calculations

15.1 Screening GC - External Standard Calibration:

15.1.1 The GC screening analysis will be done by the external standard calibration technique. See standard operating procedure NE140 for details on the PCB screening procedures used prior to final analysis by this method. The calibration curves for each section of the PCB elution profile will be calculated using the following formula:

$$\text{Calibration factor} = \frac{\text{Amount (ug) of Aroclor}}{\text{Total area of Aroclor}}$$

15.1.2 The calibration curve will be a linear fit forced through zero.

15.2 Screening GC - Sample Calculations:

15.2.1 The concentration of each Aroclor (grouped as Aroclor 1221, Aroclor 1242 and Aroclor 1260 only) in a sample will be calculated based on the extract volume (not the same weight or volume) to supply solution concentration values that show if the extract needs to be diluted for final capillary GC analysis. The solution concentration of Aroclor 1221, Aroclor 1242, or Aroclor 1260 (or all 3) in a sample is calculated as follows.

Concentration (ug/mL) = (Ax) x (CF)
Where:
Ax = Area of Aroclor of interest in sample
CF =Calibration factor in standard

15.3 Capillary GC - Internal Standard Calibration:

15.3.1 The capillary column GC analysis will be performed by the internal standard calibration technique. Octachloronaphthalene (OCN) is added to all calibration standards, CCCS and samples at a concentration of 181.8 ng/mL.

15.3.2 Relative Response Factors for each separated and identified peak in the standard will be calculated using the following formula:

RRF = (Ax/Ais)x(Cis/Cx)
Where:
RRF = Relative response factor of congener(s).
Ax = Area of peak for the congener(s).
Ais = Area of peak for the internal standard.
Cx = Concentration of the congener(s).

Cis = Concentration of the internal standard

15.4 Capillary GC - Sample calculations:

15.4.1 The concentration of each identified PCB peak in a sample will be calculated based on the sample dry weight in the case of soils, sediment, and wet weight for fish and biota samples.

15.4.2 The sample PCB concentration of each standardized PCB peak is calculated as follows:

$$\text{Concentration (ng/g or ng/L)} = \frac{[(Ax)(Cis)(V)(D)}{[(Ais)(RRF)(Ws \text{ or } Ls)]}$$

Where:
Ax = Peak area for congener(s) being measured.
Cis = Amount of internal standard added to sample extract.
D = Dilution factor, if sample was diluted prior to analysis.
V = Extract volume.
Ais = Peak area of added internal standard
RRF = Relative response factor for congener(s) being measured.
Ws = Dry or wet weight of sample.
Ls = Volume of sample extracted

15.5 The calculated PCB concentration for each PCB peak will be compared to its respective sample-specific reporting limit (RL) and method detection limit (MDL). The results for peaks with concentrations at or above the MDL but below RL will be reported as detected and flagged as estimated with a "J" Flag. The results for peaks with concentrations at or above the RL would be reported as unqualified numeric values.

15.6 The Total PCB concentration will be calculated and reported as follows.

15.6.1 All peak results above their respective MDL (both "J" flagged and unqualified results) will be summed and compared to the sample-specific Total PCB MDL and RL.

15.6.2 If no peaks are detected above their respective MDL, the Total PCB results will be reported as not detected at or above the sample-specific Total PCB MDL.

15.6.3 If the summed peaks from Section 15.6.1 are below the Total PCB MDL the result would be reported as less than ("<") the sample-specific Total PCB MDL.

15.6.4 If the summed peaks from Section 15.6.1 are at or above the Total PCB MDL but below the Total PCB RL, the summed result will be flagged as estimated ("J").

15.6.5 If the summed peaks from Section 15.6.1 are at or above the Total PCB RL, the Total PCB result will be reported as the unqualified numeric value.

15.7 Data Output and Reporting Format:

15.7.1 Several specialized software routines have been developed for high resolution PCB analysis to aid the data user in understanding and organizing the complex data generated from this extremely detailed analysis. Appendix C contains examples of the sample hard copy format that will be used in reporting sample information. This data is also available in electronic format as a PDF file.

16.0 Method Performance

16.1 Precision and Accuracy Determination: Precision and accuracy data is obtained for the method by analyzing four laboratory control spikes at a reasonable concentration above the calibration standard and below the calibration standard 1. The analyte will be added to a laboratory organic free water sample, sodium sulfate or hydro matrix and taken through all extraction and analytical procedures. Aroclor 1242 is used as the analyte and recovery on a total basis is used to calculate percent recovery. To be valid, Aroclor 1242 will be recovered between the limits of 70 to 130 percent. Also, a percent relative standard deviation will be calculated for the replicates will be less than or equal to 20% to be valid. An initial Precision and Accuracy Determination is required for each analyst/technician combination.

16.2 Method Detection Limit: A method detection limit will be determined for this method whenever major modification to the extraction or analysis procedures are made or at a minimum frequency of every 2 years. A minimum of seven laboratory organic free water samples, sodium sulfate or hydromatrix will be prepared with the mixed Aroclor calibration standard at a low level and taken through all extraction and analytical procedures. Method detection limit data will be determined for each chromatographic peak (comprising one or more PCB congeners) based on the following equation:

MDL = S * t(n-1, 1-alpha=0.99)
Where:

S = Standard deviation of the replicate analyses
n = Number of replicates
t(n-1, 1-alpha=0.99) = Student's t value for the 99% confidence level with n-1

For example: t for 8 replicates = t(7,0.99) = 2.998

16.2.1 The determined MDL must be less than the concentration spiked but greater than one tenth (1/10) the spiked concentration. If not, repeat the MDL determination at an appropriate spike concentration for affected analytes.

17.0 Pollution Prevention

17.1 Pollution prevention is practiced in the laboratory by minimizing usage of solvents and chemicals, so that disposal of waste generated is held to the smallest amount possible. This is directly linked to the types of extraction procedures in place at the laboratory to reduce the volumes of solvents used for semi-volatile extraction procedures. Northeast Analytical employs extraction procedures such as continuous liquid/liquid and solid phase extraction methods to reduce solvent requirements for water extraction protocols and ASE and Soxhlet extractions for solid matrices.

17.2 Pollution prevention also relies on minimizing to the best extent the chemicals and solvents required to perform extraction and analysis procedures. The laboratory personnel strive to purchase chemicals and standards that will be consumed based on anticipated workload. For additional information about laboratory pollution prevention, please refer to laboratory SOP NE168.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 The GC analyst is responsible for generating the data and also is the initial individual to review the data. This would include inspection of the chromatographic data, processing the raw data, producing all required data forms, inspection of calibration curves for compliance, surrogate recovery, laboratory control spike recovery, matrix spike/matrix spike duplicate recovery, and continuing calibration compliance. See table 19.1 for Acceptance Criteria.

18.2 Once the initial review of the data is performed by the analyst, decisions are made at that time to accept the data if all criteria are met or to reject sample data if any of the quality control parameters or limits are out of control. Depending on the situation, samples requiring re-extraction will be notified to the appropriate extraction personnel, sample extracts requiring re-injection will be queued for analysis, new calibrations may have to be performed, or samples re-analyzed due to failing continuing check standards.

18.3 The analyst may also consult with the quality control officer as to the best form of action to take or if the situation warrants corrective action beyond routine practices. If no recourse is available and the data is to be reported out of criteria, a Case Narrative Report is generated and the deviation is documented and reported to the client. The Case Narrative Report is filed with the data and is also useful for production of case narratives that are issued with the final data reports. If a problem exists that requires follow-up to rectify, a Corrective Action Report (CAR) is issued to document the problem found, steps taken to resolve the problem, and what samples were affected. This CAR form is filed by the quality control officer and reviewed by management to verify that appropriate actions have been taken to correct the problem.

19.0 Corrective Action for Out-Of-Control Data

19.1 The table below outlines the data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

Quality Control Acceptance Criteria and Corrective Action Plan

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Linearity Verification and Initial Calibration	• Initially verify linearity through the High- and Low-Level Linearity Verifications. • The initial calibration checks for linearity are each at 3 concentration levels. • A single-point calibration is analyzed initially and when Continuing Calibration Check standard fails criteria.	• %RSD≤20% for among the relative response factors for each peak in the linearity verifications. • Relative response factors are to be calculated using area for each quantifiable peak with internal standard method.	• Re-analyze the initial calibration standard and/or evaluate/correct instrument malfunction to obtain initial calibration and continuing calibration check standards that meet criteria. • Sample results above highest linearity verification standard concentration require dilution and re-analysis.
eContinuing Calibration Check Standard (CCC)	• Initially analyze a CCC immediately following a calibration standard analysis. • After the initial CCC of the sequence, a CCC must be analyzed after every 9 samples. • Analytical sequence must end with analysis of a CCC.	• ≤ 30% difference based on "true" concentration for peaks 7, 116. • ≤ 15% difference based on "true" concentration for peaks 37, 47, 93, and 102 and Total PCBs • Retention time of all quantitated peaks must be within RT window (reset with each initial CCC of a sequence) • The percent recovery for the internal standard (OCN) in the Continuing Calibration Check Standard must be within 50-150% of the OCN area of the associated initial calibration standard. • All samples must be bracketed by CCCs that meet all criteria stated above	• If the reason for the failure of the CCC appears to be a poor injection (or a degraded standard solution), the CCC will be re-injected (or re-prepared and re-injected) immediately following the failed CCC. This can only occur if the instrument is being attended by an analyst. If upon re-injection, the CCC meets all the acceptance criteria and there is no apparent impact on the sample data (*i.e.,* acceptable OCN areas and surrogate recoveries are observed), the analytical sequence will continue and samples will not be reanalyzed. The associated sample data will be reported. • If CCC failure was not due to a poor injection (or degraded standard solution) or the instrument was unattended at the time of the CCC failure, correct system, if necessary, and recalibrate. Initial calibration and CCC criteria

			must be met before sample analysis may begin. Samples that are not bracketed by complaint CCCs must be re-analyzed. • If acceptable CCCs are observed later in the sequence, samples bracketed by acceptable CCCs will be reported. Samples between the failed CCC and prior/ subsequent complaint CCC will be re-analyzed.

Quality Control Acceptance Criteria and Corrective Action Plan

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Retention Time (RT) Windows	• Initial RT windows are established to assist in PCB peak assignment. Three high-level CCC standards and three Supplemental Congener Standards are analyzed over at least 72-hours. The mean RT and the standard deviation of each of the GC peaks are calculated. RT window is ± 3× the determined standard deviation or a default of 0.07 minute is applied (whichever is larger). • RT windows are re-centered based on the initial CCC analyzed after calibration curve or if using the first CCC of the day to start a new sequence.	•	•
Retention Time (RT) shift	• Each CCC analysis: RT of all quantitated peaks in the CCC is evaluated against the initial CCC following the initial calibration curve. • Each sample analysis: Rely on RT windows to identify PCB congeners to report. Also use pattern recognition and professional judgment for peaks that shift from RT windows, because congener composition may shift RT for GC peaks.	• Each quantitated peak and surrogate peak should be with established windows.	• Inspect chromatographic system for malfunction, correct problem. Perform re-analysis if necessary.
Method Blank	• One per extraction batch of ≤20 samples of the same matrix per day. • Must be analyzed on each	• Concentration does not exceed the total PCB method reporting limit. • Must meet surrogate criteria	• Re-analyze method blank to determine if instrument contamination was the cause. If method blank re-analysis passes, then report samples.

	instrument used to analyze associated samples. • Must undergo all sample preparative procedures.	of 60 to 140 % recovery.	• If method blank is found to contain PCB contamination above total PCB reporting limit, then re-extract and re-analyze all associated samples. If no sample exists for re-extraction, report data flagged (B) to indicate method blank contamination or have client re-sample if possible.

Quality Control Acceptance Criteria and Corrective Action Plan

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Spike (LCS)	• One per extraction batch of ≤20 samples per matrix per day. The LCS is typically Aroclor 1242.	• Percent recovery of Aroclor 1242 on a total PCB basis must be within method limits of 60 to 140% • Must meet surrogate criteria of 60 to 140% recovery.	• Re-analyze LCS to determine if instrument was the cause. If LCS passes, then report samples. • If LCS recovery is still out of limits, then re-extract and re-analyze all associated samples. If no sample exists for re-extraction, report data flagged to indicate LCS failed recovery or have client re-sample if possible.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	• Normal method procedure is to extract and analyze a matrix spike sample. One MS per extraction batch of ≤20 samples per matrix per day. The MS is typically Aroclor 1242. • If requested, an MSD can be extracted an analyzed. The MSD would follow the above criteria as for the MS.	• Percent recovery for MS on a total PCB basis should be 60 to 140% • If MS/MSD is analyzed, relative percent difference (RPD) should be within 20%. • Must meet surrogate criteria of 60 to 140%(unless original unspiked sample is also outside of criteria).	• Re-analyze MS and/or MSD to determine if instrument was the cause. If MS and/or MSD pass, then report samples. • Check for errors such as calculations and spike preparation. • Check original unspiked sample results and surrogate recovery for indications of matrix effects. • If no errors are found, and the associated LCS is within 70 to 130%, then sample matrix effects are likely the cause. Note exceedence in case narrative.

20.0 Contingencies for Handling Out-Of-Control or Unacceptable Data

20.1 Data that is detected to be out-of-control for any reason, when compared to method acceptance criteria, will addressed in the following manner:

20.1.1 If the problem exists with the gas chromatographic instrumentation, appropriate action will be taken to repair and perform maintenance to bring the instrument back to operation condition. Once the instrumentation is determined to be correctly operating analysis can begin again.

20.1.2 If the problem exists with calibration standard solutions, the analyst will prepare new standards and discard the standard solutions that are suspect. Instrument calibration can be performed and analysis can begin once system is control.

20.1.3 If the problem exists with sample extraction and extract preparation, the extraction step that is producing the out-of-control situation will be diagnosed and rectified. Once the troubleshooting procedures correct the problem extraction can once again occur and analysis can continue.

20.1.4 In situations where data is reported under out-of-control conditions, the data will be annotated with data qualifiers and/or appropriate descriptive comments defining the nature of the excursion in the sample case narrative. If warranted, a corrective action report (CAR) will be issued to define the problem, steps to correct the problem, and final resolution.

21.0 Waste Management

21.1 All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste generated during the execution of this method.

21.2 Please refer to standard operating procedures NE089 and NE054 regarding how hazardous waste is handled and disposed of by the laboratory.

22.0 References:

22.1 US EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants," July, 1988.

22.2 Standard Methods for the Examination of Water and Wastewater, 19th Edition, Published by: American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1995.

22.3 US EPA SW-846, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," Office of Solid Waste and Emergency Response, 3rd Edition, 1986 and its updates.

22.4 New York State Department of Health, "Environmental Laboratory Approval Program Certification Manual," Wadsworth Center for Laboratories and Research, 1988.

22.5 Mullin, M.D. 1985. PCB Workshop, US EPA Large Lakes Research Station, Grosse Ile, MI, June.

22.6 M. Zell, K. Ballschmiter, Baseline Studies of the Global Pollution, III. Trace Analysis of Polychlorinated Biphenyls (PCB) by ECD Glass Capillary Gas Chromatography in Environmental Samples of Different Trophic Levels, Fresenius Z. Anal. Chem., 304, 337-349, 1980.

22.7 M.D. Mullin, C.M. Pochini, S. McCrindle, M. Romkes, S.H. Save, "High-Resolution PCB Analysis: Synthesis and Chromatographic Properties of All 209 PCB Congeners," Environ. Sci. Technol., Vol 18, No. 6, pp 468-476, 1984.

22.8 D.L. Swackhamer, "Quality Assurance Plan, Green Bay Mass Balance Study, 1. PCBs and Dieldrin, US EPA Great Lakes National Program Office" Quality Assurance Coordinator, Field and Analytical Methods Committees, University of Minnesota, December 11, 1987.

22.9 George M. Frame, Robert E. Wagner, James C. Carnahan, John F. Brown, Jr., Ralph J. May, Lynn A. Smullen, and Donna L. Bedard, "Comprehensive, Quantitative, Congener-Specific Analyses of Eight Aroclors and Complete PCB Congener Assignments on DB-1 Capillary GC Columns ", Chemosphere, Vol. 33, No. 4, pp. 603-623, 1996.

22.10 "Standard Operating Procedure for the Gas Chromatographic Analysis of Hydrophobic Organic Contaminant Extracts from Great Lakes Water Samples", USEPA Great Lakes National Program Office, 77 West Jackson Boulevard, Chicago, IL 60604-2590, GLNPO Organics SOP – 10, 6/1/94:Revision 2

22.11 "Biphenyls and Halogenated Pesticides by High Resolution Gas Chromatography", M.D. Mullin, Large Lakes Research Station, LLRS-SOP-ORG-013, revision 2, August 3, 1990, p 1-10.

22.12 Contract Laboratory Program – Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration. Document OLM3.2, 1996.

23.0 Tables, Diagrams, Flowcharts and Validation Data

APPENDIX A
Congener Composition of Multi-Aroclor Calibration Standard (6.27 ug/mL)

Congener Composition of Mixed Aroclor High-level Standard (6270ng/mL)
(Aroclor 1232, 1248, 1262 in a ratio of 25:18:18)

Number	DB-1 Peak Number [1]	IUPAC # [2]	Amount ng/mL
1	2	**001**	438.57
2	3	**002**	-
3	4	**003**	255.84
4	5	**004 010**	124.26
5	6	**007 009**	43.85
6	7	**006**	69.40
7	8	**005 008**	511.66
8	9	*014*	-
9	10	**019**	10.24
10	11	*030*	-
11	12	**011**	-
12	13	**012 013**	9.75
13	14	**015 018**	135.22
14	15	**017**	135.22
15	16	**024 027**	9.50
16	17	**016 032**	142.53
17	19	*023* **034** *054*	-
18	20	**029**	1.94
19	21	**026**	26.32
20	22	**025**	11.69
21	23	**031**	150.68
22	24	**028** *050*	192.86
23	25	**020** *021* **033 053**	145.16
24	26	**022 051**	105.99
25	27	**045**	32.52
26	28	*036*	-
27	29	**046**	14.62
28	30	*039*	-
29	31	**052** *069 073*	174.33
30	32	**043 049**	84.06
31	33	*038* **047**	36.55
32	34	**048** *075*	36.55
33	35	*062 065*	-
34	36	**035**	-
35	37	*104* **044**	157.16
36	38	**037 042 059**	95.03
37	39	**041 064 071** *072*	149.85
38	41	*068* **096**	-
39	42	**040**	34.36
40	43	**057** *103*	-
41	44	*058* **067** *100*	4.02
42	45	**063**	7.68
43	46	**074** *094 061*	69.44

Number	DB-1 Peak Number [1]	IUPAC # [2]	Amount ng/mL
44	47	**070**	124.26
45	48	**066** *076 098 080 093* **095 102** *088*	263.14
46	49	*055* **091** *121*	18.64
47	50	**056 060**	127.91
48	51	**084 092** *155*	65.78
49	52	**089**	3.66
50	53	**090 101**	65.78
51	54	*079* **099** *113*	27.04
52	55	**119** *150*	1.02
53	56	*078* **083** *112 108*	5.48
54	57	**097** *152 086*	20.46
55	58	*081* **087** *117 125* **115** *145*	42.39
56	59	*111 116* **085**	25.59
57	60	*120* **136**	27.41
58	61	**077 110** *148*	77.84
59	62	*154*	-
60	63	**082**	16.08
61	64	**151**	62.15
62	65	**124 135**	10.60
63	66	**144**	21.93
64	67	*107* **109** *147*	4.75
65	68	*123*	-
66	69	*106* **118** *139* **149**	146.19
67	70	*140*	-
68	71	**114 134** *143*	7.38
69	72	**122 131 133** *142*	1.06
70	73	**146** *165 188*	14.26
71	74	**105 132** *161*	49.52
72	75	**153**	107.64
73	76	*127 168 184*	-
74	77	**141**	62.13
75	78	**179**	53.36
76	79	**137**	2.74
77	80	**130 176**	9.50
78	82	**138 163 164**	98.68
79	83	**158** *160 186*	9.13
80	84	*126* **129**	0.47
81	85	*166* **178**	40.20
82	87	**175** *159*	7.31
83	88	*182* **187**	131.57
84	89	**128** *162*	3.66
85	90	**183**	62.13
86	91	**167**	1.79
87	92	**185**	17.17
88	93	**174** *181*	116.95

Number	DB-1 Peak Number [1]	IUPAC # [2]	Amount ng/mL
89	94	**177**	62.13
90	95	**156 171**	28.88
91	96	**157 202**	2.41
92	98	**173**	1.39
93	99	**201**	14.26
94	100	**172** *204*	20.46
95	101	*192* **197**	4.02
96	102	**180**	222.94
97	103	**193**	15.35
98	104	**191**	4.38
99	105	**200** *169*	15.71
100	106	**170**	46.78
101	107	**190**	15.35
102	108	**198**	4.38
103	109	**199**	153.50
104	110	**196 203**	157.16
105	111	**189**	1.46
106	112	**195**	20.21
107	113	**208**	9.02
108	114	*207*	3.40
109	115	**194**	65.78
110	116	**205**	4.02
111	117	**206**	24.85
112	118	*209*	0.44

1 - Note that 5 DB-1/ZB-1 peaks (PK18, PK40, PK81, PK86, PK97) have been removed from the DB-1/ZB-1 peak numbering scheme. The following low-level congeners that were designated as separately eluting peaks have been determined to co-elute with another congener. The DB-1/ZB-1 peak numbers are no longer required for these congeners, but the original DB-1/ZB-1 numbering system has remained intact for all other peaks.
PK 18 (23) now elutes in PK 19 (23,34,54)
PK 40 (68) now elutes in PK 41 (68,96)
PK 81 (176) now elutes in PK 80 (130,176)
PK 86 (166) now elutes in PK 85 (166,178)
PK 97 (157) now elutes in PK 96 (157,202)

2 - IUPAC congener numbers listed in boldface font were found to be present in at least one of the Aroclor at or above 0.05 weight percent. These congeners should be considered the primary congeners existing in a peak composed of co-eluting congeners. IUPAC congener numbers listed in italic font were absent or present below 0.05 weight percent.

APPENDIX B

DB-1/ ZB-1 Capillary GC Chromatograms:

1.) 6.27 ug/mL Calibration Standard

Sample Name:	ICAL0926A	Sample Amount:	
Sample ID:	ICAL 6.27 PPM	Dilution:	
Date Acquired:	09/26/2006 21:08:21	Processing Method:	CSGB_HL_092606

2.) Supplemental Congener Standard

Chromatogram Report Congener Specific PCB Green Bay
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518) 346-4592 Fax: (518) 381-6055

Sample Name:	SC0926A	**Sample Amount:**	1
Sample ID:	SUP CONG STD 2000/50 ng/mL	**Dilution:**	1
Date Acquired:	09/26/2006 23:21:18	**Processing Method:**	CSGB_HL_092606

APPENDIX C

Example of Reporting Format

Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, NY 12308
(518) 346-4592 Fax (518) 381-6055

PCB Congener Amount Report

Customer: CLIENT NAME
Sample Description: SAMPLE IDENTIFICATION
Comment: PROJECT INFORMATION
Date Acquired: 09/27/2006 13:46:46
Lab Sample ID: AJ11122
LRF ID: 0609110-05
Lab File ID: GC17-80-17

Type for Mixed Peak Deconvolution = S

Total PCBs in sample = 8.76 ug/g

PCB Homolog Distribution

Homologs	Weight %	Mole %
Mono	0.12	0.18
Di	0.83	1.11
Tri	14.17	16.28
Tetra	51.77	53.12
Penta	22.98	20.99
Hexa	7.78	6.56
Hepta	1.83	1.39
Octa	0.48	0.33
Nona	0.05	0.03
Deca	0.00	0.00

Nominal 'Aroclor' Distribution

Aroclor	Indicator Peak (PK # / IUPAC #)	Amount ug/g	Percent Sediment	Percent Biota
A1221	2/001	0.0101	0.913	0.767
A1242	23+24/31+28	0.7014	63.3	53.2
A1254SED	61/100	0.3591	32.4	
A1254BIO	69+75+82/149+153+138	0.5695		43.2
A1260	102/180	0.0341	3.08	2.59
A1268	115/194	0.0025	0.228	0.192

Ortho Cl / biphenyl Residue = 1.64
Meta + Para Cl / biphenyl Residue = 2.57
Total Cl / biphenyl Residue = 4.21

0609110-05 10/04/2006 Nea Lims Version : 4.1.0.1 Page 1

Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, NY 12308
(518) 346-4592 Fax (518) 381-6055

PCB Congener Amount Report

Customer: CLIENT NAME
Sample Description: SAMPLE IDENTIFICATION
Comment: PROJECT INFORMATION
Date Acquired: 09/27/2006 13:46:46
Lab Sample ID: AJ11122
LRF ID: 0609110-05
Lab File ID: GC17-80-17

Type for Mixed Peak Deconvolution = S

DB-1 Peak Number	Retention Time	Molecular Weight	Peak Area	Amount (ug/g)	Sample	MDL (ug/g)	RL (ug/g)	Qual
2	11.75	188.7	7	0.0101	0.0536	0.00672	0.160	J
3	12.78	188.7				0.167	36.6	U
4	12.88	188.7				0.00287	0.0936	U
5	13.51	223.1	98	0.0546	0.245	0.00197	0.0455	B
6	14.36	223.1	14	0.00121	0.00543	0.000789	0.0160	J
7	14.64	223.1	26	0.00456	0.0205	0.000808	0.0254	JB
8	14.84	223.1	33	0.0110	0.0494	0.00613	0.187	J
9	15.40	223.1				0.00195	0.915	U
10	15.49	257.5	63	0.00685	0.0266	0.00161	0.00374	
11	15.95	257.5				0.00169	0.915	U
12	16.01	223.1				0.00457	0.915	U
13	16.22	223.1				0.00146	0.00357	U
14	16.38	249.0	37	0.00382	0.0153	0.00250	0.0495	J
15	16.42	257.5	33	0.00711	0.0276	0.00146	0.0495	JB
16	16.73	257.5	81	0.00502	0.0195	0.000162	0.00347	
17	16.97	257.5	17	0.00200	0.00778	0.00190	0.0521	J
19	17.43	267.9	122	0.0120	0.0449	0.00117	0.915	J
20	17.63	257.5	20	0.00129	0.00500	0.000259	0.000710	
21	17.74	257.5	1358	0.126	0.487	0.000532	0.00963	
22	17.82	257.5	282	0.0197	0.0765	0.000192	0.00428	
23	18.02	257.5	3061	0.236	0.917	0.00274	0.0551	
24	18.07	257.5	6708	0.465	1.81	0.00303	0.0706	
25	18.40	259.5	190	0.0168	0.0647	0.00267	0.0531	J
26	18.66	258.7	1380	0.127	0.491	0.00163	0.0388	B
27	18.88	292.0	358	0.0293	0.100	0.000483	0.0119	B
28	19.02	257.5				0.00134	0.915	U
29	19.16	292.0	6			0.00226	0.00535	U
30	19.28	257.5				0.00170	0.915	U
31	19.45	292.0	7735	0.876	3.00	0.00311	0.0638	
32	19.62	292.0	7769	0.442	1.51	0.00150	0.0308	
33	19.74	292.0	4969	0.201	0.689	0.000556	0.0134	B
34	19.80	292.0	287	0.0164	0.0563	0.000470	0.0134	B
35	19.94	292.0				0.00195	0.915	U
36	20.01	257.5				0.00124	0.915	U

DB-1 Peak Number	Retention Time	Molecular Weight	Peak Area	Amount (ug/g)	Sample	MDL (ug/g)	RL (ug/g)	Qual
37	20.19	292.0	5094	0.368	1.26	0.00188	0.0575	
38	20.32	272.4	4204	0.389	1.43	0.00170	0.0348	B
39	20.67	292.0	5889	0.338	1.16	0.00166	0.0548	B
41	20.84	326.4	313	0.0289	0.0887	0.00185	0.915	J
42	20.93	292.0	1074	0.0723	0.248	0.000624	0.0126	
43	21.17	298.9	257	0.0169	0.0566	0.00115	0.915	J
44	21.36	298.9	404	0.0183	0.0613	0.000370	0.00147	
45	21.51	292.0	1015	0.0492	0.169	0.00134	0.00281	
46	21.68	292.0	6784	0.262	0.898	0.000807	0.0254	
47	21.81	292.0	8579	0.419	1.43	0.00197	0.0455	
48	21.93	293.5	12842	0.932	3.18	0.00295	0.0963	
49	22.23	324.7	1416	0.0822	0.253	0.000251	0.00682	B
50	22.54	292.0	7587	0.361	1.24	0.00144	0.0468	
51	22.79	326.4	2486	0.309	0.947	0.000639	0.0241	
52	22.87	326.4	10	0.000566	0.00173	0.000524	0.00134	J
53	23.03	326.4	4934	0.283	0.868	0.000660	0.0241	
54	23.23	326.4	2847	0.104	0.318	0.000444	0.00989	
55	23.51	326.4	196	0.00379	0.0116	0.000139	0.000375	
56	23.60	326.4	599	0.0348	0.107	0.000817	0.00200	
57	23.81	326.4	2069	0.0877	0.269	0.000276	0.00749	
58	23.99	326.4	4055	0.206	0.630	0.000582	0.0155	B
59	24.14	326.4	2241	0.0936	0.287	0.000369	0.00936	
60	24.29	360.9	327	0.0164	0.0453	0.000528	0.0100	B
61	24.39	326.4	6279	0.359	1.10	0.00126	0.0285	
62	24.66	360.9				0.00751	0.915	U
63	24.76	326.4	1130	0.0498	0.153	0.000248	0.00588	
64	25.05	360.9	685	0.0358	0.0993	0.000590	0.0227	B
65	25.20	350.5	509	0.0158	0.0450	0.000177	0.00388	
66	25.23	360.9	216	0.0179	0.0495	0.000251	0.00802	
67	25.32	336.8	719	0.0376	0.112	0.0000683	0.00174	
68	25.41	326.4	199	0.00982	0.0301	0.00131	0.915	J
69	25.52	337.5	6659	0.321	0.952	0.00155	0.0535	
70	25.61	360.9				0.00127	0.915	U
71	25.92	347.8	458	0.0175	0.0503	0.000747	0.00270	
72	26.12	336.8	216	0.00450	0.0134	0.000151	0.000416	
73	26.39	360.9	556	0.0243	0.0673	0.000155	0.00522	
74	26.51	347.8	4239	0.147	0.422	0.000654	0.0181	
75	26.68	360.9	2402	0.0938	0.260	0.00128	0.0394	
76	26.78	360.9				0.000917	0.915	U
77	27.20	360.9	649	0.0391	0.108	0.000644	0.0227	
78	27.28	395.3	102	0.00526	0.0133	0.000501	0.0195	J
79	27.49	360.9	235	0.0167	0.0462	0.000498	0.00100	
80	27.57	360.9	246	0.00572	0.0158	0.000193	0.00347	
82	27.86	360.9	3760	0.154	0.427	0.00146	0.0361	
83	28.05	360.9	449	0.0154	0.0427	0.000180	0.00334	
84	28.26	360.9	188	0.00123	0.00340	0.0000598	0.000173	
85	28.60	395.3	190	0.0138	0.0350	0.000467	0.0147	J
87	28.85	395.3	29	0.00168	0.00426	0.00111	0.00267	J
88	29.04	395.3	627	0.0269	0.0680	0.00126	0.0481	J
89	29.16	360.9	768	0.0182	0.0506	0.0000432	0.00134	
90	29.34	395.3	224	0.00953	0.0241	0.000668	0.0227	J
91	29.61	360.9	127	0.00326	0.00903	0.000306	0.000656	

DB-1 Peak Number	Retention Time	Molecular Weight	Peak Area	Amount (ug/g)	Sample	MDL (ug/g)	RL (ug/g)	Qual
92	29.96	394.3	48	0.00137	0.00348	0.000368	0.00628	J
93	30.33	394.3	289	0.0124	0.0316	0.00146	0.0428	J
94	30.60	394.3	262	0.0124	0.0314	0.000735	0.0227	J
95	30.89	382.2	680	0.0294	0.0770	0.000313	0.0106	
96	31.15	429.8	211	0.00128	0.00297	0.0000432	0.000883	
98	31.30	395.3	33	0.000698	0.00177	0.000256	0.000508	
99	31.71	429.8	28	0.00119	0.00278	0.000168	0.00522	J
100	31.95	395.3				0.000312	0.00749	U
101	32.19	429.8	59	0.00256	0.00596	0.000493	0.00147	
102	32.42	395.3	907	0.0341	0.0863	0.00271	0.0816	J
103	32.64	395.3	72	0.00306	0.00774	0.000159	0.00562	J
104	33.01	395.3	57	0.00204	0.00516	0.000613	0.00160	
105	33.31	429.8	47	0.00176	0.00409	0.000197	0.00575	J
106	34.48	395.3	583	0.0138	0.0348	0.000617	0.0171	J
107	34.74	395.3	134	0.00365	0.00924	0.000185	0.00562	J
108	35.61	429.8	25	0.000673	0.00157	0.000502	0.00160	J
109	35.86	429.8	225	0.0139	0.0324	0.00150	0.0562	J
110	36.38	429.8	300	0.0168	0.0392	0.00149	0.0575	J
111	37.52	395.3	67	0.00159	0.00402	0.000244	0.000584	
112	39.13	429.8	40	0.000840	0.00195	0.000229	0.00739	J
113	39.64	464.2	51	0.00293	0.00631	0.00181	0.00330	J
114	40.57	464.2						U
115	41.97	429.8	98	0.00253	0.00589	0.000800	0.0241	J
116	42.86	429.8	11			0.000799	0.00147	U
117	48.02	464.2	48	0.00130	0.00279	0.000470	0.00909	J
118	54.01	498.6				0.0000781	0.000162	U

Total Concentration = 8.76 ug/g 0.0988596 2.29255

Total Millimoles = 0.029

Average Molecular Weight = 299.1

Number of Calibrated Peaks Found = 96

Internal Standard Retention Time = 46.50 minutes

Internal Standard Peak Area = 123355.5

Northeast Analytical, Inc.
2190 Technology Drive
Schenectady, NY 12308
(518) 346-4592 Fax (518) 381-6055

PCB Congener Amount Report

Customer: CLIENT NAME
Sample Description: SAMPLE IDENTIFICATION
Comment: PROJECT INFORMATION
Date Acquired: 09/27/2006 13:46:46
Lab Sample ID: AJ11122
LRF ID: 0609110-05
Lab File ID: GC17-80-17

Type for Mixed Peak Deconvolution = S

DB-1 Peak [1] Number	Retention Time	T-CL:O-CL	IUPAC # [2]	RRT	Congeners [3]	Weight Percent	Mole Percent
2	11.75	1:1	**001**	0.2527	2	0.115	0.183
3	12.78	1:0	**002**		3	-	-
4	12.88	1:0	**003**		4	-	-
5	13.51	2:2	**004 010**	0.2905	2-2; 26	0.624	0.836
6	14.36	2:1	**007 009**	0.3083	24; 25	0.014	0.019
7	14.64	2:1	**006**	0.3148	2-3	0.052	0.070
8	14.84	2:1	**005 008**	0.3191	23; 2-4	0.126	0.169
9	15.40	2:0	*014*		35	-	-
10	15.49	3:3	**019**	0.3331	26-2	0.078	0.091
11	15.95	3:2	*030*		246	-	-
12	16.01	2:0	**011**		3-3	-	-
13	16.22	2:0	**012 013**		34; 3-4	-	-
14	16.38	2:0 3:2	**015 018**	0.3523	4-4; 25-2	0.044	0.052
15	16.42	3:2	**017**	0.3531	24-2	0.081	0.094
16	16.73	3:2	**024 027**	0.3598	236; 26-3	0.057	0.067
17	16.97	3:2	**016 032**	0.3649	23-2; 26-4	0.023	0.027
19	17.43	3:1 4:4	*023* **034** *054*	0.3748	235; 35-2; 26-26	0.137	0.153
20	17.63	3:1	**029**	0.3791	245	0.015	0.017
21	17.74	3:1	**026**	0.3815	25-3	1.433	1.665
22	17.82	3:1	**025**	0.3832	24-3	0.225	0.261
23	18.02	3:1	**031**	0.3875	25-4	2.698	3.134
24	18.07	3:1 4:3	**028** *050*	0.3886	24-4; 246-2	5.312	6.170
25	18.40	3:1 4:3	**020** *021* **033 053**	0.3957	23-3; 234; 34-2; 25-26	0.192	0.221
26	18.66	3:1 4:3	**022 051**	0.4013	23-4; 24-26	1.452	1.678
27	18.88	4:3	**045**	0.4060	236-2	0.334	0.342
28	19.02	3:0	*036*		35-3	-	-
29	19.16	4:3	**046**		23-26	-	-
30	19.28	3:0	*039*		35-4	-	-
31	19.45	4:2	**052** *069 073*	0.4183	25-25; 246-3; 26-35	10.007	10.251
32	19.62	4:2	**043 049**	0.4219	235-2; 24-25	5.047	5.170
33	19.74	4:2	*038* **047**	0.4245	345; 24-24	2.298	2.354
34	19.80	4:2	**048** *075*	0.4258	245-2; 246-4	0.188	0.192
35	19.94	4:2	*062 065*		2346; 2356	-	-
36	20.01	3:0	**035**		34-3	-	-
37	20.19	5:4 4:2	*104* **044**	0.4342	246-26; 23-25	4.200	4.302
38	20.32	3:0 4:2	**037 042 059**	0.4370	34-4; 23-24; 236-3	4.442	4.878
39	20.67	4:2	**041 064 071** *072*	0.4445	234-2; 236-4; 26-34; 25-35	3.860	3.954

DB-1 Peak [1] Number	Retention Time	T-CL:O-CL	IUPAC # [2]	RRT	Congeners [3]	Weight Percent	Mole Percent
41	20.84	5:4	*068* **096**	0.4482	24-35; 236-26	0.330	0.303
42	20.93	4:2	**040**	0.4501	23-23	0.826	0.846
43	21.17	4:1 5:3	**057** *103*	0.4553	235-3; 246-25	0.193	0.193
44	21.36	4:1 5:3	*058* **067** *100*	0.4594	23-35; 245-3; 246-24	0.209	0.210
45	21.51	4:1	**063**	0.4626	235-4	0.562	0.576
46	21.68	4:1 5:3	**074** *094 061*	0.4662	245-4; 235-26; 2345	2.994	3.067
47	21.81	4:1	**070**	0.4690	25-34	4.785	4.901
48	21.93	4:1 5:3	**066** *076 098 080 093* **095 102** *088*	0.4716	24-34; 345-2; 246-23; 35-35; 2356-2; 236-25; 245-26; 2346-2	10.646	10.850
49	22.23	4:1 5:3	*055* **091** *121*	0.4781	234-3; 236-24; 246-35	0.939	0.865
50	22.54	4:1	**056 060**	0.4847	23-34; 234-4	4.123	4.224
51	22.79	5:3 6:4	**084 092** *155*	0.4901	236-23; 235-25; 246-246	3.530	3.235
52	22.87	5:3	**089**	0.4918	234-26	0.006	0.006
53	23.03	5:2	**090 101**	0.4953	235-24; 245-25	3.235	2.964
54	23.23	5:2	*079* **099** *113*	0.4996	34-35; 245-24; 236-35	1.187	1.088
55	23.51	5:2 6:4	**119** *150*	0.5056	246-34; 236-246	0.043	0.040
56	23.60	5:2	*078* **083** *112 108*	0.5075	345-3; 235-23; 2356-3; 2346-3	0.397	0.364
57	23.81	5:2 6:4	**097** *152 086*	0.5120	245-23; 2356-26; 2345-2	1.001	0.918
58	23.99	5:2	*081* **087** *117 125* **115** *145*	0.5159	345-4; 234-25; 2356-4; 345-26; 2346-4; 2346-26	2.347	2.151
59	24.14	5:2	*116* **085** *111*	0.5191	23456; 234-24; 235-35	1.069	0.980
60	24.29	6:4	*120* **136**	0.5224	245-35; 236-236	0.187	0.155
61	24.39	5:2	**077 110** *148*	0.5245	34-34; 236-34; 235-246	4.100	3.758
62	24.66	6:3	*154*		245-246	-	-
63	24.76	5:2	**082**	0.5325	234-23	0.569	0.521
64	25.05	6:3	**151**	0.5387	2356-25	0.409	0.339
65	25.20	5:1 6:3	**124 135**	0.5419	345-25; 235-236	0.180	0.154
66	25.23	6:3	**144**	0.5426	2346-25	0.204	0.169
67	25.32	5:1 6:3	*107* **109** *147*	0.5445	234-35; 235-34; 2356-24	0.430	0.382
68	25.41	5:1	*123*	0.5465	345-24	0.112	0.103
69	25.52	5:1 6:3	*106* **118** *139* **149**	0.5488	2345-3; 245-34; 2346-24; 236-245	3.671	3.253
70	25.61	6:3	*140*		234-246	-	-
71	25.92	5:1 6:3	**114 134** *143*	0.5574	2345-4; 2356-23; 2345-26	0.200	0.172
72	26.12	5:1 6:3	**122 131 133** *142*	0.5617	345-23; 2346-23; 235-235; 23456-2	0.051	0.046
73	26.39	6:2	**146** *165 188*	0.5675	235-245; 2356-35; 2356-246	0.278	0.230
74	26.51	5:1 6:3	**105 132** *161*	0.5701	234-34; 234-236; 2346-35	1.677	1.442
75	26.68	6:2	**153**	0.5738	245-245	1.071	0.888
76	26.78	6:2	*127 168 184*		345-35; 246-345; 2346-246	-	-
77	27.20	6:2	**141**	0.5849	2345-25	0.447	0.370
78	27.28	7:4	**179**	0.5867	2356-236	0.060	0.045
79	27.49	6:2	**137**	0.5912	2345-24	0.191	0.158
80	27.57	6:2 7:4	**130 176**	0.5929	234-235; 2346-236	0.065	0.054
82	27.86	6:2	**138 163 164**	0.5991	234-245; 2356-34; 236-345	1.761	1.460
83	28.05	6:2	**158** *160 186*	0.6032	2346-34; 23456-3; 23456-26	0.176	0.146
84	28.26	6:2	*126* **129**	0.6077	345-34; 2345-23	0.014	0.012
85	28.60	7:3	*166* **178**	0.6151	23456-4; 2356-235	0.158	0.119
87	28.85	7:3	**175** *159*	0.6204	2346-235; 2345-35	0.019	0.015
88	29.04	7:3	*182* **187**	0.6245	2345-246; 2356-245	0.307	0.232
89	29.16	6:2	**128** *162*	0.6271	234-234; 235-345	0.208	0.173
90	29.34	7:3	**183**	0.6310	2346-245	0.109	0.082
91	29.61	6:1	**167**	0.6368	245-345	0.037	0.031
92	29.96	7:3	**185**	0.6443	23456-25	0.016	0.012
93	30.33	7:3	**174** *181*	0.6523	2345-236; 23456-24	0.142	0.108
94	30.60	7:3	**177**	0.6581	2356-234	0.142	0.107
95	30.89	6:1 7:3	**156 171**	0.6643	2345-34; 2346-234	0.336	0.263
96	31.15	8:4	**157 202**	0.6699	234-345; 2356-2356	0.015	0.010
98	31.30	7:3	**173**	0.6731	23456-23	0.008	0.006
99	31.71	8:4	**201**	0.6819	2346-2356	0.014	0.009
100	31.95	7:2	**172** *204*		2345-235; 23456-246	-	-
101	32.19	8:4	*192* **197**	0.6923	23456-35; 2346-2346	0.029	0.020
102	32.42	7:2	**180**	0.6972	2345-245	0.390	0.295
103	32.64	7:2	**193**	0.7019	2356-345	0.035	0.026
104	33.01	7:2	**191**	0.7099	2346-345	0.023	0.018
105	33.31	8:4	**200** *169*	0.7163	23456-236; 345-345	0.020	0.014
106	34.48	7:2	**170**	0.7415	2345-234	0.157	0.119

DB-1 Peak[1] Number	Retention Time	T-CL:O-CL	IUPAC #[2]	RRT	Congeners[3]	Weight Percent	Mole Percent
107	34.74	7:2	**190**	0.7471	23456-34	0.042	0.032
108	35.61	8:3	**198**	0.7658	23456-235	0.008	0.005
109	35.86	8:3	**199**	0.7712	2345-2356	0.159	0.111
110	36.38	8:3	**196 203**	0.7824	2345-2346; 23456-245	0.192	0.134
111	37.52	7:1	**189**	0.8069	2345-345	0.018	0.014
112	39.13	8:3	**195**	0.8415	23456-234	0.010	0.007
113	39.64	9:4	**208**	0.8525	23456-2356	0.033	0.022
114	40.57	9:4	*207*		23456-2346	-	-
115	41.97	8:2	**194**	0.9026	2345-2345	0.029	0.020
116	42.86	8:2	**205**		23456-345	-	-
117	48.02	9:3	**206**	1.033	23456-2345	0.015	0.010
118	54.01	10:4	*209*		23456-23456	-	-

Concentration = 8.76 ug/g

Total Millimoles = 0.029

Average Molecular Weight = 299.1

Number of Calibrated Peaks Found = 96

[1] - Note that five DB-1 peaks (PK18, PK40, PK81, PK86, PK97) have been removed from the DB-1 peak numbering scheme. The following low level congeners that were designated as separately eluting peaks have been determined to co-elute with another congener. The DB-1 peak numbers are no longer required for these congeners, but the original DB-1 numbering system has remained intact for all other peaks.

PK 18 (23) now elutes in PK 19 (23,34,54)
PK 40 (68) now elutes in PK 41 (68,96)
PK 86 (166) now elutes in PK 85 (166,178)
PK 97 (157) now elutes in PK 96 (157,202)

[2] - IUPAC congener numbers listed in **boldface** font were found to be present in at least one of the Aroclors at or above 0.05 weight percent. These congeners should be considered the primary congeners existing in a peak composed of co-eluting congeners. IUPAC congener numbers listed in *italic* font were absent or present below 0.05 weight percent.

[3] - PCB congener identification is denoted by position of the chlorine atoms on each ring of the biphenyl molecule. Designation used in this report has unprimed chlorines separated from primed chlorines by a hyphen that represents separation of the biphenyl rings.

[4] - DB-1 peaks may include one or more coeluting PCB congeners. In the case of some peaks, the congeners assigned to the peak consist of coeluting congeners and a congener that is resolved or is just slightly out of the normal retention time window of ± 0.07 minutes. If detection of one of the resolved congeners occurs, a comment will be included in the report narrative indicating the assigned DB-1 peak includes the presence of the resolved congener. The DB-1 peaks consisting of coeluting congeners and a congener that is resolved are as follows:

DB-1 Peak	Resolved Congener (IUPAC #)
37 (**44**,104)	*104*
48 (**66**,76,98,80,93,**95**,**102**,88)	*80,88,93*
56 (78,**83**,112,108)	*108*
61 (**77**,**110**,148)	**77**
72 (**122**,131,133,142)	**122**
89 (**128**,162)	*162*
105(**200**,169)	*169*

Appendix D
High Level Linearity Study

PCB STANDARD EVALUATION SUMMARY
THREE POINT INITIAL CALIBRATION CHECK FOR LINEARITY

LAB NAME: NORTHEAST ANALYTICAL, INC.

LAB CODE: NYS ELAP #11078

INSTRUMENT ID: GC #17

GC COLUMN ID: J&W, DB-1, 30 METER

STANDARD A:	FILENAME: CS0222A	AMT: 31.35 PPM	DATE: 02/22/07	TIME: 18:44
STANDARD B:	FILENAME: CS0222B	AMT: 6.27 PPM	DATE: 02/22/07	TIME: 20:58
STANDARD C:	FILENAME: CS0222C	AMT: 1.25 PPM	DATE: 02/23/07	TIME: 01:26

STANDARD D: FILENAME: SCS0222 AMT: SEE LIST DATE: 02/22/07 TIME: 23:12

Peak # (IUPAC#)	PCB Congener Analyzed	Conc. ppm
3 (2)	3-Chlorobiphenyl	2.000
9 (14)	3,5-Dichlorobiphenyl	0.050
11 (30)	2,4,6-Trichlorobiphenyl	0.050
12 (11)	3,3'-Dichlorobiphenyl	0.050
19 (23,34,54)	2',3,5-Trichlorobiphenyl	0.050
28 (36)	3,3',5-Trichlorobiphenyl	0.050
30 (39)	3,4',5-Trichlorobiphenyl	0.050
35 (62,65)	2,3,5,6-Tetrachlorobiphenyl	0.050
36 (35)	3,3',4-Trichlorobiphenyl	0.050
41 (68,96)	2,2',3,6,6'-Pentachlorobiphenyl	0.050
43 (57,103)	2,2',4,5',6-Pentachlorobiphenyl	0.050
62 (154)	2,2'4,4',5,6'-Hexachlorobiphenyl	0.050
68 (123)	2',3,4,4',5-Pentachlorobiphenyl	0.050
70 (140)	2,2',3,4,4',6'-Hexachlorobiphenyl	0.050
76 (127,168,184)	3,3',4,5,5'-Pentachlorobiphenyl	0.050

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PCB STANDARD EVALUATION SUMMARY
THREE POINT INITIAL CALIBRATION CHECK FOR LINEARITY

	DB-1 PEAK NUMBER (IUPAC Congener Number)	RRF STD A CS0222A	RRF STD B CS0222B	RRF STD C CS0222C	RRF STD D SCS0222	% RSD (< 20%)
1	2 (1)	0.02259	0.02901	0.02853		13.4
2	3 (2)*				0.00446	N/A
3	4 (3)	0.01321	0.01493	0.01349		6.66
4	5 (4,10)	0.05826	0.06745	0.06532		7.56
5	6 (7,9)	0.38172	0.46862	0.46682		11.3
6	7 (6)	0.17915	0.22338	0.22642		12.6
7	8 (5,8)	0.09096	0.11552	0.12834		17.0
8	9 (14)*				0.18206	N/A
9	10 (19)	0.32003	0.36149	0.33953		6.09
10	11 (30)*				0.70230	N/A
11	12 (11)*				0.06035	N/A
12	13 (12,13)	0.31192	0.31599	0.30304		2.13
13	14 (15,18)	0.29075	0.37005	0.40785		16.8
14	15 (17)	0.14609	0.18520	0.19256		14.3
15	16 (24,27)	0.55026	0.57808	0.58698		3.35
16	17 (16,32)	0.25978	0.32568	0.33834		13.7
17	18 **					**
18	19 (23,34,54)*				0.41549	N/A
19	20 (29)	0.49480	0.55272	0.50063		6.18
20	21 (26)	0.35075	0.41316	0.38550		8.16
21	22 (25)	0.48053	0.53416	0.49565		5.49
22	23 (31)	0.40680	0.49337	0.57867		17.4
23	24 (28,50)	0.45800	0.56209	0.62389		15.3
24	25 (20,21,33,53)	0.34347	0.43008	0.46038		14.8
25	26 (22,51)	0.34392	0.42267	0.44250		12.9
26	27 (45)	0.39420	0.47862	0.45889		9.9
27	28 (36)*				0.31903	N/A
28	29 (46)	0.36506	0.40325	0.38531		4.97
29	30 (39)*				0.31248	N/A
30	31 (52,69,73)	0.25601	0.33054	0.37383		18.6
31	32 (43,49)	0.52016	0.66698	0.74572		17.8
32	33 (38,47)	0.74962	0.96631	1.02130		15.7
33	34 (48,75)	0.54881	0.68828	0.68535		12.4
34	35 (62,65)*				0.83100	N/A
35	36 (35)*				0.29514	N/A
36	37 (104,44)	0.36042	0.45293	0.51976		18.0
37	38 (37,42,59)	0.42451	0.53545	0.55697		14.1
38	39 (41,64,71,72)	0.54586	0.66667	0.76262		16.5
39	40 **					**
40	41 (68,96)*				0.45828	N/A
41	42 (40)	0.48041	0.57885	0.56415		9.8
42	43 (57,103)*				0.62202	N/A
43	44 (58,67,100)	0.71833	0.71304	0.75228		2.93
44	45 (63)	0.69737	0.74557	0.74217		3.69
45	46 (74,94,61)	0.77442	0.96318	1.10146		17.3
46	47 (70)	0.59976	0.74962	0.87831		18.8

PCB STANDARD EVALUATION SUMMARY
THREE POINT INITIAL CALIBRATION CHECK FOR LINEARITY

	DB-1 PEAK NUMBER (IUPAC Congener Number)	RRF STD A CS0222A	RRF STD B CS0222B	RRF STD C CS0222C	RRF STD D SCS0222	% RSD (< 20%)
47	48 (66,76,98,80,93,95,102,88)	0.41348	0.51326	0.58991		17.5
48	49 (55,91,121)	0.54714	0.61399	0.57185		5.85
49	50 (56,60)	0.65387	0.77954	0.88680		15.1
50	51 (84,92,155)	0.25851	0.31137	0.32020		11.2
51	52 (89)	0.56478	0.54114	0.51399		4.71
52	53 (90,101)	0.49978	0.63772	0.72282		18.2
53	54 (79,99,113)	0.81510	1.01092	1.08160		14.2
54	55 (119,150)	1.53563	1.41040	1.58224		5.89
55	56 (78,83,112,108)	0.55891	0.55436	0.59615		4.02
56	57 (97,152,86)	0.73973	0.86278	0.86263		8.64
57	58 (81,87,117,125,115,145)	0.61771	0.74809	0.78074		12.1
58	59 (116,85,111)	0.76791	0.92249	0.93681		10.7
59	60 (120,136)	0.61615	0.74495	0.72593		10.0
60	61 (77,110,148)	0.51319	0.63536	0.70638		15.8
61	62 (154)*				0.74795	N/A
62	63 (82)	0.71358	0.81117	0.80435		7.02
63	64 (151)	0.55138	0.70086	0.77696		17.0
64	65 (124,135)	0.98386	1.18822	1.15365		9.9
65	66 (144)	0.41292	0.47150	0.44515		6.62
66	67 (107,109,147)	0.60733	0.53180	0.59399		6.98
67	68 (123)*				0.75721	N/A
68	69 (106,118,139,149)	0.59020	0.73584	0.87010		19.1
69	70 (140)*				0.81617	N/A
70	71 (114,134,143)	0.80053	0.80142	0.69286		8.16
71	72 (122,131,133,142)	1.18199	1.05810	1.46624		16.9
72	73 (146,165,188)	0.68697	0.78770	0.74836		6.85
73	74 (105,132,161)	0.85755	1.06403	1.12667		13.9
74	75 (153)	0.69524	0.87548	1.03850		19.7
75	76 (127,168,184)*				0.64655	N/A
76	77 (141)	0.48575	0.60953	0.63916		14.1
77	78 (179)	0.56653	0.72179	0.79553		16.8
78	79 (137)	0.37594	0.33346	0.33330		7.07
79	80 (130,176)	1.37320	1.52945	1.48899		5.54
80	81 **					**
81	82 (138,163,164)	0.68712	0.86463	0.97953		17.5
82	83 (158,160,186)	0.90898	0.97112	0.91740		3.62
83	84 (126,129)	3.59383	3.08366	2.81965		12.4
84	85 (166,178)	0.40833	0.47983	0.46097		8.24
85	86 **					**
86	87 (175,159)	0.52810	0.49041	0.42845		10.43
87	88 (182,187)	0.65075	0.82073	0.95553		18.9
88	89 (128,162)	1.21993	1.23308	1.22205		0.58
89	90 (183)	0.68082	0.84977	0.89965		14.2
90	91 (167)	0.81120	0.63450	0.85094		15.1
91	92 (185)	1.07086	1.24739	1.21824		8.03
92	93 (174,181)	0.65708	0.82060	0.93214		17.2

PCB STANDARD EVALUATION SUMMARY
THREE POINT INITIAL CALIBRATION CHECK FOR LINEARITY

	DB-1 PEAK NUMBER (IUPAC Congener Number)	RRF STD A CS0222A	RRF STD B CS0222B	RRF STD C CS0222C	RRF STD D SCS0222	% RSD (< 20%)
93	94 (177)	0.61058	0.75928	0.78218		13.0
94	95 (156,171)	0.71013	0.82515	0.77090		7.49
95	96 (157,202)	5.18082	5.76783	5.67256		5.69
96	97 **					**
97	98 (173)	1.19321	1.15191	0.91430		13.9
98	99 (201)	0.71911	0.77792	0.70789		5.12
99	100 (172,204)	0.64149	0.70896	0.66601		5.08
100	101 (192,197)	0.68289	0.69665	0.54763		12.8
101	102 (180)	0.73096	0.90383	1.07371		19.0
102	103 (193)	0.69292	0.74744	0.67318		5.46
103	104 (191)	0.72973	0.68096	0.64063		6.5
104	105 (200,169)	0.82643	0.86874	0.82524		2.95
105	106 (170)	1.21127	1.51047	1.59284		14.0
106	107 (190)	1.12545	1.25639	1.16608		5.67
107	108 (198)	1.12881	1.11950	1.11999		0.5
108	109 (199)	0.44072	0.56377	0.61375		16.5
109	110 (196,203)	0.48403	0.61754	0.68149		17.0
110	111 (189)	1.11803	1.05317	1.17492		5.46
111	112 (195)	1.46810	1.70680	1.73800		9.02
112	113 (208)	0.59157	0.55924	0.71851		13.5
113	114 (207)	1.17903	1.15468	1.43212		12.2
114	115 (194)	1.09041	1.37671	1.44694		14.5
115	116 (205)	0.97903	0.96908	1.07212		5.65
116	117 (206)	1.13252	1.28657	1.25055		6.59
117	118 (209)	1.48415	1.50043	1.53298		1.65

* - A separate PCB congener calibration standard is analyzed for these PCB congeners that do not exist at measurable levels in the Green Bay multi-Aroclor calibration. These so-called non-Aroclor PCB congeners (with the exception of peak 3(2)) are analyzed to provide accurate retention time information and relative response factors in the event they need to be quantified.

* *- Refinements in the elution position of several congeners has occurred due to the availability of individual congener standards. The following low level congeners that were designated as separately eluting peaks have been determined to co-elute with another congener. The DB-1 peak numbers are no longer required for these congeners, but the original DB-1 numbering system has remained intact for all other peaks.

PK 18 (23) now elutes in PK 19 (23,34,54)
PK 40 (68) now elutes in PK 41 (68,96)
PK 81 (176) now elutes in PK 80 (130,176)
PK 86 (166) now elutes in PK 85 (166,178)
PK 97 (157) now elutes in PK 96 (157,202)

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Low Level Linearity Study

PCB STANDARD EVALUATION SUMMARY
THREE POINT INITIAL CALIBRATION CHECK FOR LINEARITY

LAB NAME: NORTHEAST ANALYTICAL, INC.

LAB CODE: NYS ELAP #11078

INSTRUMENT ID: GC #17

GC COLUMN ID: J&W, DB-1, 30 METER

STANDARD A:	FILENAME: CS0222B	AMT: 6.27 PPM	DATE: 02/22/07	TIME: 20:58
STANDARD B:	FILENAME: CS0222C	AMT: 1.25 PPM	DATE: 02/23/07	TIME: 01:26
STANDARD C:	FILENAME: CS0222D	AMT: 0.125 PPM	DATE: 02/23/07	TIME: 03:39

STANDARD D: FILENAME: SCS0222 AMT: SEE LIST DATE: 02/22/07 TIME: 23:12

Peak # (IUPAC#)	PCB Congener Analyzed	Conc. ppm
3 (2)	3-Chlorobiphenyl	2.000
9 (14)	3,5-Dichlorobiphenyl	0.050
11 (30)	2,4,6-Trichlorobiphenyl	0.050
12 (11)	3,3'-Dichlorobiphenyl	0.050
19 (23,34,54)	2',3,5-Trichlorobiphenyl	0.050
28 (36)	3,3',5-Trichlorobiphenyl	0.050
30 (39)	3,4',5-Trichlorobiphenyl	0.050
35 (62,65)	2,3,5,6-Tetrachlorobiphenyl	0.050
36 (35)	3,3',4-Trichlorobiphenyl	0.050
41 (68,96)	2,2',3,6,6'-Pentachlorobiphenyl	0.050
43 (57,103)	2,2',4,5',6-Pentachlorobiphenyl	0.050
62 (154)	2,2'4,4',5,6'-Hexachlorobiphenyl	0.050
68 (123)	2',3,4,4',5-Pentachlorobiphenyl	0.050
70 (140)	2,2',3,4,4',6'-Hexachlorobiphenyl	0.050
76 (127,168,184)	3,3',4,5,5'-Pentachlorobiphenyl	0.050

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PCB STANDARD EVALUATION SUMMARY
THREE POINT INITIAL CALIBRATION CHECK FOR LINEARITY

	DB-1 PEAK NUMBER (IUPAC Congener Number)	RRF STD A CS0222B	RRF STD B CS0222C	RRF STD C CS0222D	RRF STD D SCS0222	% RSD (< 20%)
1	2 (1)	0.02891	0.02853	0.03116		4.82
2	3 (2)*				0.00446	N/A
3	4 (3)	0.01488	0.01349	0.01604		8.61
4	5 (4,10)	0.06723	0.06532	0.06747		1.77
5	6 (7,9)	0.46710	0.46682	0.50184		4.21
6	7 (6)	0.22265	0.22642	0.25201		6.84
7	8 (5,8)	0.11515	0.12834	0.12603		5.72
8	9 (14)*				0.18206	N/A
9	10 (19)	0.33737	0.33953	0.37428		5.91
10	11 (30)*				0.70230	N/A
11	12 (11)*				0.06035	N/A
12	13 (12,13)	0.31497	0.30304	0.33470		5.04
13	14 (15,18)	0.36884	0.40785	0.39877		5.21
14	15 (17)	0.18459	0.19256	0.18529		2.35
15	16 (24,27)	0.57620	0.58698	0.64280		5.94
16	17 (16,32)	0.32462	0.33834	0.33041		2.08
17	18 **					**
18	19 (23,34,54)*				0.41549	N/A
19	20 (29)	0.55092	0.50063	0.59326		8.46
20	21 (26)	0.41182	0.38550	0.40880		3.58
21	22 (25)	0.53243	0.49565	0.61153		10.8
22	23 (31)	0.49177	0.57867	0.57412		8.92
23	24 (28,50)	0.56027	0.62389	0.68446		9.97
24	25 (20,21,33,53)	0.42868	0.46038	0.44000		3.63
25	26 (22,51)	0.42130	0.44250	0.40433		4.52
26	27 (45)	0.47706	0.45889	0.47536		2.13
27	28 (36)*				0.31903	N/A
28	29 (46)	0.40194	0.38531	0.34615		7.58
29	30 (39)*				0.31248	N/A
30	31 (52,69,73)	0.32947	0.37383	0.39498		9.13
31	32 (43,49)	0.66481	0.74572	0.78145		8.18
32	33 (38,47)	0.96317	1.02130	1.05106		4.42
33	34 (48,75)	0.68605	0.68535	0.72837		3.52
34	35 (62,65)*				0.83100	N/A
35	36 (35)*				0.29514	N/A
36	37 (104,44)	0.45146	0.51976	0.49730		7.11
37	38 (37,42,59)	0.53371	0.55697	0.54218		2.16
38	39 (41,64,71,72)	0.66450	0.76262	0.77092		8.08
39	40 **					**
40	41 (68,96)*				0.45828	N/A
41	42 (40)	0.57697	0.56415	0.67780		10.3
42	43 (57,103)*				0.62202	N/A
43	44 (58,67,100)	0.71072	0.75228	0.72095		2.97
44	45 (63)	0.74315	0.74217	0.81364		5.35
45	46 (74,94,61)	0.96005	1.10146	1.11723		8.17
46	47 (70)	0.74718	0.87831	0.98003		13.4

PCB STANDARD EVALUATION SUMMARY
THREE POINT INITIAL CALIBRATION CHECK FOR LINEARITY

	DB-1 PEAK NUMBER (IUPAC Congener Number)	RRF STD A CS0222B	RRF STD B CS0222C	RRF STD C CS0222D	RRF STD D SCS0222	% RSD (< 20%)
47	48 (66,76,98,80,93,95,102,88)	0.51160	0.58991	0.60042		8.55
48	49 (55,91,121)	0.61200	0.57185	0.64033		5.66
49	50 (56,60)	0.77700	0.88680	0.88717		7.47
50	51 (84,92,155)	0.31036	0.32020	0.33427		3.74
51	52 (89)	0.53938	0.51399	0.46349		7.64
52	53 (90,101)	0.63565	0.72282	0.68050		6.41
53	54 (79,99,113)	1.00764	1.08160	0.96150		5.96
54	55 (119,150)	1.40582	1.58224	1.42089		6.65
55	56 (78,83,112,108)	0.55256	0.59615	0.66361		9.26
56	57 (97,152,86)	0.85998	0.86263	0.92393		4.10
57	58 (81,87,117,125,115,145)	0.74566	0.78074	0.80426		3.80
58	59 (116,85,111)	0.91949	0.93681	0.98626		3.66
59	60 (120,136)	0.74253	0.72593	0.72858		1.22
60	61 (77,110,148)	0.63330	0.70638	0.79548		11.4
61	62 (154)*				0.74795	N/A
62	63 (82)	0.80854	0.80435	0.92210		7.91
63	64 (151)	0.69859	0.77696	0.75194		5.39
64	65 (124,135)	1.18436	1.15365	1.10157		3.65
65	66 (144)	0.46997	0.44515	0.44728		3.03
66	67 (107,109,147)	0.53007	0.59399	0.60092		6.79
67	68 (123)*				0.75721	N/A
68	69 (106,118,139,149)	0.73345	0.87010	0.91065		11.1
69	70 (140)*				0.81617	N/A
70	71 (114,134,143)	0.79881	0.69286	0.67134		9.46
71	72 (122,131,133,142)	1.05467	1.10365	1.21482		7.30
72	73 (146,165,188)	0.77338	0.74836	0.84851		6.60
73	74 (105,132,161)	1.05376	1.12667	1.09001		3.34
74	75 (153)	0.86842	1.03850	1.06451		10.8
75	76 (127,168,184)*				0.64655	N/A
76	77 (141)	0.60452	0.63916	0.56596		6.07
77	78 (179)	0.71594	0.79553	0.82721		7.35
78	79 (137)	0.28921	0.33330	0.37882		13.4
79	80 (130,176)	1.50600	1.48899	1.73451		8.70
80	81 **					**
81	82 (138,163,164)	0.86057	0.97953	0.98015		7.33
82	83 (158,160,186)	0.96156	0.91740	1.02119		5.39
83	84 (126,129)	3.02729	2.81965	2.86788		3.74
84	85 (166,178)	0.47828	0.46097	0.43324		4.97
85	86 **					**
86	87 (175,159)	0.48882	0.42845	0.47475		6.81
87	88 (182,187)	0.81807	0.95553	0.98900		9.84
88	89 (128,162)	1.22907	1.22205	1.27093		2.13
89	90 (183)	0.84701	0.89965	1.00533		8.79
90	91 (167)	0.63244	0.70415	0.65569		5.51
91	92 (185)	1.24334	1.21824	1.13498		4.73
92	93 (174,181)	0.81793	0.93214	0.95125		8.01

PCB STANDARD EVALUATION SUMMARY
THREE POINT INITIAL CALIBRATION CHECK FOR LINEARITY

	DB-1 PEAK NUMBER (IUPAC Congener Number)	RRF STD A CS0222B	RRF STD B CS0222C	RRF STD C CS0222D	RRF STD D SCS0222	% RSD (< 20%)
93	94 (177)	0.75682	0.78218	0.68315		6.95
94	95 (156,171)	0.82247	0.77090	0.71856		6.74
95	96 (157,202)	5.74911	5.64512	5.50993		2.13
96	97 **					**
97	98 (173)	1.14817	1.04452	1.13947		5.18
98	99 (201)	0.77539	0.70789	0.66887		7.51
99	100 (172,204)	0.70666	0.66601	0.64435		4.70
100	101 (192,197)	0.69439	0.57289	0.57592		11.3
101	102 (180)	0.90090	1.07432	1.10591		10.7
102	103 (193)	0.74501	0.67521	0.64895		7.20
103	104 (191)	0.67875	0.64063	0.72364		6.10
104	105 (200,169)	0.86592	0.82524	0.82689		2.74
105	106 (170)	1.50556	1.59284	1.77012		8.31
106	107 (190)	1.25231	1.16608	1.23789		3.79
107	108 (198)	1.11587	1.11999	1.08282		1.84
108	109 (199)	0.56194	0.61375	0.56994		4.79
109	110 (196,203)	0.61553	0.68149	0.61836		5.84
110	111 (189)	1.04975	1.17492	1.22129		7.73
111	112 (195)	1.70125	1.73800	1.45485		9.44
112	113 (208)	0.55743	0.59984	0.61475		5.03
113	114 (207)	1.15093	1.16468	1.19848		2.09
114	115 (194)	1.37224	1.44694	1.36265		3.31
115	116 (205)	0.96593	1.07212	1.07089		5.88
116	117 (206)	1.28240	1.25055	1.23288		2.00
117	118 (209)	1.49555	1.53298	1.59618		3.30

* - A separate PCB congener calibration standard is analyzed for these PCB congeners that do not exist at measurable levels in the Green Bay multi-Aroclor calibration. These so-called non-Aroclor PCB congeners (with the exception of peak 3(2)) are analyzed to provide accurate retention time information and relative response factors in the event they need to be quantified.

* *- Refinements in the elution position of several congeners has occurred due to the availability of individual congener standards. The following low level congeners that were designated as separately eluting peaks have been determined to co-elute with another congener. The DB-1 peak numbers are no longer required for these congeners, but the original DB-1 numbering system has remained intact for all other peaks.

PK 18 (23) now elutes in PK 19 (23,34,54)
PK 40 (68) now elutes in PK 41 (68,96)
PK 81 (176) now elutes in PK 80 (130,176)
PK 86 (166) now elutes in PK 85 (166,178)
PK 97 (157) now elutes in PK 96 (157,202)

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Retention Time Study

PCB RETENTION TIME SUMMARY
INITIAL 72-HOUR RETENTION TIME WINDOW MEASUREMENT

LAB NAME: NORTHEAST ANALYTICAL, INC.

LAB CODE: NYS ELAP #11078

INSTRUMENT ID: GC #17

GC COLUMN ID: J&W, DB-1, 30 METER

	FILENAME	DATE	TIME
STANDARD #1:	CCCS0222B	02/23/07	17:02
STANDARD #2:	CCCS0227A	02/27/07	19:14
STANDARD #3:	CCCS0301A	03/01/07	15:58
STANDARD #4:	SC0222	02/22/07	23:12
STANDARD #5:	SC0227	02/27/07	17:00
STANDARD #6:	SC0301	03/01/07	13:44

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PCB RETENTION TIME SUMMARY
INITIAL 72-HOUR RETENTION TIME WINDOW MEASUREMENT

DB-1 PEAK NUMBER (IUPAC Congener Number)	MEAN R.T.	+/- 3SD	R.T. WINDOW FROM	TO
2 (1)	11.74	0.07	11.67	11.81
3 (2)*	12.77	0.07	12.70	12.84
4 (3)	12.87	0.07	12.80	12.94
5 (4,10)	13.48	0.07	13.41	13.55
6 (7,9)	14.35	0.07	14.28	14.42
7 (6)	14.65	0.07	14.58	14.72
8 (5,8)	14.84	0.07	14.77	14.91
9 (14)*	15.40	0.07	15.33	15.47
10 (19)	15.49	0.07	15.42	15.56
11 (30)*	15.95	0.07	15.88	16.02
12 (11)*	16.01	0.07	15.94	16.08
13 (12,13)	16.21	0.07	16.14	16.28
14 (15,18)	16.34	0.07	16.27	16.41
15 (17)	16.43	0.07	16.36	16.50
16 (24,27)	16.73	0.07	16.66	16.80
17 (16,32)	16.98	0.07	16.91	17.05
18 **				
19 (23,34,54)*	17.44	0.07	17.37	17.51
20 (29)	17.63	0.07	17.56	17.70
21 (26)	17.75	0.07	17.68	17.82
22 (25)	17.83	0.07	17.76	17.90
23 (31)	18.03	0.07	17.96	18.10
24 (28,50)	18.08	0.07	18.01	18.15
25 (20,21,33,53)	18.43	0.07	18.36	18.50
26 (22,51)	18.66	0.07	18.59	18.73
27 (45)	18.89	0.07	18.82	18.96
28 (36)*	19.03	0.07	18.96	19.10
29 (46)	19.17	0.07	19.10	19.24
30 (39)*	19.29	0.07	19.22	19.36
31 (52,69,73)	19.46	0.07	19.39	19.53
32 (43,49)	19.63	0.07	19.56	19.70
33 (38,47)	19.75	0.07	19.68	19.82
34 (48,75)	19.81	0.07	19.74	19.88
35 (62,65)*	19.95	0.07	19.88	20.02
36 (35)*	20.03	0.07	19.96	20.10
37 (104,44)	20.20	0.07	20.13	20.27
38 (37,42,59)	20.33	0.07	20.26	20.40
39 (41,64,71,72)	20.68	0.07	20.61	20.75
40 **				
41 (68,96)*	20.84	0.07	20.77	20.91
42 (40)	20.94	0.07	20.87	21.01
43 (57,103)*	21.20	0.07	21.13	21.27
44 (58,67,100)	21.36	0.07	21.29	21.43
45 (63)	21.52	0.07	21.45	21.59
46 (74,94,61)	21.69	0.07	21.62	21.76
47 (70)	21.82	0.07	21.75	21.89

PCB RETENTION TIME SUMMARY
INITIAL 72-HOUR RETENTION TIME WINDOW MEASUREMENT

DB-1 PEAK NUMBER (IUPAC Congener Number)	MEAN R.T.	+/- 3SD	R.T. WINDOW FROM	TO
48 (66,76,98,80,93,95,102,88)	21.94	0.07	21.87	22.01
49 (55,91,121)	22.24	0.07	22.17	22.31
50 (56,60)	22.55	0.07	22.48	22.62
51 (84,92,155)	22.78	0.07	22.71	22.85
52 (89)	22.89	0.07	22.82	22.96
53 (90,101)	23.05	0.07	22.98	23.12
54 (79,99,113)	23.24	0.07	23.17	23.31
55 (119,150)	23.51	0.07	23.44	23.58
56 (78,83,112,108)	23.61	0.07	23.54	23.68
57 (97,152,86)	23.83	0.07	23.76	23.90
58 (81,87,117,125,115,145)	24.00	0.07	23.93	24.07
59 (116,85,111)	24.15	0.07	24.08	24.22
60 (120,136)	24.27	0.07	24.20	24.34
61 (77,110,148)	24.40	0.07	24.33	24.47
62 (154)*	24.69	0.07	24.62	24.76
63 (82)	24.77	0.07	24.70	24.84
64 (151)	25.07	0.07	25.00	25.14
65 (124,135)	25.20	0.07	25.13	25.27
66 (144)	25.27	0.07	25.20	25.34
67 (107,109,147)	25.33	0.07	25.26	25.40
68 (123)*	25.42	0.07	25.35	25.49
69 (106,118,139,149)	25.52	0.07	25.45	25.59
70 (140)*	25.63	0.07	25.56	25.70
71 (114,134,143)	25.93	0.07	25.86	26.00
72 (122,131,133,142)	26.13	0.07	26.06	26.20
73 (146,165,188)	26.41	0.07	26.34	26.48
74 (105,132,161)	26.54	0.07	26.47	26.61
75 (153)	26.70	0.07	26.63	26.77
76 (127,168,184)*	26.81	0.07	26.74	26.88
77 (141)	27.23	0.07	27.16	27.30
78 (179)	27.30	0.07	27.23	27.37
79 (137)	27.51	0.07	27.44	27.58
80 (130,176)	27.66	0.07	27.59	27.73
81 **				
82 (138,163,164)	27.89	0.07	27.82	27.96
83 (158,160,186)	28.07	0.07	28.00	28.14
84 (126,129)	28.27	0.07	28.20	28.34
85 (166,178)	28.62	0.07	28.55	28.69
86 **				
87 (175,159)	28.92	0.07	28.85	28.99
88 (182,187)	29.07	0.07	29.00	29.14
89 (128,162)	29.19	0.07	29.12	29.26
90 (183)	29.38	0.07	29.31	29.45
91 (167)	29.64	0.07	29.57	29.71
92 (185)	29.99	0.07	29.92	30.06
93 (174,181)	30.36	0.07	30.29	30.43

PCB RETENTION TIME SUMMARY
INITIAL 72-HOUR RETENTION TIME WINDOW MEASUREMENT

DB-1 PEAK NUMBER (IUPAC Congener Number)	MEAN R.T.	+/- 3SD	R.T. WINDOW FROM	TO
94 (177)	30.63	0.07	30.56	30.70
95 (156,171)	30.93	0.07	30.86	31.00
96 (157,202)	31.19	0.07	31.12	31.26
97 **				
98 (173)	31.36	0.07	31.29	31.43
99 (201)	31.75	0.07	31.68	31.82
100 (172,204)	31.98	0.07	31.91	32.05
101 (192,197)	32.29	0.07	32.22	32.36
102 (180)	32.47	0.07	32.40	32.54
103 (193)	32.71	0.07	32.64	32.78
104 (191)	33.02	0.07	32.95	33.09
105 (200,169)	33.37	0.07	33.30	33.44
106 (170)	34.53	0.07	34.46	34.60
107 (190)	34.80	0.07	34.73	34.87
108 (198)	35.67	0.07	35.60	35.74
109 (199)	35.91	0.07	35.84	35.98
110 (196,203)	36.45	0.07	36.38	36.52
111 (189)	37.60	0.07	37.53	37.67
112 (195)	39.18	0.07	39.11	39.25
113 (208)	39.71	0.07	39.64	39.78
114 (207)	40.65	0.07	40.58	40.72
115 (194)	42.07	0.07	42.00	42.14
116 (205)	42.96	0.08	42.89	43.04
117 (206)	48.13	0.07	48.06	48.20
118 (209)	54.18	0.07	54.11	54.25

* - A separate PCB congener calibration standard is analyzed for these PCB congeners that do not exist at measurable levels in the Green Bay multi-Aroclor calibration. These so-called non-Aroclor PCB congeners (with the exception of peak 3(2)) are analyzed to provide accurate retention time information and relative response factors in the event they need to be quantified.

**- Refinements in the elution position of several congeners has occurred due to the availability of individual congener standards. The following low level congeners that were designated as separately eluting peaks have been determined to co-elute with another congener. The DB-1 peak numbers are no longer required for these congeners, but the original DB-1 numbering system has remained intact for all other peaks.

PK 18(23) now elutes in PK 19(23,34,54)
PK 40(68) now elutes in PK 41(68,96)
PK 81(176) now elutes in PK 80(130,176)
PK 86(166) now elutes in PK 85(166,178)
PK 97(157) now elutes in PK 96(157,202)

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Appendix E

Method Detection Limit Study

TABLE 1

Northeast Analytical Inc.

Quality Assurance/Quality Control

March 16, 2006

Revised:

Method Detection Limit Study

Method(s): Congener Specific Green Bay Method SOP: NE013_07.sop
Compound: Green Bay PCB Standard at various levels: 0.0784 ug/g, 0.784 ug/g
Matrix: Solid Sodium Sulfate - 10grams
Extraction: ASE (EPA 3545) SOP: NE143_03.sop
Date Extracted: 01/16/2006 & 01/18/2006
Date Analyzed: 02/17/2006 - 02/18/2006

Analysis: Internal Standard by Area
Instrument: GC-17 Agilent 6890
Column: DB-1; 30 meter x 0.25 mm x 0.25 µm
Detector: mirco-ECD

Peak Number (IUPAC Number)	MDL Concentration (ng/g)	MDL Recovery Sample #1 (ng/g)	MDL Recovery Sample #2 (ng/g)	MDL Recovery Sample #3 (ng/g)	MDL Recovery Sample #4 (ng/g)	MDL Recovery Sample #5 (ng/g)	MDL Recovery Sample #6 (ng/g)	MDL Recovery Sample #7 (ng/g)	MDL Recovery Sample #8 (ng/g)	Average MDL Recovery (ng/g)	Standard Deviation (ng/g)	%RSD (%)	Method Detection Limit (ng/g)	Method Detection Limit (mg/Kg)
2 (1)	5.48	4.64	5.42	5.55	5.44	5.13	5.39	5.38	5.00	5.25	0.302	5.75	0.905	0.000905
3 (2)	Supp. Congener													
4 (3)	3.20	3.05	3.09	3.12	2.99	2.97	3.11	3.14	3.38	3.11	0.127	4.09	0.381	0.000381
5 (4,10)	1.55	1.50	1.53	1.53	1.55	1.52	1.51	1.71	1.71	1.57	0.0867	5.52	0.260	0.000260
6 (7,9)	0.548	0.584	0.516	0.513	0.538	0.528	0.514	0.464	0.491	0.519	0.0350	6.76	0.105	0.000105
7 (6)	0.868	0.859	0.832	0.757	0.879	0.854	0.844	0.830	0.847	0.838	0.0360	4.30	0.108	0.000108
8 (5,8)	6.40	6.45	6.29	6.43	6.26	6.28	6.28	6.32	5.57	6.23	0.279	4.47	0.836	0.000836
9 (14)	Supp. Congener													
10 (19)	1.28	1.30	1.22	1.22	1.23	1.35	1.21	1.35	1.13	1.25	0.0743	5.94	0.223	0.000223
11 (30)	Supp. Congener													
12 (11)	Supp. Congener													
13 (12,13)	1.22	1.21	1.14	1.14	1.18	1.21	1.18	1.29	1.21	1.20	0.0483	4.04	0.145	0.000145
14 (15,18)	1.69	1.65	1.82	1.65	1.64	1.67	1.41	1.65	1.66	1.64	0.111	6.75	0.333	0.000333
15 (17)	1.69	1.59	1.67	1.62	1.65	1.66	1.47	1.63	1.58	1.61	0.0640	3.98	0.192	0.000192
16 (24,27)	0.119	0.118	0.114	0.107	0.107	0.111	0.126	0.106	0.121	0.114	0.00721	6.34	0.0216	0.0000216
17 (16,32)	1.78	1.75	1.75	1.72	1.88	1.73	1.63	1.69	1.70	1.73	0.0719	4.16	0.216	0.000216
19 (23,34,54)	Supp. Congener													
20 (29)	0.243	0.213	0.247	0.244	0.246	0.245	0.231	0.248	0.266	0.243	0.0153	6.29	0.0458	0.0000458
21 (26)	0.329	0.303	0.353	0.349	0.303	0.339	0.383	0.332	0.329	0.336	0.0263	7.82	0.0789	0.0000789
22 (25)	0.146	0.143	0.139	0.147	0.160	0.137	0.149	0.139	0.151	0.146	0.00763	5.24	0.0229	0.0000229
23 (31)	1.88	1.75	1.95	1.80	1.84	1.95	1.64	1.84	1.86	1.83	0.102	5.59	0.307	0.000307
24 (28,50)	2.41	2.59	2.42	2.40	2.31	2.72	2.22	2.37	2.45	2.43	0.156	6.39	0.467	0.000467
25 (20,21,33,53)	1.81	1.87	1.61	1.87	1.83	1.85	1.81	2.01	1.87	1.84	0.111	6.03	0.332	0.000332
26 (22,51)	1.32	1.33	1.51	1.34	1.27	1.33	1.37	1.38	1.41	1.37	0.0718	5.25	0.215	0.000215
27 (45)	0.407	0.432	0.401	0.410	0.442	0.425	0.453	0.415	0.466	0.430	0.0223	5.18	0.0668	0.0000668
28 (36)	Supp. Congener													
29 (46)	1.83	1.74	1.77	1.84	1.84	1.72	1.91	1.85	1.76	1.80	0.065	3.59	0.194	0.000194
30 (39)	Supp. Congener													
31 (52,69,73)	2.18	2.42	2.36	2.48	2.38	2.20	2.19	2.12	2.50	2.33	0.144	6.20	0.433	0.000433
32 (43,49)	1.05	1.07	0.940	0.993	0.999	1.05	1.16	1.06	1.10	1.05	0.0690	6.59	0.207	0.000207
33 (38,47)	0.457	0.511	0.463	0.465	0.441	0.503	0.498	0.458	0.482	0.477	0.0248	5.19	0.0743	0.0000743
34 (48,75)	0.457	0.486	0.481	0.447	0.432	0.473	0.436	0.473	0.452	0.460	0.0208	4.51	0.0622	0.0000622
35 (62,65)	Supp. Congener													
36 (35)	Supp. Congener													
37 (104,44)	1.96	2.00	1.71	1.89	1.83	1.89	1.83	1.88	1.89	1.87	0.0799	4.28	0.240	0.000240
38 (37,42,59)	1.19	1.03	1.15	1.13	0.972	1.16	1.13	1.15	1.00	1.09	0.0765	7.02	0.229	0.000229
39 (41,64,71,72)	1.87	1.91	1.76	1.96	1.90	1.92	1.82	1.91	1.96	1.89	0.0695	3.67	0.208	0.000208
41 (68,96)	Supp. Congener													
42 (40)	0.429	0.396	0.460	0.452	0.435	0.458	0.482	0.459	0.419	0.445	0.0271	6.09	0.0813	0.0000813
43 (57,103)	Supp. Congener													
44 (58,67,100)	0.502	0.516	0.545	0.540	0.529	0.525	0.516	0.511	0.465	0.518	0.0244	4.71	0.0733	0.0000733
45 (63)	0.959	0.946	0.875	0.955	0.951	0.940	0.922	0.962	0.877	0.93	0.0346	3.72	0.104	0.000104
46 (74,94,61)	0.868	0.901	0.851	0.897	0.848	0.822	0.795	0.870	0.866	0.856	0.0358	4.18	0.107	0.000107
47 (70)	1.55	1.55	1.54	1.45	1.51	1.38	1.43	1.58	1.56	1.50	0.0731	4.87	0.219	0.000219
48 (66,76,98,80,93,95,102,88)	3.29	3.16	3.26	3.35	3.20	3.36	3.28	3.53	3.17	3.29	0.124	3.76	0.371	0.000371
49 (55,91,121)	0.233	0.213	0.235	0.206	0.238	0.216	0.217	0.218	0.234	0.222	0.0121	5.43	0.0362	0.0000362
50 (56,60)	1.60	1.45	1.57	1.54	1.64	1.56	1.58	1.52	1.62	1.56	0.0604	3.87	0.181	0.000181
51 (84,92,155)	0.822	0.865	0.866	0.824	0.882	0.859	0.859	0.867	0.930	0.869	0.0295	3.40	0.0886	0.0000886
52 (89)	0.457	0.425	0.429	0.450	0.471	0.448	0.455	0.474	0.454	0.451	0.0174	3.87	0.0522	0.0000522

53 (90,101)	0.822	0.880	0.896	0.821	0.837	0.866	0.808	0.836	0.829	0.847	0.0306	3.61	0.0916	0.0000916
54 (79,99,113)	0.338	0.322	0.312	0.328	0.308	0.345	0.287	0.350	0.322	0.322	0.0203	6.31	0.0609	0.0000609
55 (119,150)	0.128	0.120	0.126	0.127	0.129	0.136	0.131	0.136	0.135	0.130	0.00562	4.33	0.0169	0.0000169
56 (78,83,112,108)	0.685	0.665	0.661	0.628	0.654	0.638	0.701	0.650	0.693	0.661	0.0253	3.82	0.076	0.0000757
57 (97,152,86)	0.256	0.259	0.254	0.254	0.247	0.255	0.255	0.261	0.223	0.251	0.0119	4.75	0.0357	0.0000357
58 (81,87,117,125,115,145)	0.530	0.523	0.552	0.565	0.602	0.535	0.535	0.526	0.530	0.546	0.0265	4.85	0.0794	0.0000794
59 (111,116,85)	0.320	0.324	0.313	0.320	0.306	0.348	0.337	0.330	0.332	0.326	0.0133	4.08	0.0399	0.0000399
60 (120,136)	0.343	0.322	0.341	0.345	0.381	0.321	0.323	0.376	0.329	0.342	0.0239	6.99	0.0717	0.0000717
61 (77,110,148)	0.973	0.927	1.00	0.921	0.965	0.963	0.934	1.09	0.932	0.967	0.0565	5.85	0.169	0.000169
62 (154)	Supp. Congener													
63 (82)	0.201	0.205	0.229	0.206	0.228	0.200	0.215	0.201	0.197	0.210	0.0127	6.06	0.0382	0.0000382
64 (151)	0.777	0.729	0.773	0.735	0.821	0.773	0.780	0.772	0.772	0.769	0.0285	3.70	0.0853	0.0000853
65 (124,135)	0.133	0.127	0.130	0.149	0.127	0.133	0.148	0.126	0.124	0.133	0.00992	7.46	0.0297	0.0000297
66 (144)	0.274	0.272	0.273	0.259	0.290	0.292	0.264	0.267	0.273	0.274	0.0117	4.27	0.0351	0.0000351
67 (107,109,147)	0.0594	0.0578	0.0579	0.0551	0.0571	0.0606	0.0489	0.0545	0.0548	0.0558	0.00347	6.22	0.01041	0.00001041
68 (123)	Supp. Congener													
69 (106,118,139,149)	1.83	1.70	1.90	1.97	1.88	1.89	1.81	1.79	1.86	1.85	0.0825	4.46	0.247	0.000247
70 (140)	Supp. Congener													
71 (114,134,143)	0.922	0.963	0.973	0.945	0.913	0.937	0.998	0.899	0.985	0.952	0.0348	3.65	0.104	0.000104
72 (122,131,133,142)	0.133	0.140	0.133	0.147	0.136	0.149	0.144	0.136	0.133	0.140	0.00630	4.51	0.0189	0.0000189
73 (146,165,188)	0.178	0.172	0.183	0.178	0.178	0.169	0.165	0.190	0.171	0.176	0.00818	4.65	0.0245	0.0000245
74 (105,132,161)	0.619	0.658	0.617	0.599	0.624	0.647	0.684	0.616	0.640	0.636	0.0272	4.28	0.0815	0.0000815
75 (153)	1.35	1.31	1.35	1.37	1.34	1.40	1.30	1.26	1.32	1.332	0.0460	3.45	0.138	0.000138
76 (127,168,184)	Supp. Congener													
77 (141)	0.777	0.721	0.791	0.796	0.776	0.778	0.738	0.732	0.747	0.760	0.0288	3.79	0.0864	0.0000864
78 (179)	0.667	0.666	0.634	0.691	0.671	0.690	0.694	0.657	0.702	0.676	0.0228	3.38	0.0685	0.0000685
79 (137)	0.342	0.381	0.331	0.339	0.336	0.331	0.342	0.372	0.384	0.352	0.0229	6.51	0.0686	0.0000686
80 (130,176)	0.119	0.122	#REF!	0.116	0.117	0.111	0.129	0.124	0.105	0.118	0.00751	6.36	0.0225	0.0000225
82 (138,163,164)	1.23	1.27	0.119	1.22	1.29	1.23	1.15	1.42	1.24	1.26	0.0755	5.99	0.226	0.000226
83 (158,160,186)	0.114	0.115	1.26	0.121	0.111	0.122	0.124	0.121	0.114	0.121	0.00867	7.18	0.0260	0.0000260
84 (126,129)	0.0591	0.0568	0.0592	0.0517	0.0558	0.0539	0.0550	0.0511	0.0560	0.0549	0.00267	4.86	0.00801	0.00000801
85 (166,178)	0.502	0.498	0.553	0.502	0.544	0.517	0.491	0.531	0.509	0.518	0.0225	4.34	0.0675	0.0000675
87 (175,159)	0.914	0.858	0.986	0.938	0.913	0.963	0.849	0.954	0.982	0.930	0.0530	5.69	0.159	0.000159
88 (182,187)	1.64	1.62	1.70	1.68	1.69	1.50	1.66	1.66	1.68	1.65	0.0644	3.90	0.193	0.000193
89 (128,162)	0.0457	0.0472	0.0467	0.0451	0.0462	0.0470	0.0474	0.0461	0.0505	0.0470	0.00159	3.39	0.00478	0.00000478
90 (183)	0.777	0.769	0.775	0.846	0.782	0.784	0.750	0.761	0.785	0.782	0.0287	3.67	0.0859	0.0000859
91 (167)	0.224	0.232	0.215	0.232	0.243	0.234	0.244	0.252	0.224	0.234	0.0119	5.07	0.0356	0.0000356
92 (185)	0.215	0.263	0.233	0.206	0.207	0.220	0.229	0.220	0.217	0.224	0.0180	8.02	0.0540	0.0000540
93 (174,181)	1.46	1.43	1.49	1.45	1.45	1.59	1.49	1.51	1.45	1.48	0.0515	3.47	0.154	0.000154
94 (177)	0.777	0.788	0.769	0.773	0.653	0.767	0.776	0.772	0.756	0.757	0.0429	5.67	0.129	0.000129
95 (156,171)	0.361	0.359	0.390	0.339	0.363	0.357	0.360	0.383	0.355	0.363	0.0163	4.49	0.0489	0.0000489
96 (157,202)	0.0302	0.0314	0.0304	0.0290	0.0314	0.0321	0.0285	0.0271	0.0317	0.0302	0.00181	6.00	0.00543	0.00000543
98 (173)	0.174	0.197	0.197	0.169	0.176	0.170	0.169	0.180	0.182	0.180	0.0116	6.47	0.0349	0.0000349
99 (201)	0.178	0.160	0.172	0.183	0.180	0.186	0.179	0.177	0.186	0.178	0.00869	4.88	0.0261	0.0000261
100 (172,204)	0.256	0.253	0.285	0.260	0.233	0.259	0.256	0.262	0.250	0.257	0.0143	5.55	0.0428	0.0000428
101 (192,197)	0.502	0.583	0.599	0.533	0.586	0.545	0.591	0.563	0.582	0.573	0.0232	4.06	0.0697	0.0000697
102 (180)	2.79	3.03	2.79	2.71	2.82	2.77	2.79	2.75	2.77	2.80	0.097	3.45	0.290	0.000290
103 (193)	0.192	0.194	0.198	0.181	0.203	0.190	0.200	0.188	0.189	0.193	0.00708	3.67	0.0212	0.0000212
104 (191)	0.548	0.531	0.526	0.535	0.593	0.511	0.533	0.526	0.544	0.537	0.0244	4.54	0.0732	0.0000732
105 (200,169)	0.196	0.207	0.198	0.190	0.197	0.190	0.191	0.195	0.209	0.197	0.00740	3.76	0.0222	0.0000222
106 (170)	0.585	0.661	0.578	0.599	0.577	0.568	0.595	0.594	0.586	0.595	0.0286	4.82	0.0859	0.0000859
107 (190)	0.192	0.197	0.204	0.195	0.193	0.213	0.187	0.193	0.192	0.197	0.00812	4.12	0.0243	0.0000243
108 (198)	0.548	0.535	0.547	0.578	0.563	0.594	0.546	0.540	0.559	0.558	0.0200	3.59	0.0600	0.0000600
109 (199)	1.92	1.92	1.98	2.00	2.02	1.84	1.88	2.08	1.90	1.95	0.0818	4.19	0.245	0.000245
110 (196,203)	1.96	1.77	1.98	1.92	1.87	1.99	1.92	1.93	1.91	1.91	0.0678	3.55	0.203	0.000203
111 (189)	0.182	0.162	0.188	0.183	0.164	0.171	0.190	0.182	0.188	0.178	0.0112	6.27	0.0336	0.0000336
112 (195)	0.253	0.268	0.259	0.274	0.241	0.240	0.257	0.243	0.263	0.256	0.0131	5.11	0.0392	0.0000392
113 (208)	1.13	1.17	1.02	1.09	1.12	1.22	1.23	1.09	1.16	1.14	0.0721	6.34	0.216	0.000216
114 (207)	0.425	0.362	0.372	0.350	0.353	0.381	0.356	0.390	0.360	0.365	0.0142	3.89	0.0426	0.0000426
115 (194)	0.822	0.750	0.851	0.823	0.828	0.850	0.824	0.841	0.858	0.828	0.0341	4.12	0.102	0.000102
116 (205)	0.502	0.514	0.586	0.507	0.545	0.511	0.555	0.571	0.497	0.536	0.0332	6.20	0.100	0.000100
I.S. (OCN)	internal standard													
117 (206)	0.311	0.346	0.317	0.322	0.304	0.323	0.305	0.305	0.306	0.316	0.0143	4.54	0.0430	0.0000430
118 (209)	0.0554	0.0568	0.0548	0.0574	0.0526	0.0541	0.0505	0.0476	0.0549	0.0536	0.00326	6.08	0.0098	0.0000098
												Total MDL =	12.90	0.0129

- Data from 0.0784 ug/g MDL Study

- Data from 0.784 ug/g MDL Study

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TABLE 2

Northeast Analytical Inc.

Quality Assurance/Quality Control

March 16, 2006

Revised:

Method Detection Limit Study

Method(s): Congener Specific Green Bay Method SOP: NE013_07.sop

Compound: Supplemental Congener Standard at various levels

Matrix: Solid - Sodium Sulfate 10 grams

Extraction: ASE (EPA 3545) SOP: NE143_03.sop

Date Extracted: 01/16/2006 & 01/18/2006

Date Analyzed: 02/17/2006 - 02/18/2006

Analysis: Internal Standard by Area

Instrument: GC-17 Agilent 6890

Column: DB-1; 30 meter x 0.25 mm x 0.25 µm

Detector: mirco-ECD

Peak Number (IUPAC Number)	MDL Concentration (ng/g)	MDL Recovery Sample #1 (ng/g)	MDL Recovery Sample #2 (ng/g)	MDL Recovery Sample #3 (ng/g)	MDL Recovery Sample #4 (ng/g)	MDL Recovery Sample #5 (ng/g)	MDL Recovery Sample #6 (ng/g)	MDL Recovery Sample #7 (ng/g)	MDL Recovery Sample #8 (ng/g)	Average MDL Recovery (ng/g)	Standard Deviation (ng/g)	%RSD (%)	Method Detection Limit (ng/g)	Method Detection Limit (mg/Kg)
3 (2)	200	211	204	202	199	217	210	220	208	209	7.19	3.44	21.6	0.0216
9 (14)	1.25	1.11	1.21	1.30	1.33	1.16	1.19	1.19	1.21	1.21	0.0717	5.92	0.215	0.000215
11 (30)	1.25	1.18	1.18	1.22	1.25	1.21	1.01	1.21	1.26	1.19	0.0760	6.39	0.228	0.000228
12 (11)	5.00	5.19	4.76	5.46	5.07	5.03	5.10	5.04	5.07	5.09	0.195	3.83	0.584	0.000584
19 (23,34,54)	1.25	1.24	1.21	1.28	1.28	1.20	1.13	1.26	1.29	1.24	0.0519	4.19	0.156	0.000156
28 (36)	1.25	1.20	1.18	1.25	1.16	1.18	1.19	1.31	1.16	1.20	0.0510	4.24	0.153	0.000153
30 (39)	1.25	1.14	1.22	1.16	1.23	1.28	1.19	1.24	1.14	1.20	0.0523	4.35	0.157	0.000157
35 (62,65)	1.25	1.26	1.23	1.42	1.31	1.33	1.27	1.32	1.24	1.30	0.0619	4.76	0.186	0.000186
36 (35)	1.25	1.23	1.26	1.13	1.27	1.20	1.25	1.22	1.26	1.23	0.0468	3.82	0.140	0.000140
41 (68,96)	1.25	1.37	1.23	1.34	1.10	1.27	1.26	1.28	1.30	1.27	0.0805	6.35	0.241	0.000241
43 (57,103)	1.25	1.30	1.22	1.23	1.26	1.37	1.25	1.33	1.21	1.27	0.0561	4.41	0.168	0.000168
62 (154)	5.00	4.80	4.90	5.13	5.04	4.97	4.84	4.65	5.16	4.94	0.174	3.52	0.52	0.00052
68 (123)	1.25	1.30	1.26	1.19	1.24	1.25	1.25	1.14	1.18	1.23	0.0520	4.23	0.156	0.000156
70 (140)	1.25	1.20	1.21	1.25	1.36	1.25	1.22	1.23	1.15	1.23	0.0611	4.95	0.183	0.000183
76 (127,168,184)	1.25	1.26	1.15	1.18	1.25	1.27	1.21	1.26	1.26	1.23	0.0445	3.61	0.133	0.000133

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STANDARD OPERATING PROCEDURE REVIEW

SOP Name	Review Number	Reviewers	Title	QAO Approval	Effective Date
NE013_09	01	Inga Hotaling Christina L. Braidwood Robert E. Wagner	GC Supervisor QAO Lab Director	Christina Braidwood	02/20/09
NE013_10	00	Inga Hotaling Christina L. Braidwood Robert E. Wagner	GC Supervisor QAO Lab Director	Christina Braidwood	03/24/11

Attachment H-10

Extraction and Analysis of Lipids from Fish and Biota Material

STANDARD OPERATING PROCEDURE

THE EXTRACTION OF FISH AND BIOTA MATERIAL

Reference Methods: EPA METHOD 3500A & 3600A

LOCAL SOP NUMBER:	NE158_05_01
EFFECTIVE DATE:	03/29/2011
SUPERSEDES:	NE158_05
SOP TEMPLATE NUMBER:	SOT-ALL-Q-006-rev.03

APPROVALS

Dan Pfalzer
Assistant General Manager — Date: 03/29/2011

Christina L. Braidwood
Quality Manager — Date: 03/29/2011

PERIODIC REVIEW

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

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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STANDARD OPERATIONG PROCEDURE

LABORATORY PROCEDURE NE158_05.DOC

REVISION 5 REVIEW 1 (03/29/11)

1.0 IDENTIFICATION OF TEST METHOD

1.1 This is the Standard Operating Procedure for the extraction and analysis of % lipids from fish and biota material.

1.2 The purpose of this SOP is to provide a method for extraction of lipids from fish tissue and other biota samples.

2.0 APPLICABLE MATRIX AND MATRICES

2.1 This test method is appropriate for fish and biota tissue. This method may be restricted to use by or under the supervision of a technician knowledgeable in the area of sample extraction and clean-up.

2.2 The technician should further be aware of the proper care and handling of Polychlorinated Biphenyls (PCBs) as well. The technician must have an understanding of the methods and requirements of USEPA-SW-846A "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition and SW-846 EPA Methods 3540, 3500, 2500A.

2.3 The extraction technician must also be certified to perform the procedure by an approved instructor by performing precision and accuracy requirement.

3.0 DETECTION LIMIT

3.1 The detection limit is defined by the precision of the balance used while weighing the sample. The detection limit for the Mettler Toledo PL303 balance is 0.001g. If there is a limited amount of oil to detect it may be necessary to utilize the Mettler Toledo AG204 balance, which has a detection limit of 0.0001g.

4.0 SCOPE AND APPLICATION

4.1 The following procedure is used by NEA for the extraction of fish tissue for lipid analysis, though it may in part be adapted for other biota extractions.

5.0 SUMMARY OF TEST METHOD

5.1 This method outlines the procedures used for the extraction of lipids from fish and biota material. Biota samples are extracted by a Soxhlet extractor apparatus over a defined length of time.

5.2 Once the extraction is complete, the solvent is exchanged with pure hexane utilizing a LV evaporation system.

5.3 After solvent exchange, the solvent extract is set to 25ml using a 25ml volumetric flask. Once set to volume, 10ml are withdrawn and placed in a properly labeled, pre-weighed aluminum dish.

5.4 The extracted solvent in the tin is evaporated utilizing a micro blowdown apparatus. Using the initial weight and the final weight of the aluminum dish, the % lipid concentration in the sample material can be calculated (see **Section 15.0**).

6.0 DEFINITIONS

6.1 **Method Blank (B)**- A method blank is processed with each batch of samples that are extracted to assess for contamination during prep and processing steps. The method blank is carried throughout all stages of sample preparation and extraction steps. Sodium sulfate is processed as the method blank.

6.2 **Lab Control Spike (LCS)**- A non-site sample which is prepared in the laboratory, to which a known amount of target analyte is added for assessment of laboratory performance. The laboratory control spike sample is composed of sodium sulfate with the spiked target analytes. The Laboratory Control Spike is processed with each batch of samples extracted.

6.3 **Lab Control Standard Duplicate (LCSD)**: A replicate of the Lab Control Standard to further assess analyte recovery efficiency**.**

6.4 **Matrix Spike (MS)**: A site sample to which a known amount of target analyte is added for assessment of analyte recovery efficiency.

6.5 **Matrix Spike Duplicate (MSD)**: A replicate of the Matrix Spike utilizing the same site sample and known amount of target analyte for assessment of analyte recovery efficiency**.**

6.6 **Relative Percent Difference (RPD**): A quality control measure designed to quantitate the difference in percentages for lipids/total solids, etc. of two identical samples.

7.0 INTERFERENCES

7.1 Laboratory technicians should exercise caution when completing percent lipid analysis. Technicians should be careful not to introduce laboratory contamination with extract when transferring sample to aluminum dish. For example, glass particles from pipettes may Give an incorrect value and compromise the integrity of the percent lipid analysis.

8.0 SAFETY

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

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

8.3 Safety glasses and gloves must be worn when handling glassware and samples.

9.0 EQUIPMENT AND SUPPLIES

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

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

9.3 Soxhlet Repetitive Flushing (reflux) Unit: 45/50 Pyrex #3740-M. (or equivalent)

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

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

9.6 Analytical Balance: Mettler PL-303 used to determine sample mass. (or equivalent)
9.7 Chiller: Pump driven water circulating cooling system cool flow #75 NESLABS Instruments, Inc. (or equivalent)
9.8 Turbo Vap Evaporator: Zymark #ZW640-3. (Or equivalent)

9.9 Turbo Vap Evaporator concentrator tubes: Zymark 250mL, 0.5mL endpoint. (or equivalent)

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

9.11 Vials: glass, 40mL vial & 15 mL (with Polyseal sealed cap) (20 mL & 10 mL) capacity, for sample extracts.

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

9.13 Pipettes: S/P Disposable Serological Borosilicate Pipettes.
1. 1mL X 1/10
2. 5mL X 1/10
3. 10mL X 1/10
4. Fisher Pasteur Borosilicate glass pipette 9" (or equivalent)

9.14 Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL.

9.15 Weighing Boats: Aluminum weighing boats (dish) for percent lipid weight determination.

9.16 Metal spatula.

9.17 4 oz Jars: Industrial Glassware

10.0 REAGENTS AND STANDARDS

10.1 Cellulose Extraction Thimble: Contains sample during Soxhlet extraction.

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

10.3 Boiling Chips: Chemware PTFE Boiling Stones P#0919120 (or equivalent)

10.4 Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208. (Or equivalent)

10.5 Acetone: High Purity Solvent Baxter (Burdick/Jackson) #UN1090. (Or equivalent)

10.6 1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT and STORAGE

11.1 All samples should remain frozen at all times unless being tested. Fish usually arrive whole-bodied or already filleted.

11.2 Once received, the sample must be ground and homogenized so that it may be analyzed. The homogenized fish tissue should be held for 6 to 12 months.

11.3 The fish solvent extracts should be held for 3 months. Some clients may request that the body and/or head of fish be saved once the fillets are cut out. Other biota material may have other specifications stated specifically for that project.

12.0 QUALITY CONTROL

12.1 The Method Reagent Blank (B) is used as a measure to ensure that there is no cross contamination across the sample set.

12.2 The Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are further controls which test the method to ensure proper lipid recovery is performed by calculating the RPD (see **Section 18.0**).

13.0 CALIBRATION AND STANDARDIZATION

13.1 The PL303 and the AG201 analytical balances should be calibrated daily to ensure proper measurements are maintained.

14.0 PROCEDURES

14.1 Sample Preparation

14.1.1 Throughout the entire process it should be noted that if the technician encounters any problems or difficulties with any samples or steps involved, all work should **STOP**! Any problems should be brought to the attention of the supervisor and documented in the LIMS extraction logbook.

14.1.2 The technician should first review the sample job folder and fill in the appropriate spaces on the internal sample tracking form and initial.

14.1.3 The fish samples are usually received as fillets and must be processed to produce a homogenous material prior to extraction. Once the sample has been processed, the sample is cut up into small enough pieces to fit into a standard manual meat grinder. An acetone rinsed Pyrex tray should be placed under the grinder exit to catch the minced tissue. After the fish has been ground once, it should be ground again to ensure complete homogenization of the sample.

14.1.4 The ground fish is placed into an appropriate-sized jar and labeled. The sample is then placed in the freezer for storage until the extraction process is begun.

14.2 Procedure: Sample Extraction

14.2.3 Rinse the cellulose extraction thimbles with hexane; and allow to dry out in a 4 oz. jar in a fume hood.

14.2.4 Fill a Pyrex pan with ice cubes and cold water about 1/2 inch deep. As the samples are weighed out; place the jars in the Pyrex pan to chill for at least 15 minutes prior to the drying step.

14.2.5 Into a tarred 4oz. jar, accurately weigh to the nearest 0.001g using an analytical balance about 9.5 grams of tissue. Record this weight in the laboratory extraction logbook of the LIMS system. Place the jar containing the sample into the Pyrex pan to chill. Repeat for remaining samples.

Note: All sample containers are to be returned to the appropriate refrigerator. For all empty sample containers, see the Chemical Hygiene Plan for proper disposal.

14.2.6 After the sample has been sufficiently chilled, add approximately 10 g of a 1:1 mix of magnesium sulfate/sodium sulfate to the sample, and mix well with a clean metal spatula. If the sample has not dried after a few minutes, another 10 g may be added. Once the sample is well dried and free flowing, transfer the sample to an extraction thimble. Repeat with remaining samples. Set empty mixing jar in a glass disposal bin and stir utensil aside for cleaning.

Note: Be careful not to add too much drying agent to the sample; if too much is added, the sample may not fit completely in the thimble. In this case the sample will have to be split into two different Soxhlet units.

14.2.7 Add 200 mL of a 1:1 mixture of hexane/acetone to a 250 mL round bottom flask. Add several boiling chips. Place a Soxhlet extractor on top of the round bottom flask. Label the round bottom flask with a sample number and use a pair of long-handled tweezers to place the corresponding thimble into the Soxhlet extractor. After all samples have been processed add the specified surrogate and matrix spikes required directly into thimble.

14.2.8 Rinse the inside and the outside connecting joints of the condenser units that will be used to condense the extraction solvent during the Soxhlet extraction

of the sample with hexane. Turn on chiller to cool the condensers.

14.2.9 Place the round bottom flask with attached Soxhlet extractor onto a heating mantle and attach condenser unit. Turn corresponding thermostats on to setting 5.5. Double check Soxhlets at this time for any cracks or chips which may leak solvent. Once the solvent begins to boil, a flushing action of once every two to three minutes should be achieved.

14.2.10 The samples should be extracted overnight for a minimum of **18** hours. Once the sample has finished extracting (usually in the morning), turn the heating mantle off and allow samples to cool to room temperature. Once cool, rinse the inside of the condenser with several pipette volumes of hexane. Disengage the Soxhlet and condenser unit and rinse the joint off as well into the Soxhlet.

14.2.11 Move Soxhlet units into a chemical fume hood, and flush the remaining solvent from the Soxhlet extractor by tipping the Soxhlet. Using a pair of long-handled tweezers, pull the thimbles out of the Soxhlets one at a time and allow them to drip dry by balancing the thimbles on the tops of the Soxhlets. Once dry, remove the thimbles to a sheet of aluminum foil for total solvent evaporation and disposal.

14.2.12 Rinse the Soxhlet with several pipette volumes of hexane and tip again to drain into the round bottom. Set aside the Soxhlet at this time. Procure the same number of Turbo tubes as there are samples. Using an individual Turbotube stand, label a Turbotube with the corresponding sample ID number and place in the holder. Pour the contents of the round bottom into the Turbotube, using a pipette and hexane to rinse the last drop out of the mouth of the round bottom flask. Rinse the round bottom flask with several pipette volumes of hexane, swirl gently, and decant into same turbo tube. Repeat this step twice for same sample, and then repeat all preceding steps for all other samples.

14.2.13 All glassware must be rinsed with technical grade acetone or a "for rinsing-only" labeled solvent, and dried in the hood before other cleaning steps.

14.3 Solvent Reduction: TurboVap Evaporator System

14.3.3 The Turbovap evaporator system is used in place of the Kuderna Danish (KD) concentrator apparatus.

14.3.4 The turbovap uses a heated water bath and positive pressure nitrogen flow/vortex action. The unit maintains a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract, which allows fractional reduction of the solvents without loss of higher boiling point analytes.

14.3.5 Turn the unit on (switch is located on the backside of the unit) and allow to heat up to the specified temperature for individual solvent use. This is indicated by the "Heating" display light, located above the temperate control knob on the right side of the unit. The system is at the proper temperature when the "At Temperature" light is lit. This is located above the "Heating" display light. The temperature of the unit should be set at 40 ± 2ºC.

14.3.6 As a precaution the TurboVap system regulators should be checked to assure that no residual gas pressure remains within the system and that the gas cylinder valve and gas pressure regulators are both off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty turbo tube into the water bath and close the lid. Make sure that the nitrogen gas cylinder valve is turned off and slowly turn on the gas pressure regulator. Bleed any residual gas until the regulator output pressure gauge reads "0" psi. Proceed to 8.3.4.

14.3.7 Place the Turbo tube containing the samples into the Turbovap and close the lid. Turn on the gas cylinder valve first and then begin slowly turning the pressure regulator on. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the reduction.

14.3.8 The process for solvent (hexane/acetone) reduction takes approximately 20-30 minutes. Do not leave the unit unattended as extracts may be blown to dryness.

14.3.9 Concentrate the solvent to approximately 10.0 mL. Remove the samples from the Turbovap and place in the rack. The remaining solvent will consist largely of hexane since the acetone component is fractionally removed at a faster rate than hexane; however, a solvent exchange with hexane should be completed by filling the Turbo tube up with approximately 50 mls of hexane and concentrating back down to 10.0 mls to ensure the acetone has been entirely removed.

NOTE: Not all samples will evaporate at the same rate; sample extracts containing large amounts of lipids or other non-volatile liquids may stop reducing before the 10.0 mL point is achieved. Samples that stop reducing should be removed as soon as possible.

14.3.10 Quantitatively transfer the sample extract with a disposable transfer pipette into an appropriate volumetric flask (25mL for biota extracts) with three 2mL hexane rinses. After the sample has been transferred, rinse the disposable transfer pipette with 0.5 mL of hexane into the volumetric flask. Add hexane to the volumetric meniscus mark. Invert the volumetric flask at least three times to mix completely. Decant the contents into a pre-labeled 40mL vial.

14.3.11 All dirty glassware must be rinsed with tech-acetone or a "For Rinsing-Only"

labeled solvent and dried in the fume hood before being washed.

14.4 Sample Extract concentration

NOTE: NO CLEAN-UP STEPS ARE PERFORMED FOR PERCENT LIPID DETERMINATION.

14.4.1 Weigh a pre-labeled aluminum dish for each sample extract. Record the weight in the percent lipid log book of the LIMS system.

14.4.2 Shake samples for a few seconds then using a 10 mL pipette, transfer 10 mL of the extract into the corresponding dish.

14.4.3 Concentrate the extract in the dish using the nitrogen micro-blowdown. Place a maximum of 3 dishes at one time on the nitrogen blow down. Set the temperature at 40C and the pressure at 40 PSI.

14.4.4 After concentration has been completed, place the dishes into the desiccators for at least 4 hours.

14.4.5 Weigh sample dishes and record the weight in the percent lipid logbook of the LIMS system. Calculate percent lipids as seen in **Section 15.0**.

15.0 CALCULATIONS

15.1 Calculate percent lipids as follows:

$$\text{PERCENT LIPIDS} = \frac{Vf - Ve \times 2.5 \times 100}{\text{Sample weight (g)}}$$

Vf = weight of tin tray with sample (g)
Ve = weight of empty tin tray (g)

15.2 Calculate the Practical Quantitation Limit (PQL):

$$\text{PQL} = \frac{0.001(\text{or } 0.0001)^*}{\text{sample weight}} \times 100$$

****Note: Depending on which balance was used to weigh the sample. The PL303 analytical balance would use 0.001 while the AG204 would use 0.0001.***

16.0 METHOD PERFORMANCE

16.1 Method Performance is measured through the use of laboratory duplicates (Matrix Spike/Matrix Spike Duplicates and Duplicate sample). The Laboratory Information Management System (LIMS) maintains records of laboratory duplicate analysis.

17.0 POLLUTION PREVENTION

17.1 Refer to NEA168.SOP for details about pollution prevention.

18.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

18.1 Laboratory Duplicate % RPD $\leq$ 20

18.2 Method Blank % lipid $\leq$ PQL

19.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

19.1 Ensure that analytical balances have been calibrated prior to use on each working day.

19.2 If data is still out of control, re-homogenize the sample and re-extract and re-calculate.

20.0 CONTINGINCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1 After all corrective actions are followed, with no change in the data or there is not enough sample material to perform corrective actions, report to client the data that is available, detailing how it is out of control.

21.0 WASTE MANAGEMENT

21.1 Refer to NE054.SOP, NE083.SOP, and NE089.SOP for waste management procedures.

22.0 REFERENCES

22.1 SW-846 methods 3500A & 3600A; United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Vol.1B, Cincinnati, OH 45268.

22.2 Guide to Environmental Analytical Methods, Genium Publishing Corporation, Schenectady, NY 12304.

23.0 TABLES, DIAGRAMS, FLOWCHARTS and VALIDATION DATA

23.0 No attachments.

STANDARD OPERATING PROCEDURE REVIEW

SOP Name	Review Number	Reviewers	Title	QAO Approval	Effective Date
NE158_05_01	01	Christina L. Braidwood Robert E. Wagner	QAO Lab Director	Christina Braidwood	03/29/11

Attachment H-11

ANSP Protocols for Fish Preservation, Fixation, and Curation

FISH PRESERVATION, FIXATION AND CURATION

4.1 Field preservation.

Specimens should be preserved as soon as practical after collection. Prior to preservation, specimen deterioration may be impeded by maintaining the specimen alive or by keeping the specimen cool (e.g., on ice or dry ice). The method of preservation should be determined by the goals of the study as defined by the project plan and the nature of the specimen. Unless otherwise defined by the project plan, one of the following methods of preservation should be used:

1) Formalin. Normally used where identification, size analysis or stomach analysis is a primary use of the specimen. Formalin should be diluted to around 5% for larval fishes, and at least 10% for larger fishes (the strength depending on size and condition of the specimen). Calcium carbonate may be used to buffer the formalin.

2) Ethanol. Normally used where otolith analysis or DNA analysis is a primary use of the specimen. Ethanol should be at least 70% strength.

3) Freezing. Normally used where tissue analysis is the primary use of the specimen. A more detailed description of this technique is provided in part 4.2 below.

Samples preserved in liquid should be preserved with sufficient preservative to allow free circulation of liquid throughout the sample container and to avoid deformation of the specimen. Packing specimens to allow at least 50% of the sample volume as preservative will normally be sufficient for these goals. Large specimens should be slit or injected with preservative. Specimens may be wrapped (e.g., in cheesecloth) to minimize rubbing. Samples should be cleaned of debris as much as practical. Samples preserved in liquid should contain an inner tag (pencil on high grade or plastic paper) noting at least serial or collection number, date and locality of the collection. Specimens with different serial or collection numbers may be placed in the same container if they are kept separate (e.g., by mesh bags) with individual tags or if tags are affixed to each specimen. The tag number of these individual fish should be recorded on the data sheet. Collection information may also be written on the exterior of the collecting container using pencil or "grease" pencil. However, the inner tag is considered the primary sample identifier.

4.2 Handling of Fish to be Frozen for Contaminant Analyses

Samples preserved by freezing should be wrapped in a material (as described in parts 4.2 a and b) which will not interfere with subsequent analysis. Target species will be collected by the methods outlined in the project protocol. For example, fish may be collected by backpack electrofishing, boat electrofishing, gill netting, seining, or other methods. Upon removal of fish from the water body, live specimens will be placed into a holding tub of water or in a cooler of wet ice. The coolers and tub will be cleaned with Alconox prior to the field trips. In the field, the tub and coolers will be cleaned and rinsed with the lake or river water prior to the fish being placed in them. Any visible material such as dirt or fish mucus should be cleaned with lake water between stations. If target specimens are dead at the time of collection (i.e., some gill netted fish), the fish will be placed into the cooler of ice. When either the appropriate fish are collected or the tub and/or the cooler becomes crowded with fish, biological data collection will follow. Fish will usually be measured in the field for total length in cm, although specific field data to be collected will depend on the project protocol.

Fish that are to be retained for chemical analysis will be wrapped (see parts 4.2 a and b below), labeled, and placed in a cooler of dry ice (unless field filleting is required in the project protocol, see SOP P-14-12 on preparation of fish samples for contaminant analysis, section 1.6). An inner tag (pencil on high grade or plastic paper enclosed in a zip-loc bag) will be placed in the pack without contacting the fish. The inner tag will contain at least sample number, project name, locality, date, species, and number of individuals. Fish will be thawed prior to filleting.

4.2a) Field Handling and Wrapping of Fish for Metals Analysis

Fish to be used for metals analysis will be wrapped in plastic (usually ziploc bags, whirl-paks, or trash bags). A tag (using pencil on high grade or plastic paper enclosed within a plastic bag) with at least the date, locality, species, and sample identification number will be placed inside the plastic bag, but without contacting the fish. Plastic bags may contain single specimens or multiple specimens from the same site. Larger individuals, such as channel catfish or carp, may have to be placed into heavy-duty plastic trash bags. "Grease" pencil on the bag and/or tags tied to the neck of the bag, should be used to externally identify the samples. However, the information from the inner tag takes precedence. Upon completion of the data collection and wrapping, fish will be placed into coolers of dry ice. Chains-of-custody for transporting the coolers to the Academy (or other testing locality) must be completed prior to leaving the field sites. Upon return to the Academy, the fish samples are to be removed from the coolers and placed into the ANSP Fisheries Section freezer. The samples should be recorded in the Fisheries Section's log book for frozen specimens.

4.2b) Fish Wrapping in Foil for Organics Analysis

Fish handling in field tubs and coolers will be the same as with fish used for metals analysis, but wrapping material will differ as follows. After appropriate data are collected, fish will be wrapped in heavy duty aluminum foil and secured with "freezer" tape. An inner tag (pencil on high grade paper) will be placed in the package, but without contacting the fish. Single specimens can be wrapped in a package or several fish of the same species and site can be wrapped together. Multiple species from a sample can be placed together in a single pack when field identifications are difficult. Cleaning the foil with solvents (hexane and/or methanol) may be required on some projects, and such methods will be stated in the project protocol. A "grease" pencil will be used to label the "freezer" tape on the outside of the pack. At least locality, date, sample identification number, number of specimens, and species will be recorded on the "freezer" tape, although the inside tag takes precedence. Upon completion of wrapping, the samples will be placed into a cooler of dry ice for transport to the Academy (or to another testing facility). Prior to departing from the field trip, a chain of custody record will be completed denoting the number of coolers, and number of fish packs that will transported or delivered. The samples should be recorded in the Fisheries Section's log book for frozen specimens.

4.3 Transportation

Samples should be transported in a manner which will prevent specimen deterioration. Samples preserved in liquid may be drained for short periods of travel; if draining is done, the same kind of preservative should be added as soon as possible. The preservative may be replaced with water if the trip is short. The proper preservative is then replaced upon arrival of the samples in the lab. Frozen specimens may be transported on ice, or in insulated containers without ice, if the trip is short enough so that thawing will not occur.

4.4 Receipt in laboratory, laboratory curation, and storage

Upon receipt of specimens in the laboratory, all samples should be noted in a sample log book. Notes should include serial or collection numbers (or complete ranges of serial or collection numbers), dates and localities of collection, number of containers and method of preservative, and location of specimens.

Frozen specimens should be placed in freezers maintained at less than 0°C as soon as possible after being logged in. Samples from each field trip can be placed in one large plastic bag in order to keep them separated from specimens from other projects. Within the large plastic bag, smaller bags can be used to keep species and sites separated and organized. Frozen specimens should be maintained in frozen state until laboratory analysis, at which time they should be partly or completely thawed, and handled according to the requirements of the analysis.

Specimens preserved in liquid should be checked for adequate preserving conditions as soon as possible after being logged in. If necessary, specimens may be switched to different containers, or preservative may be replaced or added. The original field tag should remain with the sample. If specimens from one container are placed in more than one container, duplicate tags should be made and placed in the additional containers. If deterioration of the field tag is noted, a copy should be made and placed in the container. The original tag may be kept in the container or dried and affixed within the bound field notebook.

Specimens preserved in ethanol can be held in ethanol until laboratory analysis. Specimens should be checked periodically and the preservative changed if necessary (e.g., as indicated by changes in color of preservative or deterioration of specimens).

Larval fishes preserved in 5% formalin may be held in 5% formalin indefinitely.

Larger specimens preserved in formalin should be held in formalin a minimum of 1 week for fixation. After fixation, formalin should be drained from the specimens, the specimens rinsed in water, drained and held in water for 12-48 hours. After the water rinse, specimens should be transferred to 50% ethanol. After a minimum of 12 hours rinse in 50% ethanol, specimens should be placed in 70% ethanol for permanent curation. Specimens which will not be held permanently may be stored in 50% ethanol until disposal.

All containers in which specimens are stored or transported should be watertight. Laboratory containers should be sufficiently airtight and watertight to prevent leakage and evaporation of preservative. Specimens should be checked periodically to determine whether liquid loss has occurred. If so, additional preservative should be added; the container should be replaced if substantial loss has occurred. Specimens should be entirely covered in preservative during holding and storage, except for short periods for transportation or analysis.

Specimens may be held for short periods of time until analysis, held for a length of time outlined in the project plan and then discarded, maintained indefinitely within the Fisheries Section for reference purposes, given to other agencies or individuals, or deposited in the permanent fish collection of the Academy. The change of custody of any specimens which are discarded, given to another group, or deposited in the fish collection should be noted in the sample log book of the Fisheries Section. Chain-of-custody forms will be filled for each transfer or moving of specimens even if not outlined in the project plan.

Attachment H-12

ANSP Protocols for Preparation of Fish Samples for Contaminant Analysis

PREPARATION OF FISH SAMPLES FOR CONTAMINANT ANALYSIS

1.1 Introduction

The following procedures are used for preparing tissue samples from whole biological specimens for contaminant analysis. Pertinent information on laboratory procedures for fish sample handling and preparation from the EPA document EPA 823-R-95-007 (U. S. EPA. 1995) and the NOAA Technical Memorandum NOS ORCA 71 (July 1993), has been incorporated into this standard operating procedure.

Fillets, whole-body, or other tissues of interest (selected organs) of target species can be analyzed. The target species, tissues or organs to be used, and specimen size ranges to be analyzed will be specified in the project protocol. Larger fish are typically filleted unless specified otherwise in the project protocol. Young-of-year fish and other small specimens (e.g., *Gambusia*- mosquitofish) can be composited into one sample using whole specimens, as outlined in the specific project protocol.

1.2 Preservation of Samples

1.2.1 Depending on the analysis required (mercury, trace metals, organic, etc.) and the project protocol, there are recommended containers, preservation and holding times for the fish tissue to be followed [refer to appendix #1]. In general, clean, muffled (3 hours at 420°C) extra heavy duty aluminum foil is used to wrap whole fish specimens that will be analyzed for organic analysis. Fish to be analyzed for metals and organic compounds will also be wrapped in muffled extra heavy duty aluminum foil (of sufficient thickness to prevent puncture). Note: Before wrapping catfish specimens, the pectoral and dorsal spines are typically folded back or removed with a clean tool. If the analysis required is for either mercury or other trace metals, clear polyethylene (i.e., Ziploc) bags are used to package the whole specimen (individually bagged specimens of the same species are typically placed in another outer poly or large kitchen type bag to prevent puncture).

1.2.2 An external tag should be affixed to the outer specimen container. If the specimen is to be analyzed for organics, permanent markers (i.e. Sharpies or Marks-A-Lot) **should not** be used. Specimens wrapped for organic analysis should have a paper outer tag written on with pencil or pen. For organic analysis, the internal tag should be a paper tag written with pencil and placed in the container so as to not contact the specimen's body. Samples to be analyzed for metals and/or mercury can have external tags and containers marked with permanent markers. An alternate internal specimen tag can also be used. The alternate internal tag is a numbered polyolefin tubing anchor tag (i.e. Floy T-Bar anchor tag) which is inserted into the cranium (or other part of the body which will not be analyzed) of the specimen for the purpose of identifying the specimen. This type of internal tag is preferred when samples are to be analyzed for mercury or metals, but not for organic samples.

1.2.3 If samples have been shipped on dry ice, they may be distributed immediately to the technician for processing or stored in a freezer at ≤ -20.0°C for later processing. If fresh (not yet

frozen) samples are being prepared, the samples must be maintained in a wrapped condition on wet ice, or "blue" encased freezer packages, or in a refrigerator for no longer than 24 hours before processing.

1.3 Types of Fish Samples and Fillets

In general, a fillet is the section from one side of the whole fish which includes the tissue from just behind the operculum (gill cover) to the base of the caudal fin (tail), and the area from the dorsal surface (below and to either side of the dorsal fin) to the length of the belly from the pectoral fin to the base of the caudal fin (tail). Depending on the specific project protocol, the fillet may or may not contain the area of tissue around the abdominal cavity, commonly called the belly flap. Typically, the fillet should not include any fin rays or bones, unless when preparing the "New York standard fillet", (see section 1.3.4). Approximately 20-50 g of fillet tissue are needed for organic analysis. More tissue may be needed if analyzing for both metals and organics (>50 g), and less tissue (5-10 g) is needed for mercury or other trace metal analysis. Depending on the project protocol and analysis required, there are various types of fish samples and fillets that may be used:

1.3.1 Fillet with skin, but with scales removed–this is the default type of tissue sample for most scaly fish (i.e. sunfish and shad). This sample consists of the entire fillet or pairs of fillets (right and left sides), overlaying skin, and belly flap meat. Scales are manually removed from the skin with a clean fish scaler or fillet knife.

1.3.2 Fillet with skin and scales–unless otherwise noted, trout and other fine-scaled fish (i.e., mackerel) will be prepared and analyzed in this way. This consists of the entire fillet or pairs of fillets and overlaying skin and scales. The fillet is taken from behind the head and pectoral fins to the base of the tail, and includes the belly flap tissue. The fillet does not include ribs or pelvic fins.

1.3.3 Fillet without skin–(i.e. gar, catfish, eel, sturgeon), This consists of the entire fillet, including the belly flap tissue, with the skin and scales removed. For sturgeon, the bony scutes and skin are removed, with gar the ganoid scales and skin are removed, and with catfish (no scales) only the skin is removed; refer to Section 1.6.6 for procedures on filleting these type of fish.

1.3.4 Fillet with skin on (except catfish and eels), scales off, and including pelvic fin, rib cage, and belly meat (equivalent to USFDA fillet and "New York standard fillet").

Other samples that may be used are:

1.3.5 Whole fish–whole body samples consist of the entire specimen, with the exclusion of surface mucous. Whole body samples may be prepared by either of two methods. If separate tissue samples are also taken, the carcass (defined as the remainder of the specimen after removal of any tissue samples) is used as a sample. The whole body contaminant concentration is

subsequently estimated as the average of the concentrations within the carcass and other tissues, weighted by the total wet weight of each tissue. If no separate tissue samples are taken, the whole specimen is used as the sample. Whole body samples should be chopped up into small pieces and all material including body fluids retained for analysis; (the removal of otoliths for aging is assumed not to affect total estimate);

1.3.6 Fish with head and viscera removed–this sample consists of the entire specimen, excluding the head, visceral mass, the swim bladder and surface mucous. This sample type is typically used only for small specimens;

1.3.7 Carcass-after removal of fillet(s)–may be used to compare fillet and whole body concentrations from same specimen(s);

1.3.8 Other portions or organs of fish–gonad(s). One or both ovaries or testes, containing the external membrane and all internal gonadal material, including ova of mature ovaries. External fat bodies will be removed from the tissue. Liver. The entire liver and external membrane;

1.3.9 Skinned predorsal strip–this consists of the section of tissue between the following cuts: 1) a vertical cut from anterior to the dorsal fin down the lateral line; 2) a vertical line just posterior to the gill cover down to the lateral line; 3) a horizontal line along the lateral line. For species without lateral lines anterior of the dorsal fin, the level of the vertebral column should be used in place of the lateral line.

1.4 General Procedures

1.4.1 Specimens to be processed for a specific project will be selected by the Fisheries Section Leader or Project Principal Investigator. The fish will either be selected from the Chemistry Fish- Pick Fish form (accessible from the Main Menu form) of the database, or by written memorandum from the Fisheries Section Leader. If database selection is used, the Pick Fish query will list all fish which were retained in the field for that specific project. Fish will be selected by assigning a yes to the chemistry column in the Pick Fish query.

1.4.2 Depending on the analysis being conducted (i.e., mercury, metals, organics, volatiles etc.), special cleaning and clean room procedures should be followed. If analysis for volatile compounds is being preformed, filleting should be completed in the Organic Laboratory (in Chemistry), or similar lab facility. The Organic lab is maintained as a chemical-free environment with fume hoods which provide for a cleaner (solvent and dust free) atmosphere, thus minimizing any chances of airborne contamination during the filleting process. Within this laboratory, care should be taken to preform all equipment cleaning procedures within an operating fume hood. All chemicals used in the cleaning process should be stored within the fume hood. The fish processor will completely clean the filleting area of the laboratory with dilute Micro, Alconox (or equivalent) cleaner . The work area will be covered with clean lab bench paper. Clean lab bench liner will be installed when the existing liner gets soiled.

Appropriately cleaned (see Sections 1.5, 1.7 and 1.9) filleting materials and tools will be obtained for use in the filleting procedure.

1.4.3 The laboratory worker will select Data Entry from under the Chemistry Fish selection in the Main Menu form of the Fisheries Database, or will be provided with a list of samples to be completed for the specific project. The fish samples listed in this query or list are the complete set of fish which were selected by the Section Leader or Principal Investigator for analysis for that specific project.

1.4.4 The laboratory worker will remove from the freezer (or refrigerator if fresh), a selected number of fish specimens (indicated in the Data Entry list) that can be successfully filleted in the amount of time he or she will be processing that day. The integrity of the wrapping should be inspected, and the identification of the samples should be confirmed by comparing the outer tag with the Data Entry listing. At this point, the worker begins filling out the Fisheries Section Internal Chain-of-Custody Form. The chain will document the fillet process from the removal of the specimen from the freezer to the transfer of the fillet to the Chemistry Department, and to the specimen's final storage disposition. The laboratory worker should also refer to the field chain-of-custody and check to see if all specimen packages were received in good order. Requirements for sample chain-of-custodies are covered in standard operating procedure # Q-00-11.

1.4.5 Unless processing fresh fish from the field, thawing of fish prior to filleting in the laboratory is necessary. Samples are partially or totally thawed by placing them in a refrigerator or leaving them out at room temperature, for an appropriate time depending on the size of the fish sample. Ideally, fish should be filleted while ice crystals are still present in the muscle tissue. Samples should remain wrapped or covered during the thawing process. Fish should be thawed only to the point where it becomes possible to make an incision into the flesh.

1.4.6 The sample package is opened and it is confirmed that the inner tag (paper tag or numbered anchor tag attached to cranium) matches the outer tag and also matches the information in the Data Entry database. The fish specimen is typically tagged in the field with a numbered head tag, which is inserted into the cranium. This tag becomes the fish tracking number and represents the inner tag of the packaged fish. Any discrepancies in labeling are noted and resolved with the Principal Investigator or Field Crew Chief of that particular project. Notes on discrepancies and their resolution should be recorded on the chain-of-custody form.

1.4.7 Taxonomic identifications are confirmed and checked against the labels and database. Additional data on the specimen(s) are taken as required in the work plan. At a minimum, the total length (cm), weight (g) and sex of the fish are recorded. Other data obtained during filleting can include: recording lesions or abnormalities and fin or mouth clips from the specimen. See Section 1.6.8.

1.5 Materials and Cleaning Methods for Organic Analysis

The materials used in filleting and processing the fish to be used for organic analysis should be of stainless steel, anodized aluminum, borosilicate glass, polytetrafluoroethylene (PTFE-teflon), ceramic, or quartz. Polypropylene and polyethylene (plastic) surfaces, implements, gloves, and containers are a potential source of contamination by organics and should not be used. If these materials are chosen, there should be clear documentation that they are not a source of contamination.

Prior to and at the end of each filleting session and between each sample, all utensils and materials will be thoroughly washed with dilute detergent solution (Micro), rinsed with deionized water, rinsed with methanol and hexane by either dipping the utensils into glass containers of the cleaners, or by spraying the utensils with a cleaner from a teflon wash bottle. Either cleaning method (dipping or spraying) is a suitable technique. The utensil should be allowed to dry under a hood before use. **Caution!** Methanol and hexane washes should take place under a suitable fume hood and the waste generated should be retained in appropriate bottles for subsequent hazardous waste disposal. Note: Pesticide grade isopropanol or acetone rinse can be substituted for the methanol/hexane rinse.

Between each unique fish sample (or composited sample), the fish scaler, fillet knife and scalpel should be washed in dilute Micro (or equivalent detergent), rinsed in distilled water, rinsed in methanol and rinsed in hexane. Glass plates, if used, are used only for one sample, or sample composite. After filleting on the glass plates, the plates are cleaned with Micro and rinsed with distilled water prior to final washing in the Chemistry washroom. All cleaned equipment will remain sealed with appropriate lids or wrapped with muffled aluminum foil until subsequent use.

Materials and equipment include:

1.5.1 Filleting materials include: high-quality, corrosion-resistant stainless steel fillet knives or high grade stainless steel disposable scalpel blades. For trace metal and organic analysis, it is preferred that knives with titanium blades and PTFE handles are used, if possible (suitable for trace metals also).

1.5.2 Cleaning and rinsing materials include: dilute Micro (or equivalent detergent), deionized water, 50 % nitric acid, methanol, hexane, isopropanol, and acetone.

1.5.3 Laboratory processing equipment includes: polyethylene gloves (not powdered), calibrated laboratory balance, clean fish measuring board, teflon or teflon-covered fish filleting board, clean large glass plates for filleting, and pre-cleaned borosilicate glass sample jars (either cleaned by the Chemistry Laboratory or purchased as certifiable clean sample jars from an appropriate vendor). The fillets are stored in these sample jars with either muffled aluminum foil or teflon cap liners.

1.5.4 Data recording equipment includes: computer with fisheries database, numbered tape with fisheries analytical code, index card tags, pencil, pen, and "freezer" tape (to seal foil package).

1.6 Preparation of Fish Samples to be Used for Organic Analysis

1.6.1 Assign a unique analytical number to the sample. Using pre-numbered adhesive tags (e.g., F0432-where the F in the number represents a Fisheries-generated sample) which are printed in triplicate, place one tag on the on the outside of an appropriately-sized fillet sample jar, another on the external tag attached to the original fish sample, and the third attached to a small scintillation vial which will hold the otoliths, if otoliths are to be removed for aging.

1.6.2 Place a pre-cleaned flat glass plate on a calibrated laboratory balance and tare the glass plate. At this point and throughout the entire filleting procedure the laboratory worker is to wear a clean pair of non-powdered clear polyethylene disposable gloves. Worker will use new gloves for each sample.

1.6.3 After removing the fish from the field wrapping/container, the fish may be rinsed with deionized water to remove excess mucous and debris (including pieces of foil) . After rinsing and allowing fish to drip dry, place the specimen on the pre-cleaned glass plate.

1.6.4 Place the glass plate with the fish specimen on the balance and enter the total weight (in grams) of the fish into the data entry mode within the Chemistry Fish Form of the Fisheries Database. The entry will go into column under LGW (laboratory gross weight). The tare is typically 0 g, so LNW (laboratory net weight) will be the true weight of the fish.

1.6.5 Remove glass plate with fish and place on measuring board (do not allow fish to touch board). Enter the total length (in cm) of the fish into the database. The total length of the specimen is the distance from the anterior most part of the snout to most posterior part of caudal fin (tail) with the lobes of the fins (if present) pressed together along the midline. If the tail is damaged or missing, the best estimate of length will be recorded based on the measurements of previously measured fish in good condition.

1.6.6 The procedure described below is for preparation of fillet without skin (i.e., catfish, gar, eels), but can be used for preparation of most fillets with minor variations. For example, if preparing the fillet with scales removed, the initial step in this procedure would include removal of scales (with pre-cleaned scaler) from the fillet area before cutting. With a clean and rinsed scalpel or knife, make a vertical cut behind the pectoral fin region starting dorsally and ending ventrally. Then, starting dorsally behind the head (where the first cut started), make a second cut dorsally along the midline of the fish, staying to one side of the midline bones. Cut as close to the midline as possible to remove as much flesh as feasible; continue this cut through to the caudal fin. The tip of the fillet knife should push against the top of the rib cage in the mid-region of the fish. More posteriorly, the tip of the fillet knife should penetrate the whole fish with the tip of the knife visible ventrally. Next, peel back the fillet with attached skin from the dorsal surface with the aid of the knife, but do not completely remove fillet. Avoid puncturing the stomach or any organs. Carefully slice the exposed fillet meat from the peeled-back section of the fish without puncturing through to the skin and scales. The fillet meat should be sliced into as small pieces as is possible. If skin and scales are required for analysis (i.e.,

trout), then simply remove the whole peeled off fillet section and mince the fillet meat with skin and scales into small pieces.

1.6.7 Place a labeled (with analytical number), clean glass sample jar onto the laboratory balance and tare out the jar. Place the minced sample meat into the sample jar and record the weight of the sample tissue into the Fisheries database-Chemistry Fish entry. Depending on project protocol and analysis required, between 20-50 g of tissue (wet weight) are needed per sample. For larger specimens, e.g., smallmouth bass and channel catfish, only one fillet may be needed to obtain enough tissue for chemical analysis. If one fillet does not provide sufficient material, fillet the other side of the fish, and/or scrape any additional flesh from the skeleton. Indicate on the lab data sheet which fillet(s) were used. Fillets can include flesh and skin from behind the head to the base of the tail, including the area along the side of the abdominal cavity.

1.6.8 Other information on the fish specimen should also be entered into the database, depending on project protocol. This information could include: the sex of the fish, the type of tissue preparation, occurrence and nature of any external or internal anomalies noted, including absence or erosion of fins; deformation of any structure; cuts, scars or areas of regenerated scales; presence of apparent lesions, occurrence of other unusual conditions, e.g., large or small quantities of fat, unusual parasitic infestation, "hollow belly", sample tags or marks, food items in stomach, reproductive condition.

1.6.9 The sample container analytical number should be recorded on the internal fisheries chain-of-custody form. The container should be sealed and placed immediately into a freezer for subsequent transfer to the Chemistry Section upon completion of the days' filleting.

1.6.10 If required, the otoliths (earbones used in aging the fish) can be extracted from the cranium at this point. Alternatively, the head of the fish can be removed with the attached head tag and either refrozen, or placed into 95% ethanol for otolith removal at a later date.

1.6.11 A soon as possible after the filleting, the remains of the fish should be re-wrapped and packaged (in original package if possible), and placed back into the freezer. These remains shall be signed back onto the Fisheries Section internal chain-of-custody form after being placed back into the freezer. The outer paper tag remains with the fish specimen after filleting. After filleting, the designation "REMAINS" is written on the outer paper tag . The third numbered laboratory analytical tag should be affixed to this paper tag for further tracking.

1.6.12 If several specimens were in a field sample package and only one or none are filleted, then total length (cm) and weight (g) will be recorded, and the identification will be confirmed on the fish not being filleted. These fish that are not filleted will be either 1) signed back into the Fisheries Section log book and refrozen for later analysis, if necessary, or 2) discarded after appropriate log-out procedures are carried out. Whether and when to discard the specimens will depend on the project protocol.

1.7 Materials and Cleaning Methods for Metals Analysis (Including Mercury)

The materials used in filleting and processing the fish to be used for metal/mercury analysis should be of polytetrafluoroethylene (PTFE-teflon), ceramic, quartz, polypropylene, or polyethylene. The predominant metal contaminants from stainless steel are chromium and nickel. If these metals are not of concern, the use of high-quality, corrosion-resistant stainless steel for sample processing equipment is acceptable. Quartz utensils are ideal but expensive. Alternatively, knives with titanium blades and PTFE handles are recommended for performing filleting. Borosilicate glass bench liners are recommended. Filleting may be done on glass or PTFE cutting boards that are cleaned properly between fish or on cutting boards covered with muffled heavy duty aluminum foil that is changed after each fish. Fillets may be stored in pre-cleaned glass or teflon jars with teflon or aluminum foil liners.

The cleaning process is similar to that of the organic method (see section 1.5), except that solvents are not required for cleaning the utensils in metals analysis. Prior to preparing each fillet sample, all utensils and materials will be thoroughly cleaned and wiped down with a dilute Micro solution, rinsed with tapwater, rinsed with 25% nitric acid, and then rinsed with deionized water. Quartz, PTFE, glass, or plastic containers should be soaked in 50% nitric acid, for 12 to 24 hours at room temperature. Acids used should be at least reagent grade. Stainless steel parts may be cleaned as stated for glass or plastic, omitting the acid soaking step.

Materials and equipment include:

1.7.1 Filleting materials include: high-quality, corrosion-resistant stainless steel fillet knives or high grade stainless steel disposable scalpel blades. If sample is to be prepared with skin on and scales off, then a stainless steel fish scaler, cleaned between distinct samples can be used to remove the scales.

1.7.2 Cleaning materials include: dilute Micro (or equivalent detergent), deionized water, and reagent grade nitric acid.

1.7.3 Laboratory equipment includes: polyethylene gloves (not powdered), calibrated laboratory balance, clean fish measuring board, teflon or teflon-covered fish filleting board, clean large glass plates for filleting, and clean borosilicate glass sample jars or teflon jars with teflon lid liners.

1.7.4 Data recording equipment includes: computer with fisheries database, numbered tape with fisheries analytical code, index card tags, pencil, pen, and "freezer" tape (to seal foil package).

1.8 Preparation of Fish Samples to be Used for Metal Analysis

The fish sample preparation procedure for metals analysis is virtually the same as the procedures used in fish sample preparation for organic analysis presented in Section 1.6. The basic differences between the organic and metals preparation are in the cleaning procedures (outlined in Section 1.7), and in the amount (only 5-10 g) of fillet material needed for metals analysis.

1.9 Materials and Cleaning Methods for Both Organic and Metals Analysis

If a single sample is prepared for the analyses of both organics and metals, precautions must be taken to use materials and cleaning procedures that are noncontaminating for both organics and metals. Quartz, ceramic, borosilicate glass, and PTFE are recommended for sample processing equipment. If chromium and nickel are not of concern, high-quality, corrosion-resistant stainless steel utensils may be used. It is preferred for organic and metals combined analyses that knives with titanium or quartz blades and PTFE handles are used, if possible. Borosilicate glass bench liners are recommended. Filleting may be done on glass or PTFE cutting boards that are cleaned properly between fish or on cutting boards covered with muffled heavy duty aluminum foil that is changed after each fish. Fillets may be stored in pre-cleaned glass or teflon jars with teflon lid liners.

The cleaning process for sample analysis of both organic compounds and metals is as follows: Prior to preparing each fillet sample, all utensils and materials will be thoroughly cleaned and wiped down with a dilute Micro solution, rinsed with deionized water, rinsed or soaked in 25% nitric acid, again rinsed with deionized water, rinsed with methanol then hexane by either dipping the utensils into glass containers of the cleaners, or by spraying the utensils with a cleaner from a Teflon wash bottle. Either cleaning method (dipping or spraying) is a suitable technique. Utensils should then be placed on a precleaned piece of foil under the hood to allow the hexane to evaporate. Note: Pesticide grade isopropanol or acetone rinse can be substituted for the methanol/hexane rinse. Quartz, PTFE, glass, or plastic containers should be soaked in 50% nitric acid, for 12 to 24 hours at room temperature. Acids used should be at least reagent grade. Stainless steel parts may be cleaned as stated for glass or plastic, omitting the acid soaking step.

1.10 Preparation of Fish Samples to be Used for Both Organic and Metal Analysis

The fish sample preparation procedure for combined organic and metals analysis is identical to the procedures used in fish sample preparation for metals analysis presented in Sections 1.7 and 1.8. The only difference between the metals and the organic and metals analysis preparation is in the amount (typically >50 g, but depends on specific project protocol) of fillet material needed for the combined organic and metals analysis.

1.11 Materials and Cleaning Methods to be used for Volatiles Analysis

The procedures for materials and cleaning methods for volatiles analysis is covered in Section 1.4.2, under General Procedures. If analysis for volatile compounds is being preformed, filleting should be completed in the Organic Laboratory located in the Chemistry Section. The Organic lab is maintained as a chemical-free environment with fume hoods which provide for a clean atmosphere, thus minimizing any chances of airborne contamination during the filleting process. Within this laboratory, care should be taken to perform all equipment cleaning procedures under an operating fume hood. All chemicals used in the cleaning process should be

stored within the fume hood. Materials and cleaning methods are identical to those presented in Section 1.9.

1.12 Field Filleting

For some projects, it will be necessary to fillet fish in the field and ship the packs frozen to the ANSP Chemistry Department or another testing laboratory for chemical analysis. The necessity to fillet fish in the field should be stated in the project protocol. Depending on the specific project protocol the filleting and cleaning procedures will be the same as in Sections 1.2 through 1.9. Filleting should be conducted within one day of capture of the fresh fish. The fish should be properly wrapped (Section 1.2) in the field and placed in a clean (washed with dilute Micro detergent and rinsed with distilled water) cooler of wet or dry ice prior to being filleted. After filleting, the properly labeled packs of fillets will be shipped to the laboratory in a clean cooler of dry ice. The coolers will be securely closed with "freezer" tape or packing tape. Chain-of-Custodies and other essential notes (e.g., destination of samples, purpose of shipping, analyses needed, and method of shipping) are required prior to sending out the fish fillets. The remains of the filleted fish should be labeled, placed on dry ice, and brought to the ANSP Fisheries Section freezer unless stated otherwise in the project protocol.

1.13 Literature Cited:

ANSP Standard Operating Procedure No. P-14-12 Rev. 0 (5/91)-Preparation of Fish Samples for Contaminant Analysis.

U.S. EPA (U.S. Environmental Protection Agency). 1995. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories-Volume 1 Fish Sampling and Analysis. 2nd Edition. EPA 823-R-95-007. Office of Water. Washington, DC.

NOAA Technical Memorandum NOS ORCA 71. Volume I: "Sampling and Analytical Methods of the National Status and Trends Program National Benthic Surveillance and Mussel Watch Projects, Overview and Summary of Methods". Silver Spring, Maryland. July 1993.

Appendix #1 (see attached page)

Recommendations for Container Materials, Preservation, and Holding Times for Fish

Table 7-1. Recommendations for Container Materials, Preservation, and Holding Times for Fish, Shellfish, and Turtle Tissues from Receipt at Sample Processing Laboratory to Analysis

			Storage	
Analyte	Matrix	Sample container	Preservation	Holding time[a]
Mercury	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE	Freeze at ≤-20 °C	28 days[b]
Other metals	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE	Freeze at ≤-20 °C	6 months[c]
Organics	Tissue (fillets and edible portions, homogenates)	Borosilicate glass, PTFE, quartz, aluminum foil	Freeze at ≤-20 °C	1 year[d]
Metals and organics	Tissue (fillets and edible portions, homogenates)	Borosilicate glass, quartz, PTFE	Freeze at ≤-20 °C	28 days (for mercury); 6 months (for other metals); and 1 year (for organics)
Lipids	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE	Freeze at ≤-20 °C	1 year

PTFE = Polytetrafluoroethylene (Teflon).

[a] Maximum holding times recommended by EPA (1995k).
[b] This maximum holding time is also recommended by the Puget Sound Estuary Program (1990e). The California Department of Fish and Game (1990) and the USGS National Water Quality Assessment Program (Crawford and Luoma, 1993) recommend a maximum holding time of 6 months for all metals, including mercury.
[c] This maximum holding time is also recommended by the California Department of Fish and Game (1990), the 301(h) monitoring program (U.S. EPA, 1986b), and the USGS National Water Quality Assessment Program (Crawford and Luoma, 1993). The Puget Sound Estuary Program (1990e) recommends a maximum holding time of 2 years.
[d] This maximum holding time is also recommended by the Puget Sound Estuary Program (1990e). The California Department of Fish and Game (1990) and the USGS National Water Quality Assessment Program (Crawford and Luoma, 1993) recommend a more conservative maximum holding time of 6 months. The EPA (1995c) recommends a maximum holding time of 1 year at ≤-10 °C for dioxins/furans.

Attachment H-13

ANSP Protocols for Extraction and Cleanup of Fish Tissue for PCB and Pesticide Analysis

ACADEMY OF NATURAL SCIENCES
ENVIRONMENTAL RESEARCH DIVISION

Procedure No. P-16-77
Rev. 1 (4/95)

EXTRACTION AND CLEANUP OF FISH TISSUE FOR PCB AND PESTICIDE ANALYSIS

Prepared by: Michelle Donnelly

Approved by: Carol J Lee   Date: 5/5/95

Carol Lee
Quality Assurance Unit

EXTRACTION AND CLEANUP OF FISH TISSUE FOR PCB AND PESTICIDE ANALYSIS

Prerequisite: Use of this method requires a working knowledge of the inherent hazards and possible routes of contamination in working with organic solvents. Also, a working knowledge of glassware cleaning and standard residue analysis techniques is required.

1.0 METHOD

This method includes instructions for extracting PCBs and pesticides from fish tissue. Also, specific criteria for gas chromatography (ECD-capillary) and quantitation on a congener and compound specific basis is included. For basic instructions on gas chromatography see SOP No. P-16-84.

2.0 SUMMARY

The fish tissue is combined with sodium sulfate, Soxhlet extracted and concentrated to 10 ml. One ml of this extract is taken and analyzed for lipid content. The remainder of the extract is mixed with concentrated acid to destroy the lipid and other biogenic material and finally cleaned up by Florisil sep-pak chromatography.

3.0 STANDARDS

3.1 PCB Standard

Mixture of Aroclors 1232, 1248, and 1262 in a 25:18:18 ratio. Individual Aroclor concentrations of 250 ng/ml (Aroclor 1232), 180 ng/ml (Aroclor 1248), and 180 ng/ml (Aroclor 1262) are recommended for total PCB concentration of 610 ng/ml.

3.2 Pesticide Standard

Mixed pesticide standard containing 19 organochlorine pesticides and industrial compounds.

3.3 Internal Standard

80 ng of 2,4,6-trichlorobiphenyl (PCB 30) and 60 ng of 2,2',3,4,4',5,6,6'-octachlorobiphenyl (PCB 204).

3.4 Surrogate Standard

210 ng of 3,5-dichlorobiphenyl (PCB 14), 50 ng of 2,3,4,4',5,6-hexachlorobiphenyl (PCB 166), and 200 ng of delta hexachlorocyclohexane (δ-HCH).

4.0 APPARATUS

4.1 Glassware (all cleaned using SOP No. P-16-37)

For Extraction: Soxhlet extractors (200 ml), Allihn condensers, 500-ml round bottom flasks, glass thimbles (4 cm x 11 cm) .

For Sample Preparation: 250-ml beakers, stainless steel spatula, 10-ml volumetric flasks, syringe with stainless steel needle, 12 ml vials with Teflon lined screw caps.

4.2 Glass wool for extraction.

4.3 Rotary Evaporator for sample reduction.

4.4 Sodium Sulfate (pre-extracted overnight in dichloromethane).

4.5 Waters Florisil Sep-pak cartridges.

4.6 Sulfuric Acid

4.7 Tekmar Tissuemizer.

4.8 Heating mantles and voltage controllers for extraction.

4.9 Teflon boiling chips (pre-extracted overnight in dichloromethane).

5.0 SAMPLE PREPARATION

5.1 Frozen fish fillets are allowed to thaw and are finely ground using the Tekmar Tissuemizer.

5.2 At the time of analysis, 10.0 g of thawed fish sample is weighed and placed into a 250-ml beaker. The sample is then combined with sodium sulfate in a 1:6 ratio (sample: sodium sulfate) and mixed with a clean spatula until the sample is homogenized.

5.3 The sample mixture is transferred to a glass thimble with glass wool at the bottom and placed into the Soxhlet extractor. At this point the surrogate standard is added. The sample is then extracted overnight (refluxing at least 16 h at 4-6 cycles/h) with 350 ml of 1:1 hexane:acetone mixture.

5.4 The sample extract is then transferred quantitatively from the original 500-ml round bottom flask to a clean flask, with two 25-ml aliquots of hexane. This is done because during extraction, fish and sodium sulfate collect at the bottom of the flask. The extract is reduced to approximately 5 ml using a rotary evaporator, exchanged three times with 25-ml aliquots of hexane, and finally evaporated to 5 ml. Between exchanges the sample is checked for water. If water is present, it is removed with a pasteur pipet.

5.5 The sample extract is then diluted to 10 ml with hexane using a 10-ml volumetric flask. The lipid content of the sample is determined at this point by placing a 1.0-ml aliquot of the extract in a preweighed aluminum pan. This is allowed to sit at room temperature overnight to dry. The pan is reweighed and the % lipid calculated.

$$\% \, Lipid = \frac{g \, of \, lipid}{total \, sample \, wt. \, (g)} \times 1000$$

5.6 The remaining sample extract is concentrated under a stream of ultra high purity (UHP) nitrogen to approximately 2 ml. It is then washed with an equal volume of sulfuric acid and stored in the refrigerator at 4°C overnight or until separation occurs. In cases where lipid content is high it may be necessary to add more sulfuric acid and hexane. The sample extract is returned to the refrigerator to separate. The hexane phase is transferred to another vial, and the acid phase is washed 2-3 times more with 1-2 ml of hexane, combining all hexane washes. The sample extract (in hexane) is then reduced to approximately 2 ml under a stream of UHP nitrogen.

5.7 The sample extract is cleaned by Florisil column chromatography using Waters sep-pak cartridges. The column is pre-rinsed with approximately 10 ml of hexane which is discarded. The sample is then passed through the column. All deliveries to the sep-pak column are made using a glass Luer-lock syringe. To collect the hexane fraction for PCB analysis, the column is then rinsed with four bed volumes of hexane and collected into a 10-ml volumetric flask, and the volume adjusted to 10 ml. After the hexane has run through the syringe an equal amount of dichloromethane is run through the sep-pak to obtain the fraction for pesticide analysis. The dichloromethane fraction is blown down to ~1 ml under N_2 then combined with an equal amount of hexane. This is repeated three more times, and the remaining sample is adjusted to 10 ml with hexane. The sample is then transferred to a 12-ml vial. The sample is now ready for analysis.

6.0 STANDARDS

(For specific volumes and directions see Organic Standards Preparation Logbook.) The following concentrations are recommended based on past GC performance and levels of contaminants typically observed in recent projects.

Working Standards:

PCB Standard: 250 ng/ml of Aroclor 1232, 180 ng/ml of Aroclor 1248, and 180 ng/ml of Aroclor 1262 to yield a total PCB concentration of 610 ng/ml.

Pesticide Standard: Mixed pesticide standard with 19 organochlorine pesticides and industrial compounds of environmental interest.

Surrogate Standard:

210 ng of 3,5 dichlorobiphenyl (PCB 14), 50 ng of 2,3,4,4',5,6 hexachlorobiphenyl (PCB 166), and 200 ng of delta hexachlorocyclohexane (δ-HCH) are added to the sample before extraction.

Internal Standard:

80 ng of 2,4,6 trichlorobiphenyl (PCB 30) and 60 ng of 2,2',3,4,4',5,6,6'- octachlorobiphenyl (PCB 204) are added to the 10-ml sample just before analysis on the GC.

7.0 QA/QC

7.1 Laboratory duplicate, laboratory blanks, and standard reference materials (SRMs) are extracted and analyzed at a frequency of 5 to 10% depending on requirements specified by the contract. Blank spikes are extracted and analyzed at an unspecified frequency to evaluate method performance. Surrogate recoveries provide some measure of method performance for individual sample matrices. Analyte recoveries for SRMs reflect method performance for a variety of compounds in a given type of matrix. Hence, SRMs are used in lieu of conventional matrix spikes in this procedure.

8.0 AROCLOR QUANTITATION

Aroclor 1254 is quantitated as the sum of congeners 52, 49, 44, 41, 74, 70+76, 95+66, 91, 60+56, 84, 101, 99, 83, 97, 87, 85, 110, 82 divided by 0.5252.

Aroclor 1260 is quantitated as the sum of congeners 178, 187, 183, 185, 174, 177, 171, 172, 180, 170, 201, 203+196 divided by 0.3747.

Attachment H-14

ANSP Protocols for Quantification of Individual Polychlorinated Biphenyl Congeners (PCBs), chlorinated Pesticides, and Industrial Compounds by Capillary Column Gas Chromatography

ACADEMY OF NATURAL SCIENCES
PATRICK CENTER FOR ENVIRONMENTAL RESEARCH

Procedure No. P-16-84
Rev. 2 (6/99)

QUANTIFICATION OF INDIVIDUAL POLYCHLORINATED BIPHENYL CONGENERS (PCBs), CHLORINATED PESTICIDES AND INDUSTRIAL COMPOUNDS BY CAPILLARY COLUMN GAS CHROMATOGRAPHY

Prepared By: Jeffrey Ashley

Approved By: Robin S. Davis Date: 6/24/99
Robin S. Davis
Quality Assurance Unit

Quantification of Individual Polychlorinated Biphenyl Congeners (PCBs), Chlorinated Pesticides and Industrial Compounds by Capillary Column Gas Chromatography

1. SCOPE AND APPLICATION

1.1. This method describes the analysis and quantification of polychlorinated biphenyls (PCBs), selected chlorinated pesticides and industrial compounds by capillary column gas chromatography (GC) with an electron capture detector (ECD). PCBs are quantified on a congener specific basis using this method. The compounds that can be determined by this method are listed in Appendices A and B.

1.2. The selection of compounds of interest may be specified in the project protocol, may be based on existing site data or based on initial screening of samples.

1.3. The analysis is preceded by extraction and clean-up as stated in the relevant SOP for each particular matrix.

1.4. Standards.

1.4.1. A PCB standard is composed of a mix of Aroclors which is composed of most congeners that would be found in environmental samples. Individual congeners of environmental interest not found in the Aroclor mix or found in amounts just above the limit of quantification may be added to the standard. The congeners can be summed for a total PCB (*t*-PCB) value.

1.4.2. A mixed pesticide standard is composed of a mixture of 19 organochlorine pesticides and industrial compounds that are found in environmental samples. Other chlorinated organic compounds of environmental interest may be added to the standard.

2. SUMMARY OF METHOD.

2.1. This method describes a procedure to determine PCBs and pesticides by capillary column gas chromatography (GC) with electron capture detection (ECD). Before using this method, refer to the appropriate sample extraction and clean-up techniques. The clean-up technique (Procedure Nos. P-16-109 and P-16-111) can generate several eluent fractions of different polarity which are analyzed separately to minimize interferences. The first fraction is eluted using a non-polar eluent (petroleum ether). This fraction contains all PCB congeners and some chlorinated pesticides and industrial compounds. The second fraction is eluted with a moderately polar eluent (50:50 dichloromethane:petroleum ether). This fraction contains the

remaining chlorinated pesticides and industrial compounds. Other more polar fractions may follow.

2.2. Samples are quantified on a congener-specific basis using a standard mixture of Aroclors 1232, 1248, and 1262. This mixture may be supplemented with individual congeners of particular environmental interest. Organochlorine pesticides and industrial compounds are quantified using a separate standard containing 19 such compounds of interest. Confirmation of selected analytes may be performed on a second capillary column possessing a different stationary phase.

3. APPARATUS AND MATERIALS.

3.1. Gas Chromatography.

3.1.1. Hewlett Packard (HP) 5890 Series II GC or similar with dual split/splitless injection ports equipped for capillary columns.

3.1.2. Columns.

3.1.2.1. Column 1: J & W Scientific DB-5 capillary column, part number 128-5052, (5% -phenyl) - methylpolysiloxane stationary phase, 50-m x 0.20-mm I.D., 0.33-μm film thickness, or equivalent.

3.1.2.2. Column 2: J & W Scientific DB-1701 capillary column, 30-m x 0.25-mm I.D., 2-μm film thickness, or equivalent.

3.1.3. HP 19233 electron capture detectors (ECDs), or equivalent.

3.1.4. HP 7673 autosampler (optional).

3.1.5. HP Vectra or other personal computer with HP 3365 Chemstation software, or equivalent.

3.1.6. HP Laserjet 4 or other printer compatible with above computer and software, or equivalent.

3.2. Gases.

3.2.1. Make-up gas - 5% methane/95% argon.

3.2.2. Carrier gas - helium or hydrogen (preferred).

4. REAGENTS, SOLVENTS, AND STANDARDS.

4.1. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used if it is determined that the reagent is of sufficiently high purity to permit its use without compromising the accuracy of the determination.

4.2. Solvents.

4.2.1. Hexane - Pesticide quality or equivalent.

4.2.2. Dichloromethane - Pesticide quality or equivalent.

4.3. Standards.

4.3.1. Standards of the Aroclors, individual congeners (for surrogates and internal standards) and organochlorine pesticides of interest are purchased from a commercial supplier.

4.3.2. Surrogate standards- 3,5-dichlorobiphenyl (PCB 14), 2,3,5,6- tetrachlorobiphenyl (PCB 65), and 2,3,4,4',5,6-hexachlorobiphenyl (PCB 166) which are used in the analysis of the nonpolar clean-up fraction and dibutylchlorendate which is used in the analysis of the moderately polar clean-up fraction are purchased from a commercial supplier. Other surrogates may be used in conjunction with or in place of the above as required for special applications.

4.3.3. Internal standards- 2,4,6-trichlorobiphenyl (PCB 30) and 2,2',3,4,4',5,6,6'-octachlorobiphenyl (PCB 204) are purchased from a commercial supplier as certified standards. Other internal standards may be used in addition to or in place of the above if appropriate for a particular application.

4.4. Performance standards.

4.4.1. PCB standard: A mixed congener standard that contains most congeners that would be found in environmental samples is made by mixing Aroclors 1232, 1248, and 1262 in a 25:18:18 ratio (250, 180, 180 ng/ml recommended for a total concentration of 610 ng/ml). This mix is supplemented with individual congeners of environmental interest which are not found or are found in very low amounts in these Aroclors. Other congeners of interest may also be added to the mixture. This standard will also contain surrogate standards (see Section 4.6 below) and internal standards (see Section 4.7 below). The absolute concentration may be changed to

accommodate individual detector sensitivities, but their same relative proportions should be maintained. This standard solution will be used to check instrument performance, reproducibility, and sensitivity. An example of an acceptable standard chromatogram is shown in Figure 1.

4.4.2. Pesticide standard: The above PCB standard will also contain 11 chlorinated pesticides and industrial compounds which elute partially or completely in the nonpolar fraction of sample clean-up with the PCBs. A mixed pesticide standard (MPS) which contains 19 chlorinated pesticides and industrial compounds (including the above 11 from the PCB standard) that would be found in environmental samples is used to quantify analytes eluting in the moderately polar clean-up fraction. This standard will also contain a surrogate standard (See Section 4.6 below) and internal standards (see Section 4.7 below). The absolute concentration may be changed to accommodate individual detector sensitivities, but their same relative proportions should be maintained. This standard solution will be used to check instrument performance, reproducibility, and sensitivity. Examples of acceptable standard chromatograms are shown in Figures 2 and 3.

4.5. Calibration standards: Calibration standards will be used to generate response factors for quantitation (see Section 5.4). The standards shall have the same composition as the performance standard (see above), but may differ in total concentration. Concentrations of the calibration standards shall be chosen based on the type of matrix being analyzed, its expected PCB concentration, and the method chosen for instrument calibration (see Section 5.4).

4.6. Surrogate standards: A surrogate standard will be used to monitor analytical recoveries of PCB congeners. Four surrogate standards may be added to each sample, matrix spike, and blank before extraction. The surrogates for the PCB analysis are PCB congeners 3,5- dichlorobiphenyl (PCB 14), 2,3,5,6- tetrachlorobiphenyl (PCB 65), and 2,3,4,4',5,6- hexachlorobiphenyl (PCB 166). These congeners will also serve as surrogates for the pesticides and industrial compounds that elute in the nonpolar fraction of sample clean-up. Recommended concentrations in the 610 ng/ml performance standard (Section 4.4.1 above) are 25, 5, and 5 ng/ml, respectively. The surrogate for the chlorinated pesticides and industrial compounds analysis eluting in the moderately polar fraction of sample clean-up is delta HCH. The recommended concentration in the MPS performance standard (Section 4.4.2 above) is 20 ng/ml. Other surrogates may be used in conjunction with or in place of the above as required for special applications.

4.7. Internal standards: Internal standards are used in the quanticiation of all PCB congeners, chlorinated pesticides, and industrial compounds. They are added to samples just before instrumental analysis. A minimum of two internal standards are required, and these include 2,4,6- trichlorobiphenyl (PCB 30) and 2,2',3,4,4',5,6,6'-

octachlorobiphenyl (PCB 204). Recommended concentrations in the 610 ng/ml performance standard (Section 4.4 above) are 8 and 6 ng/ml, respectively. Other internal standards may be used in addition to or in place of the above if they are more appropriate for a particular application.

4.8. Storage of Standards: All standard solutions are to be kept in vials or bottles with Teflon-lined screw caps and stored in a freezer and protected from light. Stock standards should be checked frequently for signs of evaporation, especially just before preparing calibration standards. Stock standards must be replaced after one year, or sooner if problems are apparent.

5. PROCEDURE.

5.1. The extraction and clean-up procedure should follow the appropriate SOP for a given matrix. Although the procedures vary to some degree for different sample matrices, a nonpolar (hexane eluent) and a moderately polar (DCM or DCM/Hex eluent) fraction can be collected for any clean-up procedure. The nonpolar will contain PCBs and 11 chlorinated pesticides and industrial compounds which elute partially or completely in this fraction. The moderately polar fraction will contain the remaining pesticides and industrial compounds.

5.2. Instrument Parameters.

5.2.1. Analysis of samples by high resolution (capillary column) gas chromatography (GC) with an electron capture detector (ECD) is required. It is assumed that GC-ECD analysis will be the method of choice for quantitation because of enhanced sensitivity to organochlorines. An example of the GC instrumental conditions is listed in Table 1. Deviations from these parameters will be acceptable provided instrument performance criteria are met (see Section 5.2.2). If a particular set of congeners is of more interest than others, then the temperature program may be modified to attain better separation in the area of interest.

5.2.2. A calibration standard will be analyzed and the instrument recalibrated with each group of 10-20 samples (depending on project requirements) to monitor resolution, reproducibility, and sensitivity.

5.3. GC Analysis.

5.3.1. Set up GC operating conditions as described in the Section 5.2.1.

5.3.2. The injection is made utilizing an autosampler. A volume of 1.0 µl is used. Manual injection, if necessary, will use at least a 2.0-µl injection. A splitter may

be used at the injector to run the sample on both the primary and confirmation column simultaneously.

5.3.3. Samples are analyzed in a set referred to as an analytical sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with calibration standards. The sequence ends when the set of samples has been injected.

5.3.4. If the sample responses result in poor chromatographic resolution, the extract is diluted and reanalyzed. Additional internal standard may be required in the diluted samples.

5.3.5. If detection is prevented by the presence of interferences further clean-up may be required, such as copper clean-up for sulfur (see SOP P-16-79, Section 4.7). Other procedures such as GPC or alumina clean-up may be called for.

5.4. Quantification .

5.4.1. Quantification of individual PCBs congeners and pesticides will be congener- or compound-specific and performed using the internal standard method. This method eliminates errors due to variation in the sample injection, and is independent of the final extract volume. The internal standards that will be used are PCB congeners 30 and 204. The internal standard will be added to each sample before GC analysis at a concentration similar to the sample components. Surrogate recoveries will provide a measure of analytical losses and are reported with the congener values for each sample.

5.4.2. Relative response factors relative to the internal standard (RRF) will be generated as required by instrument calibration criteria:

$$RRF = \left(\frac{Mass\ Congener}{Area\ Congener} \right)_{std} \div \left(\frac{Mass\ istd}{Area\ istd} \right)_{std} \qquad \textbf{(1)}$$

5.4.3. Congener masses can be calculated from the known total PCB concentration of the calibration standard and the congener composition of the standard (Mullin 1985, see Appendix A). Average RRFs can be determined in one of two ways. (1) Three calibration standards encompassing the expected range of PCB concentrations in the samples can be used to generate RRFs. These standards must encompass a range of at least one and one half orders of magnitude. The internal standard concentrations in each different standard solution must be the same. Sample concentrations that fall outside the range of the calibration

standards should be diluted or concentrated as needed and re-run. This method will be sensitive to non-linear responses in the electron capture detector and should only be used over the established linear range of a particular instrument. (2) A single calibration standard can be used to generate RRFs. This method is also sensitive to non-linear responses of the electron capture detector, and the calibration concentration should be within a factor of five of the concentrations of PCBs in the sample extracts. Sample extracts that fall outside this range should be either diluted or concentrated but only without losing less-concentrated compounds.

5.4.4. Congener concentrations will be calculated from the average RRF, and the internal standard response in the sample, by the following equations:

$$(mass\ congener)_{sample} = (area\ congener)_{sample}\ X\ RRF_{std}\ X \left(\frac{mass\ istd}{area\ istd}\right)_{sample} \quad \textbf{(2)}$$

5.4.5 For PCB analysis, congeners eluting before and including PCB 110 will be quantitated relative to internal standard PCB 30. Congeners eluting after and including PCB 82 will be quantitated relative to internal standard PCB 204.

5.4.6 For pesticide analysis pesticides eluting before and including o,p -DDE will be quantitated relative to internal standard PCB 30. Pesticides eluting after and including Dieldrin will be quantitated relative to internal standard PCB 204.

6. QUALITY CONTROL.

6.1. With each group of 10-20 samples analyzed (depends on project QC requirements), the calibration check standards should be evaluated to determine if the chromatographic system is operating properly. If any changes are made to the system, recalibration of the system must take place.

6.2. The performance of the entire analytical system should be monitored, on the basis of data gathered from analyses of blank, standard and replicate samples at a 5-10% frequency (depending on project QC requirements). Significant peak tailing must be corrected. Tailing problems are generally traceable to active sites on the GC column or to the detector operation.

6.3. A blank, a matrix spike or standard reference material sample, and a duplicate or matrix spike duplicate (if available) must be analyzed at a minimum frequency of 5-

10% of samples (depending on project QC requirements), interspersed with each extraction group.

6.4. Limits of detection (LOD) and quantitation (LOQ).

6.4.1. The LOD is defined as the signal that is equal to the sum of the mean noise and 3 standard deviations (σ) of the baseline noise (Keith et al. 1983). The area of the baseline noise over the elution time of each congener shall be determined from injections of a matrix blank that has been spiked with the performance standard to yield a concentration just above the expected LOD (1-5x est. LOD). This procedure is described in the Federal Register (1984). The mean and the standard deviation of the baseline noise for each congener will be determined from injections of seven analyses of the spiked blank. The LOQ is defined as the signal that is equal to the sum of mean noise and 10σ of the baseline noise and is determined in the same manner as the LOD:

$$LOD = \text{mean noise} + 3\sigma \text{ (expressed as peak areas)} \quad (4)$$

$$LOQ = \text{mean noise} + 10\sigma \text{ (expressed as peak areas)} \quad (5)$$

6.4.2. LOD and LOQ, expressed as mass of congener injected, can then be determined as shown in section 5.4, Equation 2. Data shall be reported as the calculated value if the concentrations are greater than or equal to the LOQ. Calculated concentrations that are less than LOQ but greater than or equal to the LOD will be reported with the LOQ indicated in parentheses.

6.4.3. The minimum target LOD is 5 pg per analyte injected for water and 25 pg injected for sediment and tissue analysis.

6.5. Precision.

6.5.1. Precision is indicated by the reproducibility of replicate analyses. Precision will be expressed as the relative percent difference (RPD) of duplicate analyses of a split sample:

$$RPD = \frac{(dup1 - dup2)}{ave} X\ 100$$

6.5.2. The average RPD for all congeners must meet established control limits for a given matrix if measured concentrations are ≥ 5X the LOD and must be within 2x the control limits if measured concentrations are < 5X the LOD. If these

objectives are not met, duplicate samples should be re-extracted and analyzed. If no additional sample is available, these data should be flagged.

6.6. Accuracy:

6.6.1. Accuracy indicates the degree to which the analytical measurement reflects the true value of the analyte in the sample:

6.6.2. Accuracy will generally be measured using surrogate spikes and standard reference materials (SRMs). Blank spikes and matrix spikes may also be used periodically to evaulate method performance and matrix effects. A known amount of the surrogate spike is added to every sample and blank prior to extraction. Thus the recovery of every extraction can be estimated by the recovery of the surrogate spike. The recoveries of analytes from SRMs, blank spikes, and matrix spikes represent the actual analytical recovery and can be used to evaluate method performance. SRMs and matrix spikes are also used to evaluate the effect of the sample matrix on analyte recovery. For a given sample set, the average percent recovery of analytes in the SRM, blank, or matrix spike and individual surrogate spike recoveries must be within established control limits for the appropriate sample matrix. If these criteria are not met, then the data from that sample set are flagged. If surrogate spike recoveries do not meet these standards, then that sample must be re-run. If they still fail QA standards, samples should be re-extracted and analyzed. If additional sample is unavailable, then the data will be flagged.

6.7. PCB and Pesticide Identification.

6.7.1. For samples analyzed by GC-ECD, PCB congeners will be identified by retention time relative to the internal standard retention time, as determined in the calibration standard. Peaks must be within 5% of the retention time in the calibration standard to be considered a correct identification. If not, the analyst must recalibrate the instrument and reanalyze the sample. For a given sample matrix, selected analytes found in 5% of the samples may be verified for correct PCB or pesticide identification by GC-MS or by retention time on a second column, depending on the project requirements. The samples chosen for verification should include a range of concentrations.

7. CORRECTIVE ACTIONS.

7.1. Sample response(s) exceed the linear range of the system: see Section 5.3.4.

7.2. Performance standards exceed acceptance criteria: see Section 5.2.2.

7.3. Surrogate recovery exceeds acceptable limits (Section 6.6): sample(s) should be re-extracted and re-analyzed.

7.4. Holding Times: holding times of extracts will be 40 days from time of extraction for PCBs, pesticides, and industrial compounds. It is recognized, however, that required re-analyses resulting from corrective actions as described above may result in holding times being exceeded for individual samples or sample groups or other contingencies may arise that compromise holding times. In these cases, all such violations of holding times must be indicated by flagging the data and by detailing the exceedances in the case narrative accompanying the sample delivery group.

7.5. Presence of interference in elution pattern: see Section 5.3.5.

7.6. Co-elution with an internal standard: see Section 5.4.

8. REFERENCES.

8.1. Keith, L.H. et al. 1983. Principles of environmental analysis. Anal. Chem., 55, 2210-2218.

8.2. Mullin, M.D. 1985. PCB Workshop, USEPA Large lakes Research Station, Grosse, Ile, MI, June.

8.3. Test Methods for Evaluating Solid Waste (SW-846), Revision 1, November 1990, Method 8000A and 8080A.

8.4. USEPA, Quality assurance plan, Green Bay Mass Balance Study. USEPA Great Lakes National Program Office, Chicago, IL, March, 1988.

8.5. Federal Register 1984. Appendix B to Part 139. Definition and procedure for the determination of the method detection limit. Vol. 49, No. 209, October 26.

Table 1. Example GC-ECD conditions for PCB and pesticide analysis[1].

column primary: confirmation:	50 m DB-5, 0.20-mm ID, 0.33-µm film thickness or equivalent[2] 30 m DB-1701, 0.25 mm ID, 0.25µm film thickness or equivalent[2]
carrier gas	hydrogen or helium
carrier linear velocity	~2 ml/min
splitless purge flow	50 to 70 ml/min
splitless purge time	0.7 - 1.0 min
injector temperature	225±25°C
initial temperature; hold time	50°C; 1 min
oven temp.ramp	1st level - 5°C/min to 130°C 2nd level - 0.5 -1°C/min to 260°C 3rd level - 10°C/min to 280°C
final temperature; time	280°C; 10 min
ECD temperature	325±25°C
make-up gas	5% Me/95% Ar
make-up gas flow rate	30 - 40 ml/min

1 These conditions are only a guideline and may be adjusted for specific applications or particular congeners of interest.
2 An equivalent column coating is required.

APPENDIX A.

CONGENER COMPOSITION OF PERFORMANCE STANDARD FOR PCBs

Mullins, U.S. EPA Large Lakes Research Station, Grosse Ile, MI, should be cited in all publications that use this information as "Mullin, M.D., Workshop, U.S. EPA Large Lakes Research Station, Grosse Ile, MI, June 1985."

A mixed Aroclor standard composed of 250 ng/ml 1232, 180 ng/ml 1248, and 180 ng/ml 1262 will have the congener composition listed on the following pages and varying amounts of individual PCB congeners commonly added to the Aroclor mixture are also listed in italics in units of ng/ml.

Procedure No. P-16-84
Rev. 1 (3/95)

Column = DB5
Hexane Fraction
Aroclors 1232 +1248 + 1262
(25:18:18)

Relative Retention Time[1]	Amount (ng/mL)	Original Amount in Mullin's mix	Compound
0.1389	*32.6**		*1245 -TeClBz**
0.1558	*24.1*		*1234 - TeClBz*
0.2091	43		1
0.2210	*11.4*		*PeClBz*
0.2534	26		3
0.2800	2.8		4+10 (SUM)
0.3138	2.2		7
0.3285	4.2		6
0.3368	50		8+5
0.3447	*2*		*HCB*
0.3537	*2*		*PCA*
0.3574	*21*		*14 (SURR)*
0.3660	1		19
0.3796	*8*		*30 (ISTD)*
0.3950	0.52		12
0.3962	0.4		13
0.4020	13		18
0.4047	7.4		17
0.4184	0.88		24+27 (SUM)
0.4313	5.3		16
0.4326	7.8		32
0.4558	0.18		29
0.4627	2.3		26
0.4663	1		25
0.4761	19		31
0.4777	19		28
0.4922	0.15		21
0.4935	14		33
0.4956	2.7		53
0.5033	0.67		51
0.5060	11		22
0.5145	2.7		45
0.5278	1.4		46
0.5386	12		52
0.5434	0.91		43
0.5456	9		49
0.5499	5		47
0.5513	4		48

Procedure No. P-16-84
Rev. 1 (2/95)

Relative Retention Time[1]	Amount (ng/mL)	Original Amount in Mullin's mix	Compound
0.5551	*5*		*65 (SURR)*
0.5729	15		44
0.5764	4.5		37
0.5782	4.3		42
0.5922	9.4		41+71(AVE)
0.5940	6.9		64
0.6071	3.3		40
0.6198	0.5		100
0.6265	*10.5*		OCS
0.6293	0.74		63
0.6360	8.1		74
0.6432	21		70+76 (SUM)
0.6487	22		66
0.6505	5.2		95
0.6613	1.4		91
0.6776	*36*	18	56+60 (AVE)
0.6834	1.2		92
0.6864	3.1		84
0.6887	*14.6*		*o,p DDE*
0.6910	0.3		89
0.6934	4.8		101
0.7021	2.3		99
0.7134	0.18		119
0.7209	0.36		83
0.7301	1.9		97
0.7379	*5.32*	0.32	81
0.7394	3		87
0.7463	2.1		85
0.7482	*20*		*p,p DDE*
0.7506	1.4		136
0.7549	1.5		77
0.7570	5.6		110
0.7755	1.3		82
0.7803	5.7		151
0.7881	2.2		135+144 (SUM)
0.7909	0.22		147+124 (SUM)
0.7963	0.33		107
0.7997	*5*		*123*
0.8018	11		149
0.8048	3.5		118
0.8189	0.585		134
0.8231	*5.265*	0.265	114
0.8259	*10.5*		*o,p DDT*

Procedure No. P-16-84
v. 1 (3/95)

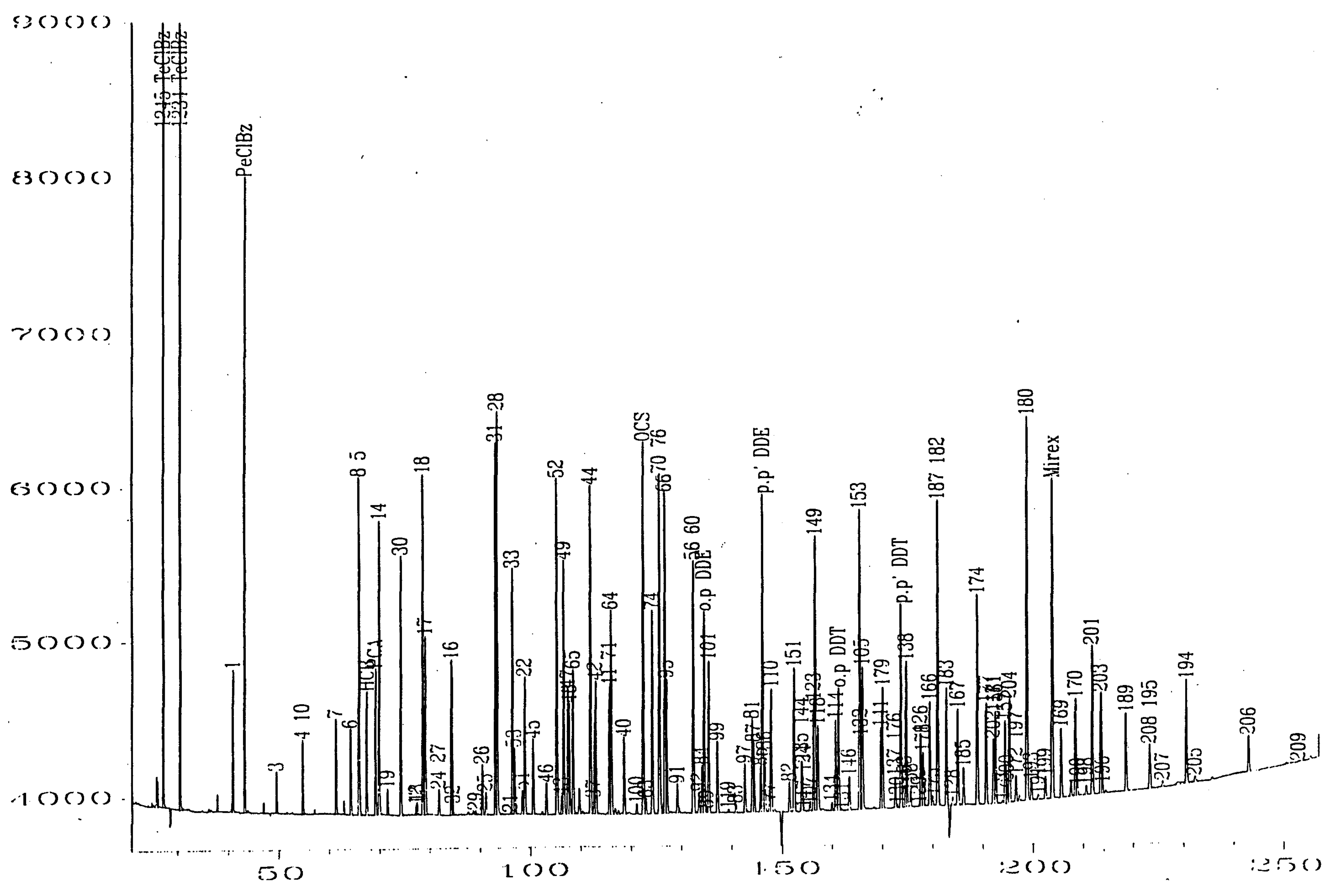
Relative Retention Time[1]	Amount (ng/mL)	Original Amount in Mullin's mix	Compound
0.8283	0.091		131
0.8369	1.6		146
0.8475	16.775		153
0.8487	2.9085		132
0.8503	*6.916*	1.916	105
0.8691	5.2		141
0.8706	*6*		*179*
0.8834	1.388		137+176 (SUM)
0.8855	0.25		130
0.8893	*21.396*		*p,p DDT*
0.8935	4.2967		163
0.8949	5.5033		138
0.8991	1.2		158
0.9082	0.3		129
0.9096	*5*		*126*
0.9119	3.4		178
0.9186	*5*		*166 (SURR)*
0.9213	0.6		175
0.9267	15		187+182 (AVE)
0.9354	7.7		183
0.9417	0.47		128
0.9471	*5.11*	0.11	167
0.9529	2.2		185
0.9669	11		174
0.9760	5.7		177
0.9834	3.69		202+171(SUM)
0.9857	*5.331*	0.331	156
0.9929	0.13		173
0.9956	*5.02*	0.02	157
0.9978	2.05		200
1.0000	*6*		*204 (ISTD)*
1.0066	2.14		172+197 (SUM)
1.0179	24		180
1.0224	1.4		193
1.0293	0.45		191
1.0366	1		199
1.0437	*20*		*Mirex*
1.0530	*5.04*	0.04	169
1.0677	9.559		170
1.0700	2.541		190
1.0786	0.67		198
1.0848	15		201
1.0934	9		203

Procedure No. P-16-84
Rev. 1 (3/95)

Relative Retention Time[1]	Amount (ng/mL)	Original Amount in Mullin's mix	Compound
1.0951	8		196
1.1193	*5.18*	0.18	189
1.1437	8.08		208+195 (SUM)
1.1568	0.48		207
1.1810	6.9		194
1:1896	0.4		205
1.2446	4.2		206
1.2971	0.095		209

[1]Relative to PCB 204, following Mullin's method

* Compounds and amounts in italics were added to the 610 mix and are not found in Mullin's Aroclor mix.



Sig. 2 in C:\HPCHEM\1\DATA\610R53__.D

APPENDIX B.

CONGENER COMPOSITION OF PERFORMANCE STANDARD FOR PESTICIDES

A mixed pesticide standard composed of 19 organochlorinated pesticides and industrial compounds will have the pesticide composition listed on the following page, in units of ng/ml.

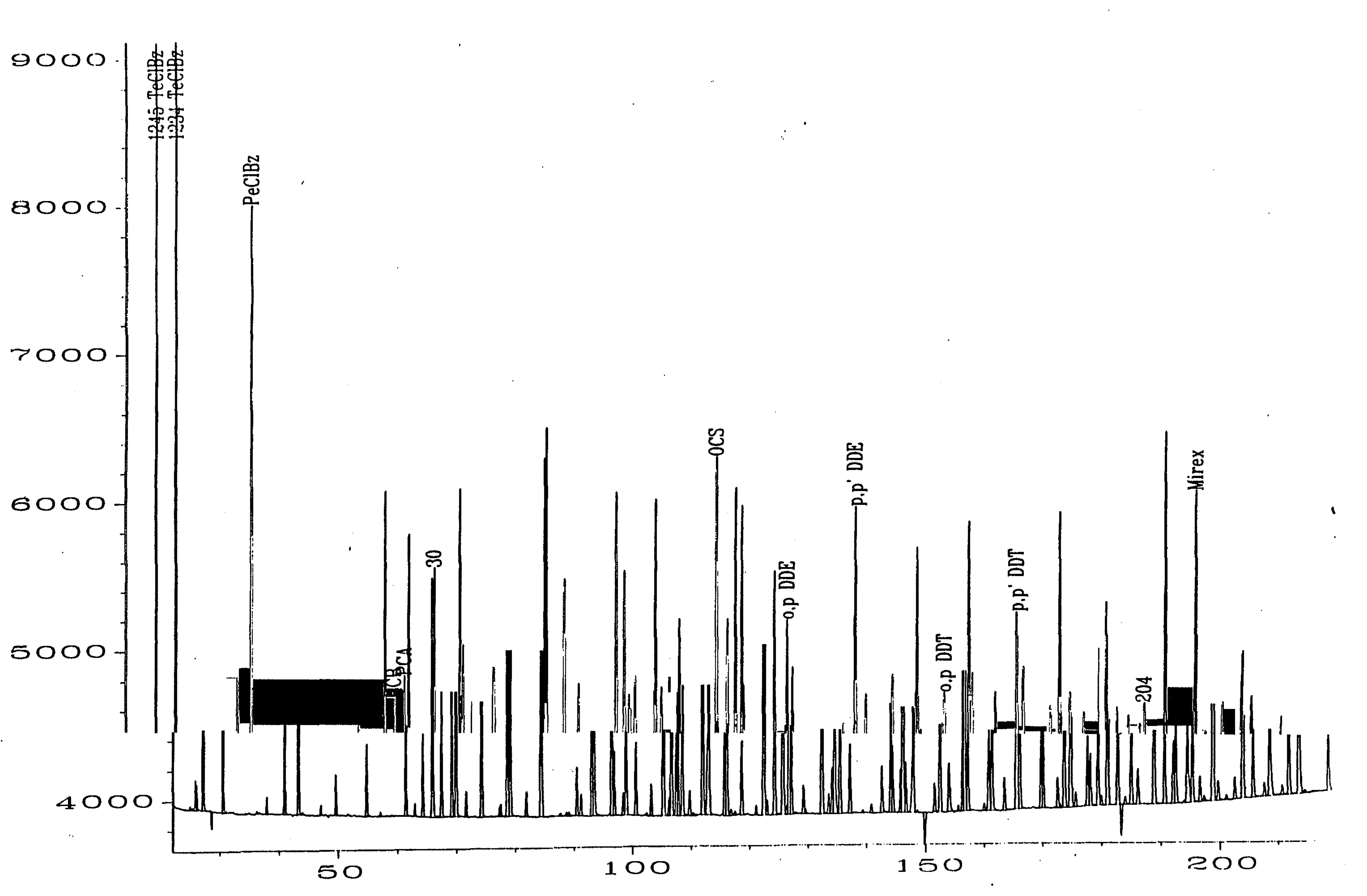
Procedure No. P-16-84
Rev.1 (3/95)

Column = DB 5

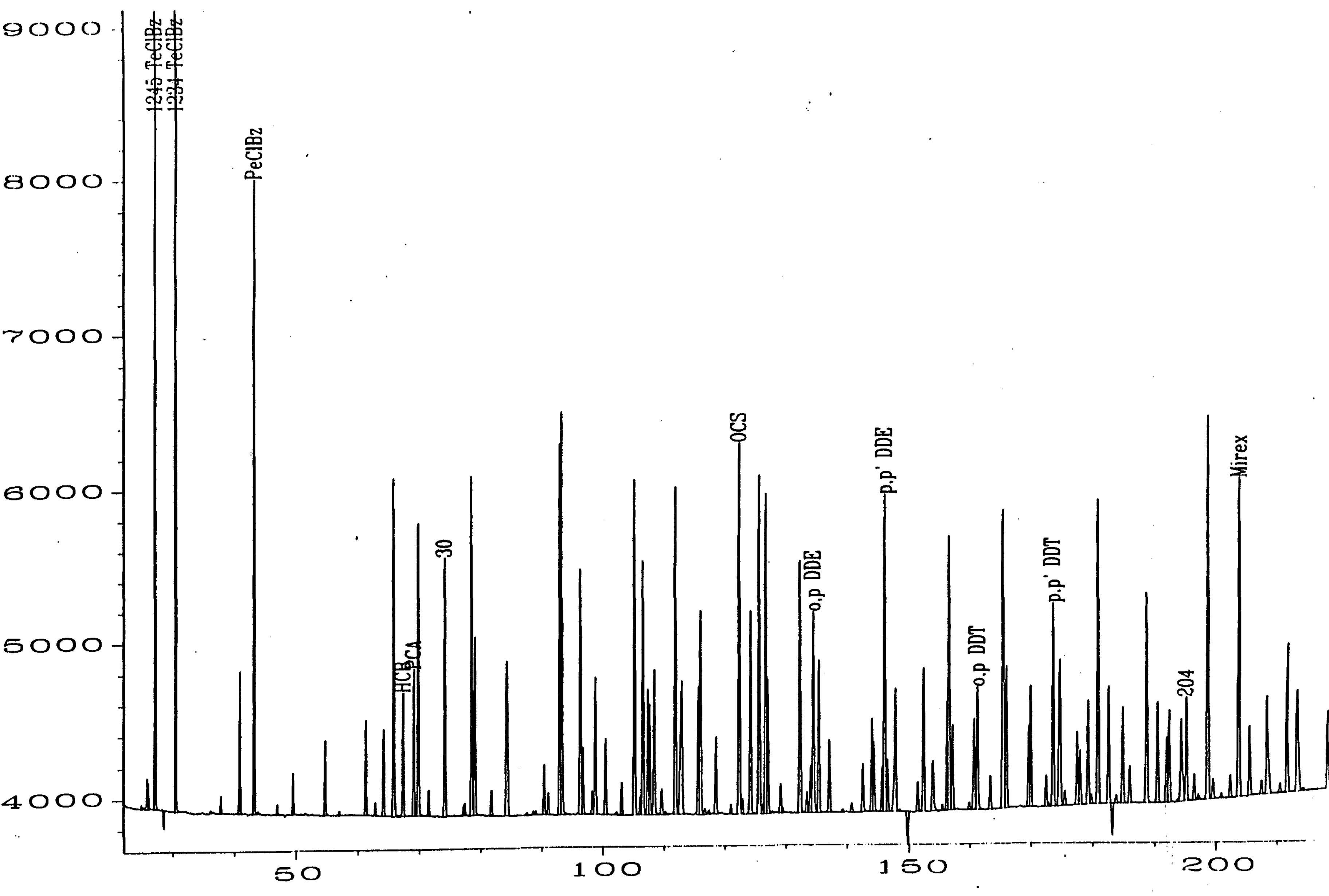
Mixed Pesticide Standard

Relative Retention Time[1]	Amount ng/mL	Compound
0.1386	32.6	1245 - TeClBz
0.1555	24.1	1234 - TeClBz
0.2204	11.4	PeClBz
0.3344	20	d-HCH (SURR)
0.3437	10	HCB
0.3529	10	PCA
0.3759	966	B-HCH
0.3789	8	30 (ISTD)
0.3829	20	a-HCH
0.4228	21.2	g-HCH
0.5123	220	Alachlor
0.5740	250	Metalchlor
0.6257	10.5	OCS
0.6886	14.6	o,p -DDE
0.7365	21	Dieldrin
0.7481	20	p,p -DDE
0.7606	18.2	o,p -DDD
0.8225	20	p,p -DDD
0.8259	10.5	o,p -DDT
0.8895	21.4	p,p -DDT
1.0000	6	204 (ISTD)
1.0440	20	Mirex

[1]Relative to PCB 204



9000
8000
7000
6000
5000
4000
1245 TeClBz
1234 TeClBz
PeClBz
HCB
PCA
30
OCS
o,p DDE
p,p' DDE
o,p DDT
p,p' DDT
204
Mirex
50
100
150
200
Sig. 2 in C:\HPCHEM\1\610R53.D



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Appendix I

Indoor Air and Soil Gas Sampling Procedures

Indoor Air and Soil Gas Sampling Procedures

I. General

This document describes the procedures to collect indoor air samples and subsurface soil gas samples from sub-slab sampling ports or soil vapor monitoring points for the analysis of: (a) volatile organic compounds (VOCs) and certain associated semi-volatile organic compounds (SVOCs) using EPA Method TO-15 (or the Method TO-15 Selected Ion Monitoring [SIM] scan); (b) other SVOCs, including polycyclic aromatic hydrocarbons [PAHs] (which cannot be analyzed by Method TO-15), using either EPA Method TO-13A (or TO-13A SIM) or EPA Method TO-17; and/or (c) polychlorinated biphenyls (PCBs) using EPA Method TO-17. Method TO-15 uses a SUMMA® passivated stainless steel canister to collect the sample. Methods TO-13A and TO-17 use a glass or stainless steel tube packed with a sorbent material; the specific sorbent material packed within each tube is selected based on the target compounds and desired reporting limits.

The procedures described herein will also be used for any ambient air samples that are collected for VOC and/or SVOC analyses for comparative purposes to the results of indoor air or soil gas samples.

For all indoor/ambient air and vapor point samples, the following cautions should be considered prior to collection of a sample:

- Field sampling personnel will have current health and safety training as required by the applicable Health and Safety Plan (HASP), be well versed in the relevant standard operating procedures (SOPs), and possess the required skills and experience necessary to successfully complete the desired field work. Personnel responsible for leading soil-gas sample collection activities must have previous soil-gas sampling experience.

- All sampling personnel should review the HASP prior to beginning work to be aware of all potential hazards associated with the job site and the specific task. Field sampling equipment must be carefully handled to minimize the potential for injury and the spread of hazardous substances.

- Sampling personnel must not handle hazardous substances (such as gasoline), use permanent marking pens (sharpies), wear/apply fragrances, or smoke cigarettes/cigars 24 hrs before and/or during the sampling event.

- Care should be taken to ensure that the appropriate sorbent is used in the TO-13A or TO- 17 tube preparation. Sorbent should be selected in consultation with the analytical laboratory and in consideration of the target compound list, the necessary reporting limits and the expected range of concentrations in field samples. The expected range of concentrations in field samples may be estimated from previous site data, release history and professional judgment informed by the conceptual site model.

- Flow rates for sample collection with TO-13A or TO-17 sorbent tubes should be determined well in advance of field work in consultation with the laboratory.

- A shipping determination must be performed by DOT-trained personnel for all environmental samples that are to be shipped, as well as some types of environmental equipment/supplies that are to be shipped.

- At the sampling location, keep the tubes in their storage and transportation container to equilibrate with ambient temperature prior to attaching to the sample train.

- Always use clean gloves when handling sampling tubes.

- Seal clean, blank sorbent tubes and sampled tubes using inert, Swagelok®-type fittings and Teflon® PTFE (polytetrafluoroethylene) compression fittings. Wrap capped tubes individually in uncoated aluminum foil. Use clean, sealable glass jars or metal cans containing a small packet of activated charcoal or activated charcoal/silica gel for storage and transportation of multiple tubes. This activated charcoal is not analyzed, but serves as a protection for the analytical sorbent tube. Store the multi-tube storage container in a clean environment at 4ºC.

- Keep the sample tubes inside the storage container during transportation and only remove them at the monitoring location after the tubes have reached ambient temperature. Keep out of direct sunlight if possible. Store sampled tubes in a refrigerator at 4ºC inside the multi-tube container until ready for analysis.

- The purge flow rate of 100 ml/min should be suitable for a variety of silt and sand conditions but will not be achievable in some clays without excessive vacuum. A low vacuum (<10” of mercury) should be maintained. Record the measured flow rate and vacuum pressure during sample collection. The cutoff value for vacuum differs in the literature from 10” of water column (ITRC 2007) to 136” of water column or 10” of mercury (http://www.dtsc.ca.gov/lawsregspolicies/policies/SiteCleanup/upload/SMBR_ADV_activesoilgasinvst.pdf). A detailed discussion of the achievable flow rates in various permeability materials can be found in Nicholson (2007). Related issues of contaminant partitioning are summarized in ASTM D5314- 92. Passive sampling approaches can be considered as an alternative for clay soils. However, most passive sampling approaches are not currently capable of quantitative estimation of soil gas concentration.

- Record the canister ID number, flow controller ID number, canister pressure, and start and stop times on a proper field sampling form. Observe and record the time/pressure at a mid-point in the sample duration. It is a good practice to lightly tap the analog pressure gauge with your finger before reading it to make sure it isn’t stuck.

- Ensure that there is a minimum vacuum of 6” inHg in the SUMMA® after sampling. Sometimes the analog gauges sent from labs have offset errors, or they stick.

- If possible, have equipment shipped two or three days before the sampling date so that all materials can be checked. Order replacements if needed.

- Requesting extra canisters and extra sorbent tubes from the laboratory should also be considered to ensure that you have enough equipment on site in case of an equipment failure.

- Shallow exterior soil-gas sampling should not proceed if rain in anticipated during the sampling event or within 5 days following a significant rain event (1/2-inch of rainfall or more).

The following sections provide a list of the necessary equipment and detailed instructions for the collection of indoor/ambient air or soil vapor samples for TO-15 (VOCs), TO-13A (SVOCs) and TO-17 (PCBs or SVOCs) analysis.

II. Equipment List

- Appropriate personal protective equipment (PPE), as provided in the applicable HASP.

- If applicable, TO-13A and/or TO-17 tubes, and sample flow rate calibration tubes (provided by the laboratory) (specific sorbents will be recommended by the laboratory considering the target compound list and the necessary reporting limits);

- Stainless steel SUMMA® canisters (1-liter, 3-liter, or 6-liter; order at least 5% extra, if feasible) (batch certified canisters or individually certified canisters as required by the project);

- Flow controllers with in-line particulate filters and vacuum gauges, which will be pre-calibrated to a specified sample duration (e.g., 30 minutes, 8 hours, 24 hours) or flow rate (e.g., 200 milliliters per minute [mL/min]) (confirm with the laboratory that the flow controller comes with an in-line particulate filter and pressure gauge [order at least 5% extra, if feasible]; flow rate should be selected based on expected soil type [see below]);

- Decontaminated Swagelok® type stainless-steel or comparable two-way ball or needle valve (sized to match sample tubing) with T-fitting union attached (Figure I-1);

- 1/4-inch outer diameter (OD) tubing (Teflon® or Teflon®-lined polyethylene);

- Swagelok® type stainless steel (male/male) straight fitting union (1/4-inch);

- Teflon® PTFE (polytetrafluoroethylene) and stainless steel or comparable Swagelok® or equivalent compression fittings for 1/4-inch OD tubing or compression fittings for 3/8-inch OD tubing if sampling permanent soil vapor point where 3/8-inch tubing was used;

- Swagelok® type stainless steel four-way fitting (if sample train will be assembled with an inline vacuum gauge;
- Swagelok® type stainless steel duplicate "T" fittings unions;
- Two (2) portable vacuum pumps (e.g., Gilian or SKC Low Flow Module) capable of producing very low flow rates (e.g., 10 to 200 mL/min) with low flow calibration valve;
- Two (2) 60cc syringes for low purge volumes;
- Vacuum gauge;
- Rotameter or an electric flow sensor if vacuum pump does not have a flow gauge (e.g., BIOS Dry Cal DC-Lite "L" or equivalent);
- Tracer gas testing supplies (refer to Appendix NN SOP);
- Photoionization detector (PID) capable of detecting parts per billion (e.g., ppb RAE);
- Appropriate-sized open-end wrench (typically 9/16-inch, 1/2-inch, and 3/4-inch);
- Two (2) Tedlar bags for purge gasses;
- Portable weather meter capable of temperature, wind speed, humidity, and barometric pressure (e.g., Kestrel or similar);
- Box, chair, tripod, or similar item to hold canister above the ground surface (although, if necessary, canisters can be hung using zip-ties or rope on a fence or tree);
- Extra ¼"Teflon® lined sample tubing;
- Nitrile gloves;
- Chain-of-custody (COC) form;
- Sample collection log;
- Camera (if site allows);
- Gel ice (for TO-13A or TO-17 samples); and
- Field notebook.

See Appendix NN for Tracer Gas equipment (if applicable)

III. Indoor Air Sampling Procedures for EPA Method TO-15

Initial Building Survey for Indoor Air Samples (if applicable to project)

1. Complete the appropriate building survey form and product inventory form (Attachment I-2) as necessary 72 to 48 hours in advance of sample collection.

2. Survey the area for the apparent presence of items or materials that may potentially produce or emit constituents of concern and interfere with analytical laboratory analysis of the collected sample. Record relevant information on survey form and document with photographs.

3. Record date, time, location, and other relevant notes on the inventory form.

4. Items or materials that contain constituents of concern and/or exhibit elevated PID readings shall be considered probable background sources of VOCs. Request approval of the owner or occupant to have these items removed to a structure not attached to the target structure at least 48 hours prior to sampling if possible.

5. Set a date and time with the owner or occupant to return for placement of SUMMA® canisters.

Preparation of SUMMA®-Type Canister and Collection of Sample

1. Record the following information on the sampling form (use a hand-held weather meter, contact the local airport or other suitable information source [e.g., weatherunderground.com] to obtain the following information):

   - ambient temperature;

   - barometric pressure;

   - wind speed; and

   - relative humidity.

2. Choose the sample location in accordance with the sampling plan. If a breathing zone sample is required, place the canister on a ladder, tripod, box, or other similar stand to locate the canister orifice 3 to 5 feet above ground or floor surface. If the canister will not be overseen for the entire sampling period, secure the canisters as appropriate (e.g., lock and chain). Canister may be affixed to wall/ceiling support with nylon rope or placed on a stable surface. In general, areas near windows, doors, air supply vents, and/or other potential sources of "drafts" shall be avoided.

3. Record SUMMA® canister serial number and flow controller number on the sampling log and chain of custody (COC) form. Assign sample identification on canister ID tag, and record on the sample collection log (Attachment I-1d), and COC form.

4. Remove the brass or stainless steel cap from the SUMMA® canister and connect an independent digital pressure gauge (if available) making sure all connections are secured and tight, open the canister valve for 1 second and then shut the valve. Record the pressure on the field form, and disconnect the digital gauge. If the initial vacuum pressure registered is not above -26 inches of Hg, then the SUMMA® canister is not appropriate for use and another canister should be used. Attach the flow controller with in-line particulate filter and vacuum gauge to the SUMMA® canister with the appropriate-sized wrench. Tighten with fingers first, then gently with the wrench. Use caution not to over tighten fittings.

5. Open the SUMMA® canister valve to initiate sample collection. Record the date and local time (24-hour basis) of valve opening on the sample collection log, and COC form. Collection of duplicate samples will include collecting two samples side by side at the same time.

6. Record the initial analog vacuum in the SUMMA® canister on the sample log and COC form. If the initial analog vacuum registers less than 26 inches of Hg and the digital gauge read significantly higher, the flow regulator is not appropriate for use and another regulator should be used.

7. Take a photograph of the SUMMA® canister and surrounding area, if possible.

8. Check the SUMMA® canister approximately half way through the sample duration and note conditions as progress on sample logs.

Termination of Sample Collection

1. Arrive at the SUMMA® canister location at least 1-2 hours prior to the end of the sampling period (e.g., 8-hour, 24-hour).

2. Stop collecting the sample when the canister vacuum reaches approximately 6 inches of Hg (leaving some vacuum in the canister provides a way to verify if the canister leaks before it reaches the laboratory) or when the desired sample time has elapsed.

3. Record the final canister vacuum. Stop collecting the sample by closing the SUMMA® canister valve. Record the date, local time (24-hour basis) of valve closing on the sample collection log, and COC form.

4. Remove the particulate filter and flow controller from the SUMMA® canister, reinstall brass cap on canister fitting, and tighten with wrench.

5. Package the canister and flow controller in the shipping container supplied by the laboratory for return shipment to the laboratory. The SUMMA® canister does not require preservation with ice or refrigeration during shipment.

6. Complete the appropriate forms and sample labels as directed by the laboratory (e.g., affix card with string).

7. Complete COC form and place requisite copies in shipping container. Close shipping container and affix custody seal to container closure. Ship to laboratory via overnight carrier (e.g., Federal Express) for analysis.

IV. Soil Gas Sampling Procedures under EPA Methods TO-15, TO-13A, and TO-17

The TO-15 method uses 1-liter 3-liter, or 6-liter SUMMA® passivated stainless steel canisters. An evacuated 6-liter SUMMA® canister (less than 28 inches of mercury [Hg]) will provide a recoverable whole-gas sample of approximately 5 liters when allowed to fill to a vacuum of approximately 6 inches of Hg. A 1L canister will fill to approximately 800 mL and a 3L canister will fill to approximately 2400 mL when the gauge reaches 6 inches Hg. The whole-air sample will then be analyzed for VOCs using a quadrupole or ion-trap gas chromatograph/mass spectrometer (GS/MS) system to provide compound detection limits of 0.5 parts per billion volume (ppb). Optionally the canister sample can also be analyzed for fixed gasses such as helium, methane, carbon dioxide, and oxygen by ASTM Method D-1946. Following sample collection, the canister will be sent to the laboratory where the sample will be analyzed for the target compounds. The following sections provide detailed instructions for the collection of soil gas samples for analysis using Methods TO-15, TO-13A, and TO-17.

Selection of Sorbent and Sampling Volume (TO-13A/TO-17 Methods) to be Completed Prior to Sampling Event

1. Identify the necessary final reporting limit for the target compound(s) in accordance with the project quality assurance plan and/or in consultation with the data end user.

2. Identify the necessary method reporting limit(s). The laboratory will be helpful in providing this information as it is typically specific to the sensitivity of the instrumentation and the media used.

3. The minimum sampling volume is the volume of soil-gas sample that must be drawn through the sorbent in order to achieve the desired final reporting limit. Calculate the minimum sampling volume using the following equation:

$$\text{Minimum Sampling Volume (L)} = \frac{\text{Final Reporting Limit } (\mu g)}{\text{Action Level } (\mu g/m^3)} \times \frac{1000 \text{ L}}{m^3}$$

Where:

L = liters

µg = microgram

m = meter

4. If a timed sample duration is specified in the work plan, calculate the minimum flow rate. The minimum flow rate is the flow rate necessary to achieve the minimum sampling volume using the following formula:

$$\text{Minimum Flow Rate (L/min)} = \frac{\text{Minimum Sampling Volume (L)}}{\text{Sample Duration (min)}}$$

Where:

min = minutes

Then compare the minimum flow rate calculated to the requirements for maximum soil gas sampling without excessive danger of short circuiting, normally stated as 0.2 liters/minute, although it can be lower in tight soils. Soil vapor sampling flow rates should not exceed 200 ml/min.

5. Compare the minimum sampling volume to the safe sampling volume (SSV) for the sorbents selected. SSV for specific sorbents can be provided by the manufacture or the laboratory, being used. Ensure that the compounds will not breakthrough when sampling the volume calculated above.

Calibrate the Sample Pump Prior to Assembly of Sampling Train (For TO-13A/TO-17 Only)

- Attach the electric flow sensor using silicone tubing to the purge pump.

- If sampling using Method TO-13A or TO-17: connect the flow rate calibration tube provided by the laboratory to a PTFE compression fitting and tightening nut. Attach the tube to one side of a male/male Swagelok® fitting. On the other side of the fitting, connect a 0.5-foot piece of Teflon® or Teflon®-lined tubing using a Swagelok® or equivalent stainless steel compression fitting and tightening nut. The TO-13A sample flow rate calibration tube should be clearly marked by the laboratory with an arrow indicating flow direction (or as otherwise specified by the laboratory); if not, confirm with laboratory. Connect the tubing section, added to the calibration tube, to the “IN” port of electric flow sensor using a tight fitting silicone connection. Attach the “OUT” port of the electric flow sensor to the vacuum side of the purge pump.

- Turn on the sample pump and adjust the flow rate on the sample pump to achieve the desired minimum flow rate (calculated above) as measured by the electric flow sensor. A low flow calibration tube may be required when the desired purge rate is below 200 mL/min. Follow the same applicable steps if using a syringe to purge the point.

- If using multiple pumps repeat the steps above until each sampling pump has been properly calibrated to its appropriate flow rate.

- Keep this calibration assembly together as it will be used again during the "Purge Sampling Assembly Prior to Collection of TO-13A/TO-17 and TO-15 Samples" section below.

Assembly of Sampling Train

- Record the following information on the appropriate field form (Attachment I-1a, I-1b, or I-1c) using the portable weather unit. If weather unit is unavailable contact the local airport or other suitable information source [e.g., site-specific measurements, weatherunderground.com] to obtain the information:

  a. wind speed and direction;

  b. ambient temperature;

  c. barometric pressure; and

  d. relative humidity.

- If samples are being collected from soil vapor points with approximately two feet of sample tubing already attached to the vapor point(s), remove the cap or plug from the sample tubing and proceed to the next step. If there is less than two feet of tubing connected to the vapor point or when collecting samples from a permanent flush sub-slab port, remove the cap or plug from the sampling port. Connect a two foot piece of Teflon® or Teflon®-lined tubing to the sampling port using a Swagelok® or equivalent stainless steel compression fitting. If connecting to an existing piece of tubing and there is a need to extend the sample train, use a female compression fitting on the sample tubing already connected to the vapor point and a male compression adapter on the added tubing to secure the two lengths of tubing together.

- For newly installed locations follow tracer gas testing (see Appendix NN) prior to proceeding to the next step.

- With the tubing line connected to the vapor point, connect the opposite end to one side of a stainless steel T-fitting (Figure I-1). On each one of the other two sides, add approximately 0.5 to 1-foot of the Teflon® or Teflon®-lined tubing using a Swagelok® or equivalent stainless-steel or comparable compression fitting.

- Prior to connecting sample tubing to the canister, remove the brass or stainless steel cap from the SUMMA® canister and connect a digital pressure gauge (if available). Make sure all connections are secured and tight, open the canister valve for 1 second and then shut the valve. Record the pressure on the field form, and disconnect the digital gauge. If the initial pressure registered is not between -30 and -25 inches of Hg, then the SUMMA® canister is not appropriate for use and another canister should be used.

- Connect the flow controller with in-line particulate filter and analog vacuum gauge to the SUMMA® canister. Do not open the valve on the SUMMA® canister. Record on the field form and COC form the flow controller number along with the appropriate SUMMA® canister number.

- Connect the open end of the flow controller to the bottom side of the T-fitting assembly using a stainless steel compression fitting and tightening nut. Note, attach the sample tubing from the collection point on one side and the 0.5 to 1 foot section of Teflon® or Teflon®-lined tubing on the other.

- From the remaining 0.5 to 1 foot section of Teflon® or Teflon®-lined tubing section from the T-fitting, using a Swagelok® or equivalent stainless steel compression fitting and tightening nut, attach the tubing to one side of a Swagelok® or equivalent stainless steel two-way valve. Close the valve.

- On the opposite side of the two-way valve, using a Swagelok® or equivalent stainless steel compression fitting, attach a 0.5-foot length of Teflon® or Teflon®-lined tubing. Make sure to check all fittings and hand tighten all connections. Confirm that the sampling train is assembled as shown on Figure I-1. The TO-15 leg of the combined sampling train is now complete. Sample collection procedures for TO-13A and TO-17 are shown below in the section titled, "TO-13A/TO-17 Sample Collection".

Purge Sampling Assembly Prior to Collection of TO-15 Samples Only

1. If performing sampling using Method TO-15 only and using a purge pump with an electronic flow sensor, connect the vacuum side of flow sensor using tight-fitting silicone tubing or Swagelok® fitting to the tubing connected to the two-way valve. If using a syringe, connect the tip of the syringe using tight-fitting silicone tubing or Swagelok® fitting to the tubing connected to the two-way valve. If collecting an indoor or ambient air sample simultaneously, all purged air must be collected in a tedlar bag and emitted outside and/or down gradient from the indoor/ambient air location.

2. Calculate the purge volume required at each sampling point. Using the formula of a cylinder ($V = \pi r^2 \cdot h$) add the volume of air in the vapor point and the sampling train together, then multiply by 3. This is the volume to be removed prior to sample collection.

3. Open the two-way valve and begin purging three volumes of air from the vapor probe and sampling line. When using a purge pump, confirm that the flow rate is the same as calibrated previously, and make adjustments as necessary to the low flow regulator connected to the pump. Measure organic vapor levels with a PID in the Tedlar bag or effluent line. Lower flow rates may be necessary in silt or clay to avoid excessive vacuum. The vacuum reading shown on the Summa® canister or supplemental gauge should not be greater than 10 inHg.

Purge Sampling Assembly Prior to Collection of TO-13A/TO-17 and TO-15 Samples

1. If performing sampling using Method TO-13A and/or TO-17, as well as potentially TO-15, and using a purge pump with an electronic flow sensor, attach a Swagelok® or equivalent stainless steel compression fitting and tightening nut to the tubing connected from the two-way valve. Using the Teflon® compression fitting attached to the flow calibration tube, connect the calibration tube and Teflon® or Teflon®-lined tubing section using a straight fitting Swagelok® union.

2. The remaining set up should be completed as identified in the previous section, "Calibrate the Sample Pump Prior to Assembly of Sampling Train (For TO-13A/TO-17 Only)." Make sure to check all fittings and hand tighten all connections.

3. Connect the vacuum side of flow sensor using tight-fitting silicone tubing or Swagelok® fitting to the tubing connected to calibration tube. If using a syringe, connect the tip of the syringe using tight-fitting silicone tubing.

4. Calculate the purge volume required at each sampling point. Using the formula of a cylinder ($V = \pi r^2 \cdot h$) add the volume of air in the vapor point and the sampling train together, then multiply by 3. This is the volume to be removed prior to sample collection.

5. If collecting an indoor or ambient air sample simultaneously, all purged air must be collected in a tedlar bag and emitted outside and/or downgradient from the indoor/ambient air location. Open the two-way valve and begin purging three volumes of air from the vapor probe and sampling line. When using a purge pump, confirm that the flow rate is the same as calibrated previously. If needed make adjustments as necessary to the connected pump. Measure organic vapor levels with a PID from the air within the Tedlar bag or effluent line. Lower flow rates may be necessary in silt or clay to avoid excessive vacuum. Vacuum reading shown on the SUMMA® canister or supplemental gauge, should not be greater than 10 inHg.

6. If performing tracer gas evaluation, check the seals established around the soil vapor probe and the sampling train fittings. See Appendix NN.

7. When three volumes of air have been purged from the vapor probe and sampling line, stop the purge pump and close the two way valve. Document all flow measurements and calculated volumes removed as well as any PID readings noted during purge. Sampling can now begin.

TO-15 Sample Collection

1. Open the SUMMA® canister valve to initiate sample collection. Record on the sample log (attached) the time sampling began and the canister vacuum.

2. Take a photograph of the SUMMA® canister and surrounding area (unless photography is restricted by the property owner).

3. If using a timed sample, check the SUMMA® canister approximately half way through the sample duration and note vacuum readings on sample logs. If canister draws too quickly, confer with the project manager on whether the sample needs to be re-collected.

TO-15 Sample Termination

1. If sample is timed, arrive at the SUMMA® canister location at least 30 to 60 minutes prior to the end of the sampling interval.

2. Record the final vacuum. Stop collecting the sample by closing the SUMMA® canister valves. The canister should have a minimum amount of vacuum (approximately 6 inches of Hg or slightly greater).

3. Record the date and time of valve closing in the field notebook, sample collection log, and COC form.

TO-13A/TO-17 Sample Collection

1. Record on the field form and COC form the tube number located on the tube. Note: TO-13A tubes sometimes do not have ID numbers but should be placed in a resalable bag and labeled with the vapor point location. Ensure that TO-13A samples are collected before TO-17 samples (if collecting both).

2. Confirm that the two-way valve is closed. Replace the flow calibration tube with the sample tube, confirming flow direction and tightening down all fittings. Make sure to use a Teflon® compression fitting, not stainless steel, and do not over tighten connections, which could cause the tube to crack or leak.

3. Open the valve in line with the tube; turn on the sample pump to begin sample collection. If necessary adjust the flow rate using the low flow regulation on the pump. Use a timer to ensure accuracy in pumping time. Record on the field sample log the time sampling began and the flow rate from the sample pump.

TO-13A/TO-17 Termination of Sample Collection

1. If preforming a tracer test, measure the helium concentration in the soil-gas purged out of the effluent line when sampling outside or from the Tedlar bag when collecting samples inside. Document this value on the field form.

2. Record the final flow rate. The post-sampling flow rate should match within 10% of the pre-sample flow rate. Average the pre-sampling and post-sampling flow rate and record on the sample collection log. Stop the sample pump after the desired volume of soil-gas has passed through the sorbent, and close the two-way valve next to the sample tubes.

3. Record the stop time, and calculate the sample volume using the average of the pre-sample and post sample flow rate. Record the sample volume on the sample collection log, and on the COC.

4. Ensure that the two-way value is closed and disconnect the sorbent tube from the sampling train and immediately cap both ends of the tube. Do not over tighten connections, which could cause the tube to crack or leak. If multiple tube samples are to be collected, replace the tube with the next sample tube, confirming flow direction and tightening down all fittings. Complete sample collection as described in the steps 1 to 3 shown above in the prior section, "TO-13A/TO-17 Sample Collection."

5. Package the tubes according to laboratory protocol on gel ice and ship to the laboratory for analysis.

V. Waste Management

The waste materials generated during sampling activities should be minimal. PPE, such as gloves and other disposable equipment (i.e., tubing), will be collected by field personnel for proper disposal.

VI. Data Recording and Management

Measurements will be recorded on the field form at the time of measurement with notations of the project name, sample date, sample start and finish time, sample location (e.g., coordinates, distance from permanent structure), tube type and number and sample volume. Field sampling logs and COC records will be transmitted to the Project Manager.

VII. Quality Assurance and Quality Control

EPA Methods TO-13A, TO-15, and TO-17 specify the minimum quality assurance/quality control (QA/QC) requirements for implementation of those methods, including frequency requirements for duplicates, distributed volume pairs, field blanks, lab blanks, etc. These requirements are summarized below.

Field duplicates and distributed volume pairs assess precision for the sampling of soil gas. Field duplicates should be collected for one out of every 20 samples or one per sampling event, whichever is more frequent, unless otherwise provided in the project-specific work plan. Field duplicate soil gas

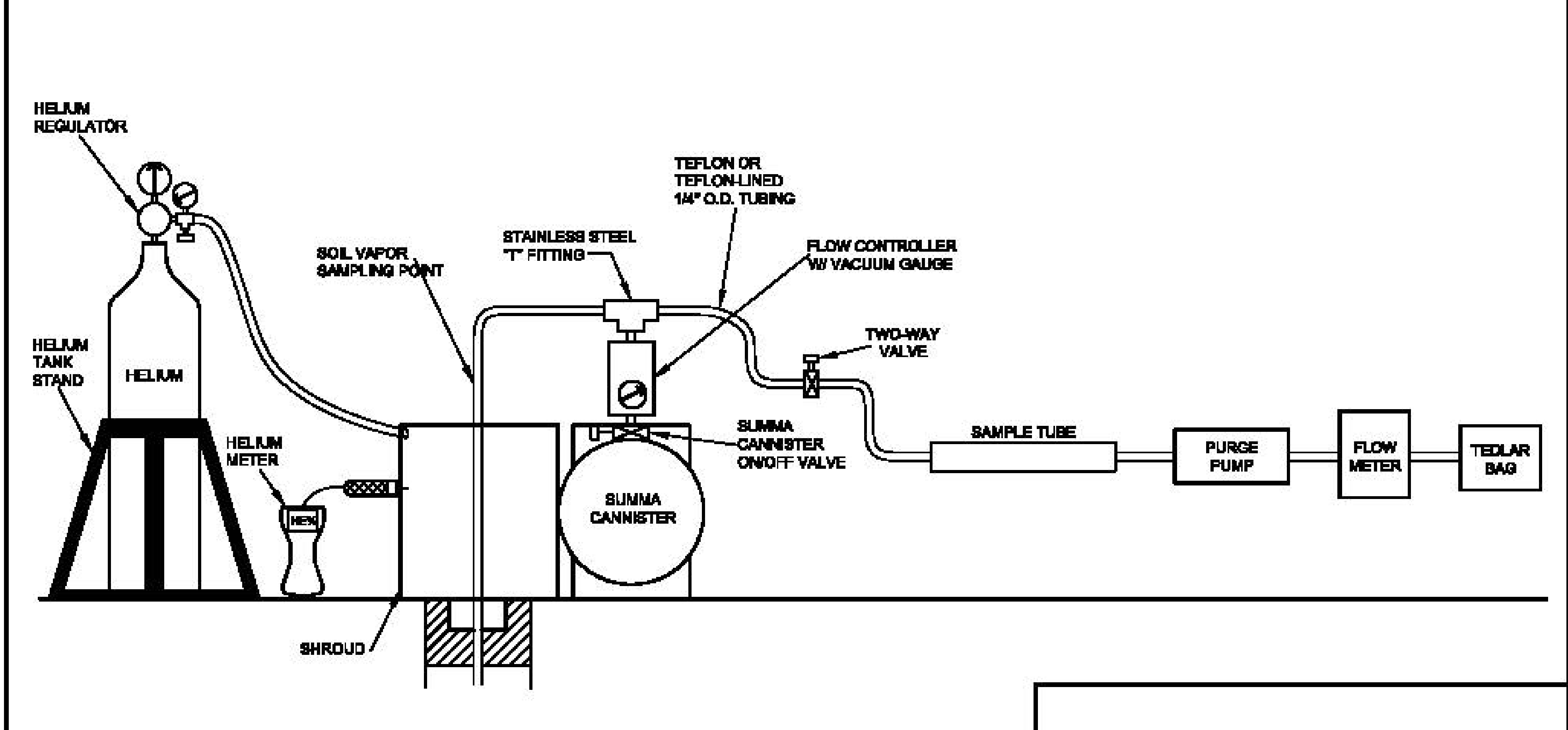
samples should be collected via a split sample train, allowing the primary and duplicate sample to be collected from the soil gas probe simultaneously. EPA Method TO-17 advises the collection of distributed volume pairs for every sample collected. The project-specific work plan will address whether distributed volume pairs will be collected.

Equipment blanks assess potential sample contamination resulting from the transportation and storage of samples, and are required for EPA Methods TO-13A and TO-17. The equipment blank sample containers go through every step of the cleaning and transportation process with the exception of sampling. The equipment blanks are then analyzed to determine if any contamination occurred during the process. For EPA Method TO-13A, a minimum of one equipment blank is required out of every 20 samples or one per sampling event, whichever is more frequent. For EPA Method TO-17, two equipment blanks are required per sampling event. EPA Method TO-15 does not require an equipment blank; however, the sample canisters must be certified clean by the laboratory and documentation of the cleaning must be provided.

The laboratory QA/QC requirements for calibrations, method blanks, laboratory control samples, laboratory duplicates, and internal standards are presented in Table 4 of the main Field Sampling Plan/Quality Assurance Project Plan (FSP/QAPP), and QC limits for laboratory control sample analyses are presented in Table 5 of the main FSP/QAPP. .

Additional QA/QC measures may be proposed in the project-specific work plan. Any project-specific QA/QC measures must be at least at stringent as the EPA method requirements unless otherwise approved by EPA.

Figure 1 – Soil Vapor Sampling Equipment Arrangement



SOIL VAPOR SAMPLING EQUIPMENT ARRANGEMENT

ARCADIS

FIGURE

1

Attachment I-1a

Soil Gas Sample Collection Log (TO-15)

Soil Gas Sample Collection Log

Sample ID:

Client:		**Date/Day:**	/ /
Project:		**Weather:**	
Location:		**Temperature:**	
Project #:		**Wind Speed/Direction:**	(ft/min) / (mph)
Samplers:		**Subcontractor:**	
Logged By:		**Equipment:**	
Background PID Ambient Air Reading:	ppb	**Moisture Content of Sampling Zone (circle one):**	Dry / Moist
Sampling Depth:	' – '		
Probe (circle one):	Permanent / Temporary	**Approximate Volume of Sampling Train::**	____ mL= (___' of ____" ID tubing)
Time of Collection:	Start: Finish:	**Approximate Purge Volume:**	_____ mL= [(____) * (3v)]

Nearby Groundwater Monitoring Wells/Water Levels:

Well ID	Depth to Groundwater (feet)

SUMMA Canister Information

Size (circle one): 1 L 3 L 6 L Other: ___L

Canister ID: ____________________

Flow Controller ID: ____________________

Tracer Gas Information (if applicable)

Tracer Gas: ____________________

Canister Pressure (inches Hg):

Reported By Laboratory	Measured Prior to Sample Collection	Measured Following Sample Collection
-	Analog: - / Digital: -	Analog: - / Digital: -

Tracer Gas Concentration (if applicable):

Measured from Soil Vapor Tubing		Measured in 'Concentrated' Area		
Post Purge	**Post Sample**	**Prior to Purging**	**Post Purging**	**Post Sampling**
ppm	ppm	%	%	%

General Observations/Notes:

Photo ID:	ppb reading on the PID following sample
Analog reading (-) after ( ) hrs.	Collection from soil vapor tubing.
Analog reading (-) after ( ) hrs.	
Differential Pressure:	

Approximating One-Well Volume (for purging temporary points):
Each foot of ¼-inch tubing will have a volume of approximately 10 mL.

Attachment I-1b

Soil Gas Sample Collection Log
(TO-15 DUP)

Soil Gas Sample Collection Log

Sample ID: / DUP-___-___-___

Client:		**Date/Day:**	/ /
Project:		**Weather:**	
Location:		**Temperature:**	
Project #:		**Wind Speed/Direction:**	(ft/min) / (mph)
Samplers:		**Subcontractor:**	
Logged By:		**Equipment:**	
Background PID Ambient Air Reading:	ppb	**Moisture Content of Sampling Zone (circle one):**	Dry / Moist
Sampling Depth:	’ – ’		
Probe (circle one):	Permanent / Temporary	**Approximate Volume of Sampling Train::**	____ mL= (___’ of ____” ID tubing)
Time of Collection:	Start: Finish:	**Approximate Purge Volume:**	_____ mL= [(____) * (3v)]

Nearby Groundwater Monitoring Wells/Water Levels:

Well ID	Depth to Groundwater (feet)

SUMMA Canister Information

Size (circle one): **1 L** **3 L** **6 L** **Other: ___L**

Sample / DUP

Canister ID: __________/__________

Flow Controller ID: __________/__________

Tracer Gas Information (if applicable)

Tracer Gas: ____________________

Canister Pressure (inches Hg):		
Reported By Laboratory	**Measured Prior to Sample Collection**	**Measured Following Sample Collection**
(Sample) (DUP) - -	(Sample) (DUP) Analog: - Analog: - Digital: - Digital: -	(Sample) (DUP) Analog: - Analog: - Digital: - Digital: -

Tracer Gas Concentration (if applicable):				
Measured from Soil Vapor Tubing		**Measured in ‘Concentrated’ Area**		
Post Purge	**Post Sample**	**Prior to Purging**	**Post Purging**	**Post Sampling**
ppm	ppm	%	%	%

General Observations/Notes:

Photo ID:	ppb reading on the PID following sample
Analog reading (-) after ( ) hrs.	Collection from soil vapor tubing.
Analog reading (-) after ( ) hrs.	
Differential Pressure:	

Approximating One-Well Volume (for purging temporary points):
Each foot of ¼-inch tubing will have a volume of approximately 10 mL.

Attachment I-1c

Soil Gas Sample Collection Log
(TO-15, 17, 13a)

Soil Gas Sample Collection Log

Sample ID:

Client:		**Date/Day:**	/ /
Project:		**Barometer:**	Start: Stop:
Location:		**Temperature/Humidity:**	Start: *F / % Stop: *F / %
Project #:		**Wind Speed/Direction:**	(mph)
Samplers:		**Subcontractor:**	NA
Logged By:		**Equipment:**	PPB RAE/Helium Detector
Background PID Ambient Air Reading:	ppb	**Moisture Content of Sampling Zone (circle one):**	Dry / Moist
Sampling Depth:	' _ '		
Probe (circle one):	Permanent / Temporary	**Approximate air volume in probe and sampling line:**	mL= (' of ¼" ID tubing) + (11mL in probe)
SUMMA Time of Collection:	Start: Finish:	**Approximate Purge Volume:**	mL= [( ) * (3v)]

Nearby Groundwater Monitoring Wells/Water Levels:

Well ID	Depth to Groundwater (feet)

SUMMA Canister Information

Size (circle one): **1 L** **3 L** **6 L** **Other: ___L**

Canister ID: ____________________

Flow Controller ID: ____________________

Tracer Gas Information (if applicable)

Tracer Gas: Helium

Canister Pressure (inches Hg):

Tracer Gas Concentration (if applicable):

Measured from Soil Vapor Tubing		Measured in 'Concentrated' Area		
Post Purge	**Post Sample**	Analog: - **Prior to Purging**	**Post Purging** Anal -	**Post Sampling**
ppm	ppm	%	%	%

TO-17/TO-13a Sorbent Tube:

Sample ID and Analysis	Air Volume & flow rate (L or ml/min)	Sample Collection Start/End Time

General Observations/Notes:

Photo ID: ppb reading on the PID following sample
Purge rate: mL/min Purge time: (to) collection from soil vapor tubing.
Digital pre-sample canister pressure: - Vacuum pressure: Approximately[ ]inHg during sorbent tube sample
(Day 2): Purge rate: mL/min Purge time: (to)
Barometer/Temperature/Humidity (Day 2): [Start: inHg/ *F / %] [Stop: inHg/ *F / %]
Notes:

Approximating One-Well Volume (for purging temporary points): Each 0.5'x 3/8-inch ID vapor point will have a volume of approximately 11mL. Each foot of ¼-inch ID tubing will have a volume of approximately 10 mL.

Attachment I-1d

Indoor Ambient Air Sample Collection Log

	# Indoor/Ambient Air Sample Collection Log **Sample ID:** -___-___-___

Client:		**Date/Day:**	
Project:		**Sample Intake Height:**	' ALS
Location:		**Subcontractor:**	
Project #:		**Miscellaneous Equipment:**	
Samplers:			
Coordinates:	(See attached Figure)	**Weather Conditions:**	
Outdoor/Indoor:		**Start/Stop Time:**	

Instrument Readings:

Time	Canister Pressure (inches Hg)	Temperature (F)	Relative Humidity (%)	Air Speed (ft/min)	Barometric Pressure	PID (ppb) / (ppm)

SUMMA Canister Information

Size (circle one): **1 L** **3 L** **6 L** **Other: ___L**

Canister ID: _______________

Flow Controller ID: _______________

General Observations/Notes:

Photo(s):
Initial Digital reading pre-sample:
Final Digital reading following sample collection:

Attachment I-2

Indoor Air Quality Questionnaire and Building Inventory

INDOOR AIR QUALITY QUESTIONNAIRE AND BUILDING INVENTORY

This form must be completed for each residence involved in indoor air testing.

Preparer's Name ____________________________________ Date/Time Prepared ______________

Preparer's Affiliation ________________________________ Phone No. ______________________

Purpose of Investigation ____

1. OCCUPANT:

Interviewed: Y / N

Last Name: __________________________ First Name: __________________________

Address: ____

County: _________________

Home Phone: ____________________ Office Phone: ____________________

Number of Occupants/persons at this location _______ Age of Occupants ______________________

2. OWNER OR LANDLORD: (Check if same as occupant ___)

Interviewed: Y / N

Last Name: __________________________ First Name: __________________________

Address: ____

County: _________________

Home Phone: ____________________ Office Phone: ____________________

3. BUILDING CHARACTERISTICS

Type of Building: (Circle appropriate response)

Residential	School	Commercial/Multi-use
Industrial	Church	Other: _________________

If the property is residential, type? (Circle appropriate response)

Ranch	2-Family	3-Family
Raised Ranch	Split Level	Colonial
Cape Cod	Contemporary	Mobile Home
Duplex	Apartment House	Townhouses/Condos
Modular	Log Home	Other:_______________

If multiple units, how many? ________

If the property is commercial, type?

Business Type(s) ______________________________________

Does it include residences (i.e., multi-use)? Y / N If yes, how many? ______

Other characteristics:

Number of floors ______ Building age______

Is the building insulated? Y / N How air tight? Tight / Average / Not Tight

4. AIRFLOW

Use air current tubes or tracer smoke to evaluate airflow patterns and qualitatively describe:

Airflow between floors

____
____
____

Airflow near source

____
____
____

Outdoor air infiltration

____
____
____

Infiltration into air ducts

____
____
____

5. BASEMENT AND CONSTRUCTION CHARACTERISTICS (Circle all that apply)

a. Above grade construction:	wood frame	concrete	stone	brick
b. Basement type:	full	crawlspace	slab	other________
c. Basement floor:	concrete	dirt	stone	other
d. Basement floor:	uncovered	covered	covered with _________________	
e. Concrete floor:	unsealed	sealed	sealed with	
f. Foundation walls:	poured	block	stone	other ________
g. Foundation walls:	unsealed	sealed	sealed with _________________	
h. The basement/slab is:	wet	damp	dry	moldy
i. The basement is:	finished	unfinished	partially finished	
j. Sump present?	Y / N			

k. Water in sump? Y / N / not applicable

Basement/Lowest level depth below grade: ________(feet)

Identify potential soil vapor entry points and approximate size (e.g., cracks, utility ports, drains)

____

____

6. HEATING, VENTING and AIR CONDITIONING (Circle all that apply)

Type of heating system(s) used in this building: (circle all that apply – note primary)

Hot air circulation	Heat pump	Hot water baseboard	
Space Heaters	Stream radiation	Radiant floor	
Electric baseboard	Wood stove	Outdoor wood boiler	Other___________

The primary type of fuel used is:

Natural Gas	Fuel Oil	Kerosene
Electric	Propane	Solar
Wood	Coal	

Domestic hot water tank fueled by: ______________________________

Boiler/furnace located in:	Basement	Outdoors	Main Floor	Other___________
Air conditioning:	Central Air	Window units	Open Windows	None

Are there air distribution ducts present? Y / N

Describe the supply and cold air return ductwork, and its condition where visible, including whether there is a cold air return and the tightness of duct joints. Indicate the locations on the floor plan diagram.

____

____

____

____

7. OCCUPANCY

Is basement/lowest level occupied? Full-time Occasionally Seldom Almost Never

Level **General Use of Each Floor (e.g., familyroom, bedroom, laundry, workshop, storage)**

Basement ____

1st Floor ____

2nd Floor ____

3rd Floor ____

4th Floor ____

8. FACTORS THAT MAY INFLUENCE INDOOR AIR QUALITY

a. Is there an attached garage? Y / N

b. Does the garage have a separate heating unit? Y / N / NA

c. Are petroleum-powered machines or vehicles stored in the garage (e.g., lawnmower, atv, car) Y / N / NA
Please specify ___________________

d. Has the building ever had a fire? Y / N When? _________________

e. Is a kerosene or unvented gas space heater present? Y / N Where? ________________

f. Is there a workshop or hobby/craft area? Y / N Where & Type? ________________

g. Is there smoking in the building? Y / N How frequently? _______________

h. Have cleaning products been used recently? Y / N When & Type? ________________

i. Have cosmetic products been used recently? Y / N When & Type? ________________

j. Has painting/staining been done in the last 6 months? Y / N Where & When? _______________

k. Is there new carpet, drapes or other textiles? Y / N Where & When? _______________

l. Have air fresheners been used recently? Y / N When & Type? ________________

m. Is there a kitchen exhaust fan? Y / N If yes, where vented? ____________

n. Is there a bathroom exhaust fan? Y / N If yes, where vented? ____________

o. Is there a clothes dryer? Y / N If yes, is it vented outside? Y / N

p. Has there been a pesticide application? Y / N When & Type? _________________

Are there odors in the building? Y / N
If yes, please describe: ____

Do any of the building occupants use solvents at work? Y / N
(e.g., chemical manufacturing or laboratory, auto mechanic or auto body shop, painting, fuel oil delivery, boiler mechanic, pesticide application, cosmetologist

If yes, what types of solvents are used? ____

If yes, are their clothes washed at work? Y / N

Do any of the building occupants regularly use or work at a dry-cleaning service? (Circle appropriate response)

Yes, use dry-cleaning regularly (weekly)
Yes, use dry-cleaning infrequently (monthly or less)
Yes, work at a dry-cleaning service
No
Unknown

Is there a radon mitigation system for the building/structure? Y / N Date of Installation: ____________
Is the system active or passive? Active/Passive

9. WATER AND SEWAGE

Water Supply:	Public Water	Drilled Well	Driven Well	Dug Well	Other: _______
Sewage Disposal:	Public Sewer	Septic Tank	Leach Field	Dry Well	Other: _______

10. RELOCATION INFORMATION (for oil spill residential emergency)

a. Provide reasons why relocation is recommended: ______________________________________

b. Residents choose to: remain in home relocate to friends/family relocate to hotel/motel

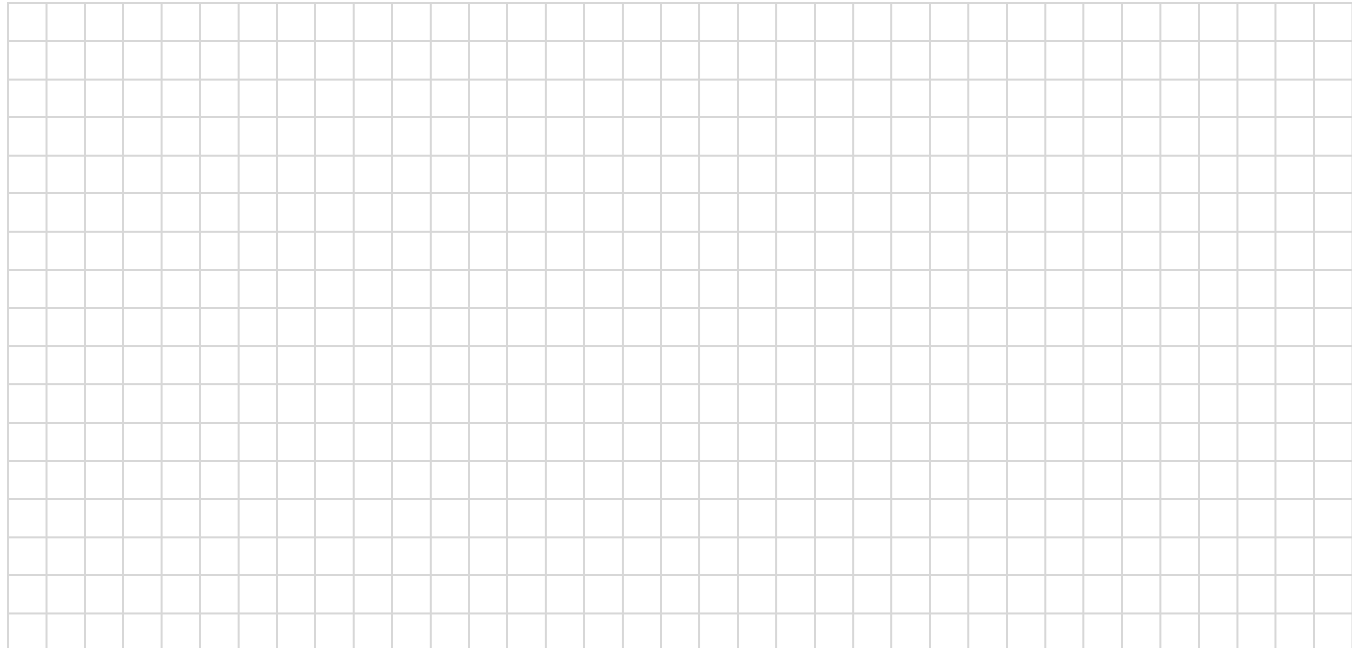
c. Responsibility for costs associated with reimbursement explained? Y / N

d. Relocation package provided and explained to residents? Y / N

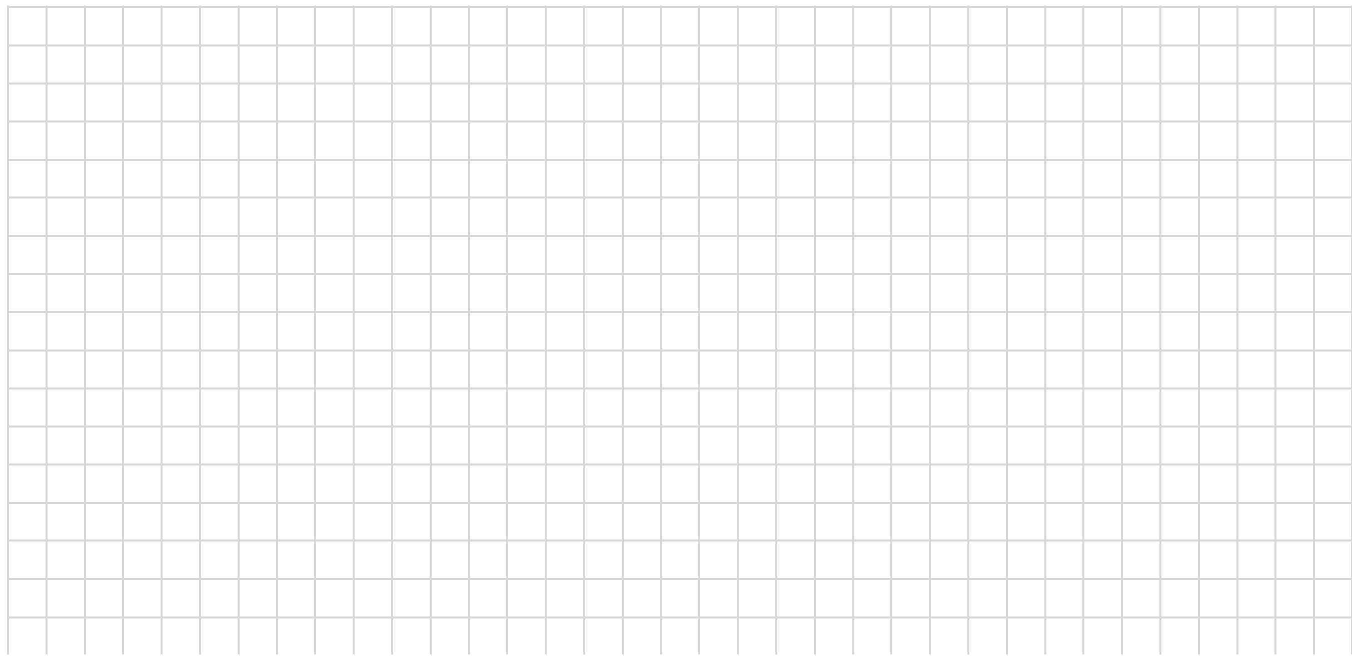
11. FLOOR PLANS

Draw a plan view sketch of the basement and first floor of the building. Indicate air sampling locations, possible indoor air pollution sources and PID meter readings. If the building does not have a basement, please note.

Basement:



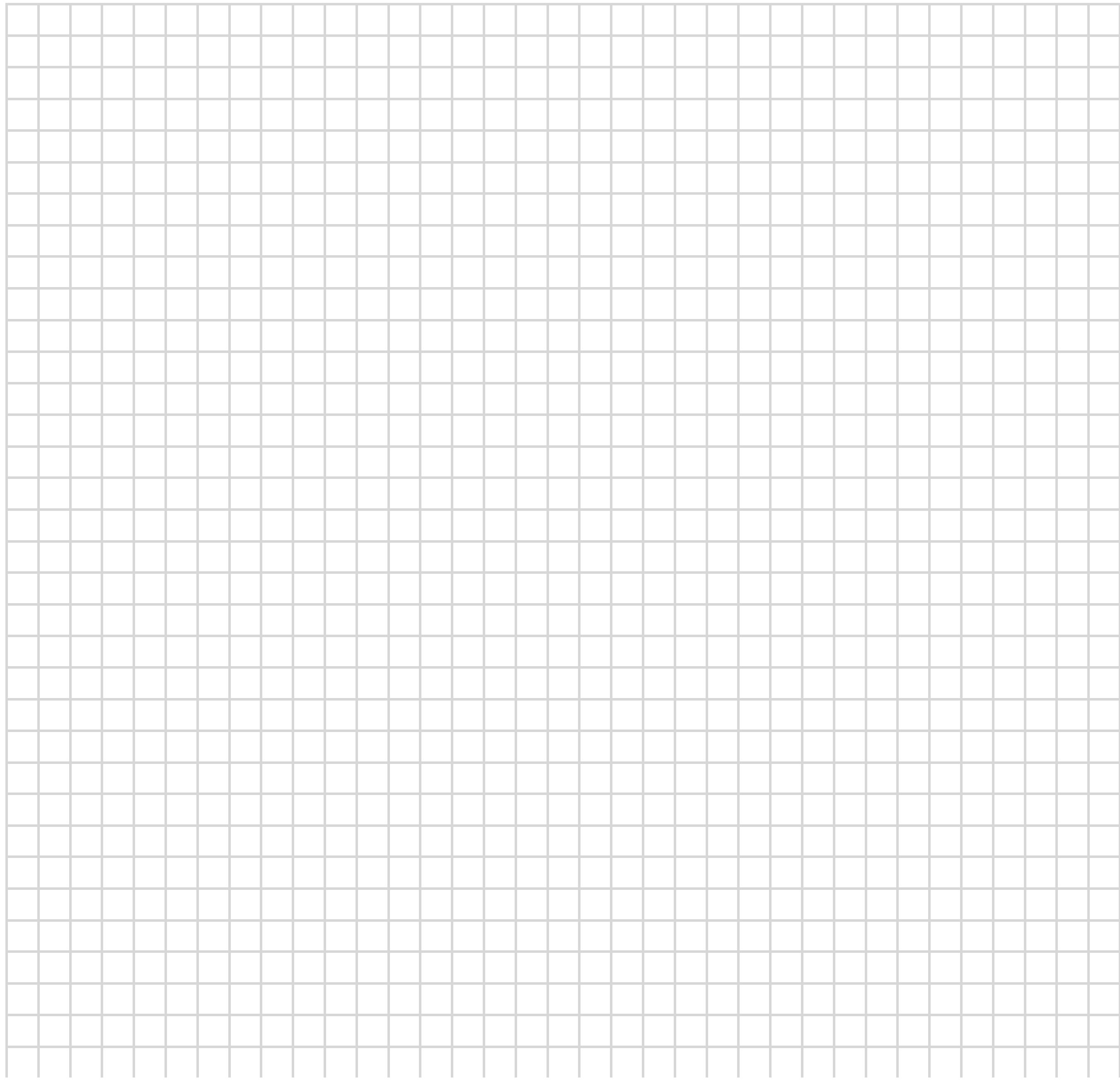
First Floor:



12. OUTDOOR PLOT

Draw a sketch of the area surrounding the building being sampled. If applicable, provide information on spill locations, potential air contamination sources (industries, gas stations, repair shops, landfills, etc.), outdoor air sampling location(s) and PID meter readings.

Also indicate compass direction, wind direction and speed during sampling, the locations of the well and septic system, if applicable, and a qualifying statement to help locate the site on a topographic map.



13. PRODUCT INVENTORY FORM

Make & Model of field instrument used: (e.g., PPB RAE)

List specific products found in the residence that have the potential to affect indoor air quality.

Location	Product Description	Size (units)	Condition*	Chemical Ingredients	Field Instrument Reading (units)	Photo** Y / N

* Describe the condition of the product containers as **Unopened (UO), Used (U),** or **Deteriorated (D)**
** Photographs of the **front and back** of product containers can replace the handwritten list of chemical ingredients. However, the photographs must be of good quality and ingredient labels must be legible.

13. PRODUCT INVENTORY FORM (Cont.)

Location	Product Description	Size (units)	Condition*	Chemical Ingredients	Field Instrument Reading (units)	** Photo Y / N

Appendix J

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J-1 Laboratory SOP for Extraction Method

J-2 Laboratory SOP for Test Method

1. Project Description

1.1 General

Berkshire Environmental Consultants, Inc. (BEC) performs, on behalf of General Electric Company (GE), ambient air monitoring for particulate matter and polychlorinated biphenyls (PCBs) for numerous projects in Pittsfield, Massachusetts. Ambient air monitoring is performed as part of remediation or site assessment activities to address concerns about potential air pathway exposures to dust and/or PCBs. This Appendix presents the standard operating procedures (SOPs) to be used for particulate matter monitoring and for high-volume ambient air sampling for PCBs. Procedures for other types of air monitoring activities (e.g., low-volume sampling for PCBs, monitoring for other constituents) that may be required for a particular project will be presented in the project-specific work plan.

1.2 Project Objectives

The objective of the air monitoring program is to provide valid and representative data on ambient air levels of particulate matter and/or PCBs in order to ensure that remedial activities are not causing an unacceptable increase in ambient air concentrations of particulates and/or to assist in evaluating air pathway exposures to PCBs.

1.3 Format of Appendix

Section 2 of this Appendix presents the procedures to be followed for ambient air monitoring for particulate matter. Section 3 presents an overview of the ambient air monitoring program for PCBs. Sections 4 through 14 provide additional details regarding PCB air monitoring activities, including quality assurance objectives, sampling procedures, sample custody, analytical procedures, calibration procedures, data validation and reduction, internal quality control checks, preventative maintenance, routine quality assurance procedures, corrective action, and reporting.

1.4 Meteorological Monitoring

In connection with either particulate matter monitoring or PCB monitoring (or both), meteorological data from the Automated Surface Observation System (ASOS) Monitor operated at the Pittsfield Municipal Airport in Pittsfield, Massachusetts, will be included with the sampling results. This ASOS Monitor is operated by the National Weather Service, Federal Aviation Administration, and the U.S. Department of Defense. The ASOS Monitor measures and records wind speed, wind direction, precipitation, temperature, sky conditions, barometric pressure, and relative humidity.

The collected meteorological data are used to help evaluate receptor population exposures to ambient particulate matter and PCB levels.

2. Particulate Monitoring

Where called for in a project-specific work plan, real-time particulate monitoring will be conducted during the excavation portion of remedial action at a given area. Monitoring will be conducted daily during the hours of excavation. Approximately 10 hours a day of sampling data, from 7:00 a.m. to 5:00 p.m., are anticipated. Particulate monitoring will occur throughout the period of excavation or as otherwise provided in the work plan.

Particulate monitoring will be conducted using a real-time airborne particulate monitor, which may be any of the following: MIE Model pDR-1000, MIE Model DR-2000, MIE Model DR-4000, Met One E-BAM Mass Monitor, or equivalent monitor approved by EPA for air sampling of particulate matter with a diameter less than 10 micrometers (PM10) or total suspended particulates (TSP).

The dataRAM Model pDR-1000 (pDR) uses a passive sampling technique and light scattering photometer to determine particulate concentrations. The dataRAM pDR has a measurement range of 0.001 to 400 mg/m3.

The dataRAM DR-2000 or 4000 (DR) is a high-sensitivity nephelometric monitor. The DR samples the air at a constant, regulated flow rate by means of a built-in diaphragm pump. Like the pDR, the DR uses a light scattering photometer optimized for the measurement of airborne particle concentrations. The DR has a measurement range of 0.0001 mg/m3 (0.1µg/m3) to 400 mg/m3.

The Met One E-BAM uses a Beta Attenuation technique for measuring particulate. The E-BAM samples the air at a constant regulated flow onto a continuous glass fiber filter tape. The density of particulate collected onto the filter tape is quantified based on the response of a scintillator photo multiplier tube to a C14 beta pulse on the filter tape. The Met One E-BAM has a measurement range of 0-10 mg/m3.

For any of these instruments, particulate data will be logged by the instrument's datalogger and averaged and recorded for each 15-minute period hour and for each sampling day. During its operation, the instrument will report, on a real-time basis, the instantaneous particulate reading, the highest discrete reading that has been recorded during the monitoring period, and the cumulative average for the current monitoring period.

Both the DR and the pDR have an inherent measurement sensitivity to moisture and thus to humid conditions. The DRs are equipped with relative humidity indicators and air inlet heaters to both evaporate moisture and, if necessary, automatically adjust the particulate measurement for humidity. The pDR has no technique to adjust for humidity. The MET One E-BAM is also sensitive to moisture and is equipped with both a relative humidity sensor and in-line heater to evaporate moisture in order to prevent it from condensing on the filter tape. As a result of the sensitivity to moisture, the monitors are carefully observed during humid or rainy weather. GE or its contractor may, at times, use professional engineering judgment to determine the reliability of data collected during very high humidity conditions. Data summaries will exclude the time period when moisture is clearly a factor. The raw data file will be marked and maintained. Any such judgments will be noted appropriately on the data summary tables.

Calibrations and maintenance will be conducted at the frequency and in accordance with the procedures recommended by the manufacturer. All calibrations will be recorded.

2.1 Monitoring Locations

The monitoring locations at each area will be determined prior to the initiation of excavation activities. All areas will be monitored in at least three locations for areas subject to the Consent Decree and at least one location for other areas (with the specific number of monitoring locations to be determined on a project-specific basis). As required, additional monitors may be operated at a given area to adequately assess ambient particulate concentrations. The specific monitoring location(s) will be established based on the following: location of excavation, truck and vehicle traffic on-site, downwind receptors, obstructions, and accessibility. As excavation proceeds and conditions change, the monitoring locations may be moved.

A background particulate sampler will be installed at an upwind or at an off-site representative location, as specified in the project-specific work plan. Data from this site will be used to normalize ambient particulate concentrations during remedial action.

2.2 Quality Assurance and Quality Control Procedures

Specific quality assurance and quality control for the particulate sampling will be based on manufacturer's recommendations.

2.3 Documentation and Reporting

Particulate data will be summarized daily. Data which exceed the notification levels described below will be reported to the GE Project Manager and to EPA or the MDEP (as appropriate) in accordance with Section 2.4. Daily particulate and meteorological data will be summarized weekly and provided in a written summary report to the GE Project Manager on Monday for the previous week. All field data recorded during ambient monitoring will be documented according to the procedures in the Field Sampling Plan/Quality Assurance Project Plan (FSP/QAPP). The monitoring data will be provided to the regulating agency (EPA or MDEP, as appropriate) at a frequency agreed upon between GE and the agency. A written report summarizing the results will be provided to GE at the conclusion of sampling and will include the following:

- Date and Time of Sampling
- Sampling Locations
- Calibration and Maintenance Activities
- Pollutants Monitored
- Sampling Frequency
- Data Results

- Quality Assurance Assessment
- Meteorological Data Summary
- Discussion of Problems or Disruptions

2.4 Notification and Action Levels

For each day of monitoring, the particulate data from the downwind monitor will initially be compared with the data from the background monitor. In addition, the average 10-hour PM10 concentrations at the on-site monitors will be compared with a notification level of 120 µg/m^3 – which represents 80% of the current 24-hour National Ambient Air Quality Standard (NAAQS) for PM10 (150 µg/m^3). This level has been selected to allow notice to GE before concentrations reach the level of the 24-hour NAAQS the action level). If the average 10-hour PM10 concentration at any on-site monitor exceeds the notification level of 120 µg/m^3, the exceedance will be reported to the regulating agency (EPA or MDEP) as soon as practicable, but no later than 24 hours following receipt of the data showing the exceedance. In addition, GE will provide written notice of the exceedance to the regulating agency within 72 hours after receipt of the data showing the exceedance.

Any exceedance of the NAAQS (the action level) will be reported to the regulating agency (EPA or MDEP) immediately after receipt of the data showing the exceedance, but within 24 hours after receipt of the data. In addition, GE will provide written notice of the exceedance to the regulating agency within 72 hours after receipt of the data showing the exceedance.

In the event of any exceedance of the notification level or the action level, GE will take the response actions set forth for such exceedance in Section 6 of Attachment D (Ambient Air Monitoring Plan) to GE's Project Operations Plan (POP).

3. PCB Monitoring – General

Where PCB ambient air monitoring is called for in a project-specific work plan, BEC will install and operate General Metal Works Model PS-1 or equivalent samplers to monitor ambient PCB levels. Monitoring programs consist of one to several monitoring sites, depending on the scope of the specific remediation or site assessment activity. Each monitoring program includes downwind sites, at least one upwind or background site, and a co-located site. The samplers will typically operate for 24 hours from 7 a.m. to 7 a.m. during site remediation activity. The specific number of sampling sites and days will be determined on a project to project basis. Where PCB ambient air sampling is called for in a project-specific work plan, PCB air sampling will be performed on two occasions prior to the start of the remediation and no less frequently than once every 4 weeks (determined on a cumulative basis) during remediation activity for that area, unless otherwise provided in the work plan or otherwise agreed between GE and the pertinent regulatory agency (EPA or MDEP). For those cases where the total duration of the remediation project is less than 4 weeks and PCB ambient air sampling is called for in a project-specific work plan, PCB air sampling will be conducted at least one time during the remediation activity.

The sampling method for PCBs is USEPA Compendium Method TO-4A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using High Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD); Second Edition, January 1999. This method employs a high volume sampler and a sampling cartridge consisting of a glass fiber filter with a polyurethane foam (PUF) absorbent to sample ambient air at a rate of 0.20 - 0.28 m3/minute. The filter and PUF cartridge are placed in clean, sealed containers and returned to the laboratory for analysis. The PCBs are recovered by Soxhlet extraction with 10% diethyl ether/hexane. The extracts are reduced in volume using Kuderna-Danish (K-D) concentration techniques and subjected to column chromatographic cleanup. The extracts are analyzed for PCBs using capillary gas chromatography with electron capture detection (GC/ECD), as described in Method TO-4A.

Analytical laboratories are required to follow quality assurance measures and performance criteria as described in Method TO-4A.

4. PCB Data Quality Assurance Objectives

4.1 Quality Assurance

The objective of this quality assurance plan is to ensure that the data collected on ambient levels of PCB are adequate to meet the objectives of the specific monitoring program and the intended uses of the data. The following objectives were used as guidelines to assuring quality in the design and implementation of the monitoring program.

- The sampling and analytical procedures should follow the EPA Compendium Method TO-4A and EPA recommended guidelines, where applicable. The EPA Compendium Methods are not standard EPA reference methods. Pursuant to conversations with Method TO-4A author, Robert G. Lewis, EPA, RTP, in October 1999, deviations from the compendium methods and procedures may be made on a case-by-case basis and, if required, will be presented in the project-specific work plan.

- All phases of the sampling program will be adequately documented. Documentation will be maintained to evidence the validity of calibrations, sample collection, flow calculations, sample custody, analytical performance, data reduction and audit procedures. Field records will be maintained to record and reconstruct sampling events, calibration procedures, maintenance and repair activity, and any other information pertinent to the assurance of quality in the sampling program.

- The GE Project Manager will be kept informed of sampling activity with update memoranda.

- Sampling and analytical data quality will be measured and reported, where applicable, in terms of completeness, precision, accuracy (bias), representativeness, and comparability.

4.2 Data Quality in Terms of Representativeness, Comparability, Completeness, Precision and Accuracy

Sample Validity - A valid collected sample is defined as an air sample that is collected over 24 hours, +60 minutes, from approximately 7 a.m. to 7 a.m., at a flow rate of 0.20-0.28 m3/min. For a sample to be valid, the minimum sample volume must be no less than 276 standard cubic meters (scm) and no greater than 420 scm. (The target flow rate and sample volume are 0.225 m3/min and 325 scm, respectively.) The sample must be collected and analyzed under conditions which meet the specified objectives of precision, accuracy and, where applicable, representativeness.

Representativeness - The sampling network and frequency of sampling will be designed to provide data that are representative of the ambient levels of PCB. The sampling sites and the frequency and duration of sampling will be presented on a project by project basis with the rationale and procedures for sampling selection.

Comparability – Samples will be collected using the procedures set forth in Method TO-4A. Results for total PCB will be reported in μg/m^3 corrected to EPA standard conditions of 25°C and 760 mm Hg.

Completeness – The project completeness requirements are as follows:

- 90% validity of the total project samples (including co-located samples, background, and trip blanks);
- 90% validity at each sampling site over the course of the project (including co-located samples, background, and trip blanks); and
- No sampler site may have two or more invalid samples for consecutive sampling events.

Precision – Sampling precision will be measured by collecting a replicate sample at a co-located monitor at one monitoring site during each sampling event. Each compound with a detectable concentration at least two times greater than the practical quantitation limit (PQL) identified below must have a relative percent difference (RPD) value that is less than 50%.

Accuracy – Sampling accuracy is measured by auditing the flow rate of the samplers before and after each sampling event using a flow transfer standard. The accuracy criterion before and after using the flow transfer standard is +/-10% of the set point. The difference between the audit flow measurement and the calculated flow based on the sampler flow indicator (magnehelic gauge) and a calibration curve will be used to calculate accuracy.

Analytical accuracy or recovery is determined by the laboratory using an internal laboratory surrogate standard reflective of PCB. Sample recoveries ranging from 65 to 125% are considered acceptable.

4.3 Detection and Reporting Limits

The laboratory's Method Detection Limit (MDL) for PCBs in air samples, which was established for PCB Aroclor 1254, is 0.03 µg/PUF; and the target PCB practical quantitation limit (PQL) for this project, which is consistent with Method TO-4A, is 0.1 µg/PUF. (At the target air volume of 325 scm, these limits translate into PCB air concentrations of 0.00009 µg/m^3 and 0.0003 µg/m^3, respectively.) The target reporting limit (RL) based on the PQL of 0.1 µg/PUF and a target air volume of 325 scm is 0.0003 µg/m^3. This reporting limit may be higher or lower for individual samples based on the exact air volume that is collected for each sample. The PQL and RL were established in consideration of the following:

- Massachusetts Allowable Ambient Levels (AAL) for total PCBs in air, as used in the Air Toxics Program (which consist of a 24-hour average of 0.003 µg/m^3 and an annual average of 0.0005 µg/m^3); and

- Analytical detection capabilities as limited by sampling duration and sampling rate.

4.4 Sampling Flow Rate and Total Volume

Method TO-4A (Appendix I) recommends a sampling rate of 0.225 m^3/min with an acceptable flow rate range within +/- 10% (i.e. 0.20-0.28 m^3/min) and a target total volume of 325 scm. The anticipated operating rate is 0.225 m^3/min. At this sampling rate, a total flow volume of 324 m^3/air sample will be achieved over a 24-hour sampling period.

4.5 Summary of Project Detection Limits

Target Sampling Rate	0.225 m^3/min
Target Sample Volume	325 m^3/PUF
Lab MDL	0.03 µg/PUF
Lab PQL	0.1 µg/PUF
Allowable Project RL	0.0003 µg/m^3

5. PCB Sampling Procedures

5.1 Sampling Sites

Sampling sites for PCB air monitoring will be selected based on the physical site characteristics, receptor locations, source location and strength, site access, site security and the availability of electric power.

5.2 Sampling Frequency

PCB air samples will be collected over 24 hours from approximately 7 a.m. to 7 a.m. at the established frequency for each project.

5.3 Sampling Methods

The sample collection SOPs are based on the TO-4A Compendium Method.

5.4 Sampling Forms

A field data form is used to record all field data associated with the sampling event.

5.5 PUF and Filter Preparation and Cleanup

For initial cleanup, PUFs and filters (not necessarily at the same time) will be extracted and prepared in accordance with Method TO-4A, Section 10.2 (Preparation of Sampling Cartridge). Each PUF will be placed in a glass sampler cartridge, wrapped in hexane rinsed foil, placed in a labeled zip-loc bag and sealed. At least one PUF in each batch of 20 will be certified in accordance with Method TO-4A, Section 10.3 (Procedure for Certification of PUF Cartridge Assembly).

Glass cartridges will be cleaned and reused after each sampling event. New PUFs and filters will be used for each event. Used PUFs and filters will be discarded after each sampling event.

5.6 Sample Containers

The aluminum sample cylinders will be assembled in the office/lab with a PUF cartridge and glass fiber filter. A cover plate will be placed over the filter and a foil covering is placed over the coupler for transport of the module to the field. The assembled cylinders will be transported to and from the field in a hexane rinsed ice chest.

For transport to the laboratory, the filter and PUF cartridge will be removed from the sampler cartridge. The filter will be folded and placed in the glass sampling cartridge atop the PUF. The cartridge will be wrapped in hexane-rinsed aluminum foil. The samples are placed in zip-loc bags and labeled.

5.7 Sample Holding Times and Preservation Methods

For PCB samples collected Monday through Friday, delivery at the laboratory will be made no later than 10 a.m. on the day following sample collection. Samples collected on Saturday and/or Sunday will be packaged for shipment and refrigerated. These samples will be shipped on Monday morning for delivery by 10 a.m. on Tuesday. All samples will be shipped in an ice chest with adequate blue ice packs to maintain the temperature at 4°C.

All samples will be extracted by the analytical laboratory within 7 days after sample collection. Concentrates will be stored refrigerated in vials until analyzed. Concentrates will be analyzed within 40 days.

5.8 Documentation

PCB sample numbers, sampling conditions and analyses, etc. will be recorded on a sampling data form. Original field copies of all sampling data forms will be maintained.

6. PCB Sample Custody

6.1 Field Sample Operations

6.1.1 PUF and Filter Receipt and Handling

Cleaned, labeled PUF cartridges will be received from the laboratory via a commercial carrier or courier. Upon receipt, each batch of PUF cartridges will be sorted by date of extract and logged. The PUFs with filters will be stored in ice chests. The batches will be used in order of extraction date. As the PUFs in each batch are used, the date and site where each is used will be logged. PUFs are considered clean and usable for 30 days after initial extractions.

6.1.2 Sample Collection

Each PUF and filter will be assembled into a sampling cylinder in the office/lab in accordance with procedures contained in Method TO-4A, Section 11.3.2 (Preparing Cartridge for Sampling) and assigned to a specific sampler. The sampler, PUF cartridge numbers, and all field sampling data will be recorded on a dedicated field data sheet. Equipment calibration and sampling procedures will follow those specified in Method TO-4A unless a specific variation is proposed in the project work-plan.

When sampling is completed, the PUF/filter will be kept together in the sampling cylinder for transport to the office. In the office/lab, the filter will be removed and placed in the glass sampling cylinder atop the PUF. The PUF will be taken from the sampling cylinder, wrapped in foil and bagged. The samples will be labeled and identified with sample numbers. The sample numbers will be logged. All information relating to date, time and conditions of sampling will be recorded on the field data sheet. Samples will be refrigerated for cooling prior to shipment.

6.1.3 Sample Shipment

Samples will be shipped in sealed ice chests on ice or blue ice with a chain-of-custody (COC) seal over the chest lid. Samples will be shipped under COC to the analytical laboratory.

The COC record will be completed, signed, and mailed inside the ice chest. Samples will be shipped by a commercial carrier or courier and require a delivery signature at the analytical laboratory.

6.2 PCB Laboratory Operations

At the analytical laboratory, the samples will be received, signed for, and inspected by a sample custodian. The COC record will be verified with the received samples. Any inconsistencies will be noted on the COC record. From that point on, the samples will be handled according to the laboratory's SOPs.

All sampling COC field records will be maintained in the sampling file at the BEC office in Pittsfield. All COC records and log sheets for the laboratory will be maintained at the analytical laboratory.

7. PCB Analytical Procedures

7.1 Extraction Method

The Compendium Method TO-4A procedure for extracting PCB from the PUF and filter will be followed. The PUFs will be extracted within 7 days after the sample is collected.

The laboratory SOP for the extraction method is included as Attachment J-1 to Appendix J.

7.2 Test Method

The analytical procedure to determine PCBs will be as described in Method TO-4A. and as specified in Section 4 of Attachment D (Ambient Air Monitoring Plan) to GE's POP. Any deviations from Method TO-4A, if required, will be proposed in the project-specific work plan.

To corroborate the results using GC/ECD, samples may be analyzed with high resolution GC/MS. Results of both methods will be reported.

The laboratory SOP for the test method is included as Attachment J-2 to Appendix J.

8. PCB Calibration Procedures

Calibration for all PCB sampling equipment will be conducted in accordance with the procedures specified in Method TO-4A or the EPA High Volume Reference Method (as applicable). All data and calculations for calibrations will be recorded on log sheets and maintained in a calibration log file. Method variations are noted.

8.1 Flow Rate Transfer Standard

Frequency of Calibration:	Annually
Reference Procedure:	Flow Rate Transfer Standard (NIST Primary Standard)
Variations From Reference:	None.
Accuracy:	±2%.

Summary: Calibrated against a positive displacement standard volume meter at various flow rates. Calculate the linear least squares slope and intercept of the line representing the relationship. The orifice calibration is performed by an independent contractor.

8.2 High Volume Sampler (Multi-Point Calibration)

Frequency of Calibration: Upon receipt; every 6 months; following motor, ball valve, magnehelic gauge, or other major equipment repair or replacement; or any time the difference between a one-point audit and the sample flow rate deviates +10%.

Reference Procedure: Method TO-4A, Section 11.2.2

Variations From Reference: None

Accuracy: Correlation coefficient greater than 0.95.

Summary: The high volume sampler will be calibrated against a certified orifice flow transfer standard. A calibration curve will be drawn and a least square regression calculated. The equation will be used to determine standard flow during sampler operation. The calibrations for each monitor will be recorded on a worksheet and maintained in a calibration log.

9. PCB Data Reduction, Validation, and Notifications

9.1 Data Reduction

The PCB sampling data and analytical results will be combined to report an ambient concentration of PCBs in µg/m^3 for each sample.

Sampling Data

The sampling flow rate and the total volume of air sampled will be calculated from the pressure readings collected during sampling and the elapsed sampling time.

1. Using appropriate calibration tables for each sampler, Convert $P_1 \ldots P_n$ to $Q_1 \ldots Q_n$

   where: $P_1 \ldots P_n$ = magnehelic readings from sampling event recorded on the field data sheet

   Q1 … Qn = flow readings from calibration table corrected to standard conditions (m3/min)

2. Determine the average flow rate:

$$QSTD = \frac{Q_1 + Q_2 \ldots Q_n}{N}$$

where: QSTD = average flow rate in standard conditions (m3/min)

N = number of flow readings

3. Calculate the total elapsed time:

$ETM_{Finish} - ETM_{Start} = ET$

where: ETM_{Finish} = elapsed time meter reading at the end of sampling

ETM_{Start} = elapsed time meter reading at the start of sampling

ET = total elapsed time (hours)

4. Calculate the total volume of air sampled under ambient conditions:

$V_a = \sum n_{i-1}(T_i \times F_i)/1000 \text{ L/m}^3$

where: V_a = total volume of air sampled (m^3)

T_i = length of sampling segment between flow checks (min)

F_i = average flow during sampling segment (L/min)

5. Correct the air volume to EPA standard temperature and standard pressure

$V_s = V_a \times [(P_b - P_w)/760 \text{ mm Hg}] \times (298K/t_A)$

where: V_s = volume of air at standard conditions (std. m^3)

V_a = total volume of air sampled (m^3)

P_b = average ambient barometric pressure (mm Hg)

P_w = vapor pressure of water at calibration temperature (mm Hg)

T_A = average ambient temperature, °C + 273

Analytical Data

The laboratory will reduce the analytical results for each sample to total PCBs measured in µg/PUF. The procedures for this determination and calculation are defined in the USEPA Method TO-4A.

Sample Concentrations Conversions

The analytical data provided by the laboratory will be reduced for comparison with standards.

$$CA = \frac{\mu g/PUF}{Vm/PUF}$$

where: CA = concentration of PCBs in sample ($\mu g/m^3$)

V_s = total standard volume of air

9.2 Data Validation

All PCB air data will be validated in accordance with Validation Annex F to the FSP/QAPP.

9.3 Notifications

The notification and action levels for PCBs are specified in Section 6 of Attachment D (Ambient Air Monitoring Plan) to GE's POP. In the event of an exceedance of the notification or action level for PCBs, GE will make the notifications specified in that section and will take the other response actions set forth in that section for the type of exceedance in question.

10. PCB Internal Quality Control Checks

10.1 Sampler Flow Checks

In addition to the standard equipment calibration procedures identified in Section 7.0, routine quality control checks to verify flow will be conducted during PCB sampler operation.

Procedure	Frequency	Control Limit	Corrective Action
One-Point Audit	Before and After Each Sampling Event	± 10% Deviation from Calculated Value	Note sample flow volume as estimated; Recalibrate
Magnehelic Zero Check	Before and After Each Sampling Event	± 2" H_20	Adjust
Leak Check	Before and After Each Sampling Event; at Each Calibration	No Leaks	Repair Leak
Magnehelic Readings	Every 6 Hours During Sampling	± 10" H_2O	Note reading; adjust instruction

10.2 Field Sampling Precision Check

As a precision check on field sampling for PCBs, two samplers are co-located at one sampling site. The samplers are located 2 - 4 meters apart. One sampler will be identified as the primary sampler and the other will be designated as the duplicate sampler. The calibration, sampling, and analysis procedure for the two samplers will be the same as for all samplers. The co-located sampler will be operated whenever the routine sampler operates.

The variation between the ambient PCB concentrations measured at the two samplers will be compared and observed. The target limit of variation (precision) is that each compound with a detectable concentration at least two times greater than the laboratory PQL of 0.1 µg/PUF must have an RPD value less than 50%.

10.3 Process and Field Blanks

10.3.1 Laboratory Process Blank

Prior to shipment to the field, one PUF cartridge and filter from each batch of approximately 20 clean PUFs and prepared filters will be analyzed for PCBs. (This will be called a laboratory process blank.) In order for the PUF batch to be considered acceptable for use, the blank level must be below the laboratory PQL of 0.1 µg/PUF.

10.3.2 Trip (Field) Blank

For each sampling event, one PUF cartridge and filter will be carried to the field and returned in a clean sample holder. (This will be called a trip blank, and is also referred to in Method TO-4A as a field blank.) The sample will be handled as any other sample except that no air will be drawn through the cartridge. The aluminum sample cartridge will be installed on the sampler at the beginning of the sampling event and immediately removed. The aluminum cartridge will remain in a hexane rinsed ice chest during sampling and will be recovered and prepared for shipment to the analytical laboratory for analysis in the same manner as the remaining project samples. The blank level for the trip (field) blank is a level that is less than the laboratory PQL of 0.1 µg/PUF.

10.3.3 Field Spike

Before each sampling episode, one PUF plug from each batch of approximately 20 will be spiked with a known amount of the standard solution. The spiked plug will remain in a sealed container and will not be used during the sampling period. The spiked plug will be extracted and analyzed with the other samples. This field spike will act as a quality assurance check to determine matrix spike recoveries and to indicate sample degradation.

10.3.4 Solvent Process Blank

During the analysis of each batch (approximately 20) of samples, one process blank from the laboratory stock will be carried through the procedure (all steps conducted but no filter/PUF cartridge included) and analyzed to assure that the extraction solvent is free from PCB contamination. (This will be referred to as a solvent process blank.) To provide such assurance, the result for this solvent process blank should be less than the laboratory PQL of 0.1 µg/PUF.

10.3.5 Analytical Spike Recovery

The procedures and QA limits for sample extraction, clean-up and analysis are specified in Method TO-4A.

11. Preventative Maintenance for PCB Samples

11.1 Schedule

Preventative maintenance on the PCB sampling equipment will be performed on a routine basis. Records of all maintenance activities will be maintained.

Sampling Unit

At least once quarterly, the sampler housing will be inspected for wear and tear, making sure all moving parts, doors, lids, etc. are in good order. The electrical cords and connections will be inspected for integrity. The coupler connection, digital timer, magnehelic gauge, and ETM will all be visually inspected.

Sampler Motor

The motor will be inspected quarterly and brushes replaced as needed according to the manufacturer's recommendations. All motor brushes are to be replaced between 500 and 1000 hours of operation. After the motor brushes are replaced, the motor will be recalibrated following a sufficient break in period. The top and bottom rubber gaskets on the sampler motor will be inspected quarterly and replaced as needed.

Sampling Cartridge and Gaskets

The sampling cartridges will be visibly inspected each time they are used. Prior to each sampling event, the cartridges will be completely disassembled, cleaned with hexane, and checked. Gaskets in the cartridge will be checked each time the cartridges are used. They will be cleaned and replaced as needed.

11.2 Spare Parts Inventory

A sufficient inventory of spare parts consisting of at least two (except where noted) of each of the following will be maintained for the high volume samplers:

- Dual Sampling Modules
- Filter Gaskets
- Silicone Gaskets for Upper Module
- Silicone Gaskets for Lower Module
- Glass Cartridges with Support Screens and PUFs
- Replacement Motors
- Replacement Motor Brushes
- Calibration Orifice (one spare calibration orifice)

12. Corrective Action for PCB Sampling

12.1 Responsibility

Corrective action may be initiated as a result of disruptions in PCB sampling or problems associated with the quality control checks; calibrations; or performance, system, and quality assurance audits. The responsibility for implementing corrective action lies with the BEC Project Manager. Any non-routine corrective actions will be discussed with the GE Project Manager before implementation.

12.2 Internal Quality Control Checks

Corrective actions for internal quality control checks are described in Section 13 of this plan.

12.3 Calibrations

The corrective action for problems in calibration is to recalibrate and, if necessary, repair, replace, or conduct a calibration audit using the designated audit orifice standard.

12.4 Performance and System Audits

Any sampler flow problems identified during the one-point audits require that data for that sampling event be checked for accuracy. Equipment calibration audits may indicate the need for recalibration, repair, or replacement.

12.5 Sampling Data Completeness and Validity

All samples must meet the criteria for sample validity identified in Section 4.2 of this plan. Samples which do not meet these criteria are invalid. For any sampling event where more than one sample is defined as invalid due to sampling error, the sampling event will be rerun on the next available day.

12.6 Laboratory Analyses

The sample extract volume will provide sufficient extract to complete at least two additional analyses if there is a problem in the initial analyses. Decisions for repeating any sampling events due to invalid data from the lab will be made on a case-by-case basis.

13. PCB Monitoring Reports to GE

At the end of each PCB air monitoring project, a report will be prepared and delivered to the GE Project Manager. The report will summarize the sampling activity for the project and include the following information:

- A summary of activities including a review of any sampling disruptions or problems which may have occurred, the corrective actions taken, and a discussion of what impact the problems may have on data quality.
- Sampling and analytical results.
- Summary of data quality in terms of the quality assurance objectives.
- Calibration, data validation, quality control, and audit activity.
- Meteorological data summary.

The final report will present a quantitative assessment of ambient PCB concentrations.

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3. Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. I - Principles. EPA-600-9-76-005, March 1976.

4. Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. II - Ambient Air Specific Methods. EPA-600-4-77-027a, May 1985.

5. Sampling and Analysis of Toxic Organics in the Atmosphere. ASTM Technical Publication 721, August 1979.

Attachment J-1

Extraction and Cleanup of Polyurethane Foam Air Cartridges (PUFS) for EPA Method TO-4A PCBs or Pesticides in Air Cassette Media

STANDARD OPERATING PROCEDURE

EXTRACTION AND CLEAN-UP OF POLYURETHANE FOAM AIR CARTRIDGES (PUFS) FOR EPA METHOD TO-4A POLYCHLORINATED BIPHENYLS OR PESTICIDES IN AIR CASSETTE MEDIA

Reference Methods: SW-846 EPA METHOD TO-4A

LOCAL SOP NUMBER: NE151_08

EFFECTIVE DATE: 06/05/2012

SUPERSEDES: NE151_07

SOP DOCUMENT NUMBER: S-NY-O-151-rev.08

APPROVALS

6/5/2012

Dan Pfalzer
Assistant General Manager

Date

05/30/2012

Christina L. Braidwood
Quality Manager

Date

PERIODIC REVIEW

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

Signature	Title	Date
Signature	Title	Date

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1.0 IDENTIFICATION OF TEST METHOD

1.1 Standard Operating Procedure for the extraction and cleanup of high volume polyurethane foam (PUF) air cassette samples for polychlorinated biphenyl (PCB) or pesticide analysis using the Soxhlet extraction technique (Modified SW-846 Method 3540C/EPA Method TO-4A for subsequent analysis by SW-846 Method 8082 or 8081. Note: The Determinative Method (EPA Method 8082) requires secondary GC column analysis on different column for PUF samples.

1.2 The purpose of this SOP is to provide to the chemist the procedures required to perform extractions of PCBs or pesticides in PUF (air cassette) samples, using the Soxhlet extraction technique and to perform the subsequent extract volume reduction and cleanup.

2.0 APPLICABLE MATRICES

2.1 This test method is appropriate for polyurethane foam air cartridges (PUFs) & air filters (TO-4A).

3.0 DETECTION LIMIT

3.1 Please see determinative methods (Lab SOP NE148, NE131) for details.

4.0 SCOPE AND APPLICATION

4.1 The following procedure is utilized by Pace Analytical Services, Inc. for the extraction and cleanup of PCBs and pesticides from PUF (air) samples using the Soxhlet extraction method of Method TO-4A for subsequent analysis by SW-846 Method 8082 or 8081.

5.0 SUMMARY OF TEST METHOD

5.1 Set up a Soxhlet extractor apparatus for each sample using 10% ether/hexane as the solvent.

5.2 Load the PUF into the Soxhlet. Add the necessary surrogates and/or matrix spikes.

5.3 Load the extraction apparatus into its heating mantle and condenser and allow it to extract for 18 $\pm$ 2 hours.

5.4 Solvent exchange and set to volume.

5.5 Samples are put through a series of cleanup steps, including sulfuric acid, Florisil slurry, and mercury shake. Submit for GC analysis.

6.0 DEFINITIONS

6.1 **Method Blank (B)**- A method blank is processed with each batch of samples that are extracted to assess for contamination during prep and processing steps. The method blank is carried throughout all stages of sample preparation and extraction steps. A PUF is processed as the method blank.

6.2 **Lab Control Spike (LCS)** - A non-site sample which is prepared in the laboratory, to which a known amount of target analyte is added for assessment of laboratory performance. The laboratory control spike sample is composed of a PUF with the spiked target analytes. The Laboratory Control Spike is processed with each batch of samples extracted.

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

6.4 **Matrix Spike**: A site sample to which a known amount of target analyte is added for assessment of analyte recovery efficiency.

6.5 **Matrix Spike Duplicate**: An exact copy of the Matrix Spike utilizing the same site sample and known amount of target analyte for assessment of analyte recovery efficiency.

6.6 **Surrogate Compound Spike**: In chemical composition and chromatography similar to the analytes of interest. Usually not found in environmental samples. These compounds are spiked into all samples, standards, blanks, and matrix spike samples prior to analysis. Percent recoveries are calculated for each surrogate.

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

7.0 INTERFERENCES

7.1 Laboratory contaminants including phthalate esters may be introduced during extraction and subsequent cleanup procedures. The extraction technician should exercise caution that scrupulously cleaned glassware is used and that plastic tubing and other plastic materials do not contact samples or extracts. Please see determinative methods (Lab SOP NE148, NE131) for details.

8.0 SAFETY

8.1 The chemist should have received in-house safety training and should know the location of first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment. Safety glasses, a lab coat and gloves must be worn when handling glassware and samples.

8.2 Polychlorinated biphenyls have been tentatively classified as known or suspected carcinogens. The chemist must review the Material Safety Data Sheets (MSDS) for PCBs and all reagents used in the procedure before handling them. All equipment and solvents should be handled within a lab fume hood.

9.0 EQUIPMENT AND SUPPLIES

9.1 Water Cooled Condenser: Pyrex 45/50 #3840-MCO.

9.2 250ml Round Bottom Flask: Pyrex #4100.

9.3 Soxhlet Repetitive Flushing (reflux) Unit: 45/50 Pyrex #3740-M.

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

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

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

9.7 Turbo Vap Evaporator: Zymark #ZW640-3.

9.8 Turbo Vap Evaporator concentrator tubes: Zymark 250ml, 0.5ml 1ndpoint.

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

9.10 Vials: glass, 4 dram & 40mL (with Polyseal sealed cap), for sample extracts.

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

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

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

9.14 Pipettes: S/P Disposable Serological Borosilicate Pipits.

1. 1mL X 1/10 #P4650-11X
2. 5mL X 1/10 #P4650-15
3. 10mL X 1/10 #P4650-110
4. Kimble Pasteur Borosilicate glass pipette 9" #72050 (or equivalent)

9.15 Quartz Microfiber Filters (QMF): 10.16 cm Dia., 100 circles (Whatman Cat# 1851-101) (or equivalent) Baked at 450 degrees Celsius for 4 hours. **(Only use with TO-4A)**

9.16 Tweezers: Laboratory stainless steel tweezers used to place PUFs into Soxhlet and to squeeze extracted solvent out of PUFs into Soxhlet.

9.17 Replacement PUFs: 75mm, pre-cleaned and tested. CAT# P226131

9.18 Boiling Chips: Hengar #5785 Alltech Associates, Inc. (or equivalent)

10.0 REAGENTS AND STANDARDS

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

10.2 Diethyl Ether: Nanograde Mallinckrodt #3434-08 (or equivalent)

10.3 Hexane/Ether: 90% hexane/10% ether for TO-4A by volume solvent mixture prepared in the lab. To prepare: Mix 400mL of diethyl ether with 3600mL of hexane in a 4 L bottle.

10.4 Florisil: deactivated (10% Florisil), Deactivated with DI water. EM Science #FX0282-1.

10.5 Mercury: Triple distilled Mercury Refining Co, Albany, NY #328502. (or equivalent)

10.6 Sulfuric Acid: Na_2SO_4 (concentrated) Mallinkrodt #2468 #UN1830. (or equivalent)

10.7 Surrogate Spike Solution: Laboratory prepared from primary stock solution tetra-chloro-meta-xylene 0.05 ug/mL and decachlorobiphenyl at 0.500 ug/mL.

10.8 Laboratory Control Spike Solution: Laboratory prepared from primary stock solution of PCB Aroclor at 1.00 ug/mL

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 Samples are collected as per EPA method TO-4A and the client's field sampling and analysis plan. Pace Analytical does not provide field sample collection services for air monitoring projects. Samples should be stored at < 4 degrees Celsius until shipping to laboratory.

11.2 Field samples are shipped to the laboratory in a cooler chilled with ice (< 4 °C).

11.3 Upon receipt samples are stored in laboratory under refrigeration at < 4 °C until extraction.

11.4 Samples must be extracted within 7 days of collection and analysis must be performed within 40 days of extraction.

12.0 QUALITY CONTROL

12.1 Verification PUF sample:

NOTE: A verification (a.k.a. certification) PUF sample is a cartridge and filter assembly that has been pre-cleaned as described in SOP NE_153 before delivery to field personnel or laboratory client

12.1.1 Extract and prepare one pre-cleaned PUF cartridge/filter assembly at a batch frequency described in the client's sampling/analytical plan. Note: Method TO-4A requires one verification PUF/Filter per extraction batch (or of 10 % of batch whichever greater).

12.1.2 Submit extract for analysis by GC-ECD (EPA Method 8082 and/or 8081) as described in 14.5.

12.1.3 GC analysis of verification PUF must exhibit chromatogram free of PCB Aroclors (< practical quantitation limit) and also be free of interfering non-target co-eluting contaminants. If PUF exhibits either contamination, re-prepare batch according to SOP NE_153. The default practical quantitation limit for Method TO-4A is 0.100 ug total PCB.

12.2 Laboratory Method Blank

12.2.1 A Laboratory method blank sample is prepared and extracted with each site sample extraction batch of up to 20 samples. A pre-cleaned PUF and filter is spiked with surrogate solution and extracted and prepared identically to project samples. The analyte concentration must be less than the practical quantitation limit. If the blank concentration exceeds the PQL the laboratory client is notified and data is qualified (B-flagged) and a case narrative is generated. All analysis must cease until the source of contamination is isolated and the problem is resolved. The default practical quantitation limit for Method TO-4A is 0.100 ug total PCB.

12.3 Laboratory Control Spike/ Laboratory Control Spike Duplicate Sample

12.3.1 A laboratory control spike (LCS)/ laboratory control spike duplicate (LCSD) sample is prepared by spiking a pre-cleaned PUF cassette and filter with aroclor of interest applicable to the project. If the aroclor of interest is unknown rotate the spike between the 7 common aroclors: Aroclor 1016, 1221, 1232, 1242, 1248 1254 and 1260. See table 23.1 for spike information. The percent recovery must meet project specified or laboratory established limits. The default recovery limit is 60-120 %.

12.3.2 Prepare LCS and LCSD samples at frequency specified in the clients sampling and analysis plan. The laboratory default is one LCS, LCSD per batch or 20 site samples whichever is greater.

12.3.3 If the LCS/LCSD does not meet recovery limits the extraction of samples must stop until the problem is identified and corrected. The client is notified and a case narrative is issued to the client along with the affected data describing the LCS failure. Re-extraction of PUF samples is not possible.

12.4 Field Spike Sample

12.4.1 A field spike sample is prepared for each 20 PUF cartridges supplied to field personnel or as the client's field sampling analysis plan requires. The spike is prepared in the same fashion as an LCS sample and is shipped to the field and then returned to the laboratory unopened. The field spike sample is extracted and analyzed with the sample batch. The percent recovery criteria and corrective action are the same as the LCS/LCSD sample described in section 12.3.

12.5 Surrogate Spike

12.5.1 Every site sample and QC sample is spiked with the TCMX/DCBP surrogate solution described in table 23.1. The Surrogate recovery must meet project specified limits or default limits (60-120%). If the surrogate recovery does not meet specified limits then identify the problem, re-analyze extract by GC if necessary and provide case narrative describing the problem along with associated sample concentration results.

12.6 Field Blank Sample

12.6.1 A field blank sample consists of a pre-cleaned cartridge assembly that as

packaged and shipped to field personnel un-opened. The un-opened PUF is returned to the laboratory and analyzed with the sample batch. The PCB concentration should be less than the practical quantitation limit. If PCBs are observed greater than the PQL compare results with laboratory method blank. Notify the client/field personnel of the problem and generate a case narrative that is issued with the analytical results.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Please see determinative methods (Lab SOP NE148, NE131) for details.

14.0 PROCEDURES

14.1 Sample Preparation

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

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

14.1.3 Prior to extraction all surfaces and fume hoods used must be cleaned and wiped down with hexane and then lined with aluminum foil. It is also advisable to remove any PCB solid or liquid waste containers from the fume hood.

14.1.4 PUF samples require all glassware to be pre-rinsed with hexane. PUF samples are for extremely low level PCB concentrations and require clean, hexane rinsed glassware.

14.1.5 Use extreme caution while using ether during this extraction. Ether and its vapors are extremely flammable and must be used in a fume hood.

14.1.6 Prior to being used in the field all TO-4A PUFs and quartz micro fiber filters are pre-cleaned in the lab utilizing SOP NE153.

14.2 Procedure: Sample Extraction

14.2.1 Rinse enough 250 mL round bottoms and Soxhlets for each sample, blank, and QC sample. Place in fume hood and let dry.

14.2.2 After the glassware dries label them with a sample ID. To each round bottom add a few boiling chips and approximately 200 mL of 10% ether/hexane mixture.

14.2.3 Place a Soxhlet onto each round bottom, checking for cracks or chips that would cause solvent to leak out. Record the ID number of each Soxhlet and round bottom in LIMS.

14.2.4 Blank, QC PUFs, and baked QMF filters (filters only for TO-4A) should be prepared prior to extraction using the PUF preparation SOP (NE153). Each sample must be handled using a clean pair of tweezers. Use the pre-cleaned replacement PUFs and pre-baked QMF filters for the Blank and QC samples.

14.2.5 For each sample, use a pair of tweezers to pull the PUF out of its PUF cartridge and push it into the appropriate Soxhlet. Try to handle as PUF as little as possible. **Rinse tweezers with Hexane in between handling each sample.**

14.2.6 Using tweezers, fold the glass fiber filter that came with the sample and push it into the Soxhlet. Use the tweezers to push the filter down to the PUF. Be sure that both the PUF and the filter are below the capillary tube on the soxhlet to ensure proper drainage of the Soxhlet during extraction.

14.2.7 Spike surrogate and spike compound solutions directly into the Soxhlet onto the PUF. The addition of spiking material to a sample, blank, or QC must be witnessed by another extraction technician. Record the names of the technicians spiking and witnessing, surrogate and spike concentration, the amount spiked, and the spike solution reference code in LIMS.

14.2.8 Rinse the inside and the outside connecting joints of the condenser units that will be used with hexane. Turn on chiller to cool the condensers.

14.2.9 Place the round bottom flask with attached Soxhlet extractor onto a heating mantle and attach condenser unit. Turn corresponding control units on to setting 3.5. At this time, double check the Soxhlets for cracks or chips. Once the solvent begins to boil, a flushing action of three or more flushes per hour should be achieved.

14.2.10 The samples should be extracted for 18 hours $\pm$ 2 hours, usually overnight. Once the sample has finished extracting (usually in the morning), turn the heating mantle off and allow samples to cool to room temperature.

12.2.11 Once cool, disengage the Soxhlet from the condenser and move all round bottom/ Soxhlet units to a fume hood. Using hexane pre-rinsed tweezers remove the filter, and then squeeze the extracted solvent from the PUF into the Soxhlet. Tip the Soxhlet/round bottom unit to get the solvent in the Soxhlet to drain into the round bottom.

12.2.12 Rinse Soxhlet with Hexane and tip to allow the unit to drain into the round bottom. Disconnect the Soxhlet from the round bottom and rinse the connecting joint of the Soxhlet into the round bottom. Set the Soxhlet aside at this time and leave it in the hood to dry.

12.2.13 Label turbo tubes with sample ID, one per sample, and record the ID of each Turbo Tube in LIMS. Rinse turbo tubes with hexane and allow them to dry in the

hood.

12.2.14 Add sodium sulfate to each round bottom, swirling contents. Add as much sodium sulfate as necessary until the drying agent is free flowing.

12.2.15 Pour the contents of the round bottom into the turbo tube, decanting off the sodium sulfate. Using a pipette and hexane, rinse the last drop out of the mouth of the round bottom.

12.2.16 Add three Pasteur pipettes full of hexane to the round bottom. Swirl gently, and decant into same turbo tube. Repeat twice more for each sample.

12.2.17 All glassware must be rinsed with acetone, and dried in the hood before being washed as per NE256.

14.3 Solvent Reduction: TurboVap Evaporator System

14.3.1 The TurboVap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The TurboVap evaporator system is used to reduce the sample volume. The TurboVap uses a heated water bath and positive pressure nitrogen flow / vortex action. The unit maintains a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract, which allows fractional reduction of the solvents without loss of higher boiling point analytes.

14.3.2 Turn the unit on and allow to heat up to 40°C $\pm$ 2°C.

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

14.3.4 Wipe down inside of TurboVap with a Hexane wetted paper towel including top lid and pins. Place TurboTubes containing the sample extracts into the TurboVap and close lid. Slowly open the pressure regulator. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces, maintaining uniform flow throughout the volume reduction.

14.3.5 The process for solvent (hexane/ether) reduction takes approximately 30-45 minutes. Do not leave the unit unattended as extracts may be blown to dryness and PCB loss may occur. Immediately notify a supervisor if an extract is blown to dryness.

14.3.6 Concentrate the solvent to approximately 2.5 mL. Remove the samples from the TurboVap and place in a rack.

14.3.7 Using a disposable Pasteur pipette, transfer the sample extract into a pre-rinsed volumetric. PUF samples are generally set to 5X, but client specifications may require a different set volume. Rinse Turbo Tube with two Pasteur pipette volumes of hexane, and transfer the rinse to the volumetric.

14.3.8 Add hexane to the volumetric meniscus mark. Decant the contents into a pre-labeled 4 dram vial. At this time samples for pesticide analysis may be handed in for analysis.

14.4 Sample Extract Cleanup

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

14.4.2 Not all clean-up procedures need to be performed on every sample and several are sample matrix specific. The experience of the analyst combined with the sampling site history should guide the selection of which clean-up procedures are necessary. The sequence and number of repeats of cleanup steps performed are recorded by the sample preparation chemist in LIMS.

14.4.3 Samples to be analyzed for pesticides should **NOT** undergo the following cleanup steps. Once the sample is set to volume it can be handed in for analysis.

14.4.4 Sulfuric Acid Wash

14.4.4.1 Sulfuric acid removes hydrocarbons and other organic compounds which are co-extracted with the PCB residues.

14.4.4.2 Add approximately 3mL of concentrated H_2S0_4 to vial and shake for 30 seconds by hand. Centrifuge for approximately 1 minute on a speed setting of ¾.

14.4.4.3 Transfer the hexane layer (top layer) to a new properly labeled 4 dram vial.

14.4.5 Florisil Absorption (Slurry)

14.4.5.1 The Florisil slurry removes co-extracted polar compounds, residual water, and residual acid.

14.4.5.2 Add one spatula (approximately 0.5g) of tested and approved 10% Florisil to each extract vial.

14.4.5.3 Vigorously shake the vial for approximately 30 seconds by hand.

14.4.5.4 Allow the Florisil to settle before proceeding on to the next step.

14.4.5.5 Transfer the hexane layer to a new properly labeled 4 dram vial.

14.4.6 Elemental Sulfur Clean-up

14.4.6.1 Elemental sulfur is soluble in the extract solvents used for PUF samples. It is commonly found in many PUF/sediment/soil samples, decaying organic material, and some industrial wastes. Large amounts of sulfur can cause the electron capture detector (ECD) to signal saturate for long periods during the elution envelope of PCBs. Even small amounts of sulfur can interfere with PCB measurement as a co-eluting chromatographic peak. PUF samples normally have less sulfur than sediment/soil samples.

14.4.6.2 Two techniques exist for the elimination of elemental sulfur in PCB extracts: mercuric precipitation (mercury shake) and the tetrabutylammonium (TBA) sulfite procedure. Tetrabutylammonium sulfite causes the least amount of degradation of a broad range of pesticides and organic compounds, while mercury may degrade organophosphorus and some organochlorine pesticides. The TBA procedure also has a higher capacity for samples containing high concentrations of elemental sulfur.

14.4.6.3 Removal of Sulfur Using Mercury

14.4.6.3.1 Add 1-3 drops of mercury to the sample extracts, cap, and place on the wrist shaker for 30 minutes. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental sulfur.

14.4.6.3.2 Transfer the extract a properly labeled 4 dram vial.

14.5 Final Extract

14.5.1 From the final extract vial pipette approximately 1mL into a white screw top vial.

14.5.2 Complete all information in LIMS. Submit samples and project folder to the GC Analyst.

15.0 CALCULATIONS

15.1 Please see determinative method (Lab SOP NE148) for details.

16.0 METHOD PERFORMANCE

16.1 Please see determinative method (Lab SOP NE148) for details.

17.0 POLLUTION PREVENTION

17.1 See SOP NE168.

18.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

18.1 Please see determinative method (Lab SOP NE148) for details.

19.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

19.1 Please see determinative method (Lab SOP NE148) for details.

20.0 CONTINGINCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1 Please see determinative method (Lab SOP NE148) for details.

21.0 WASTE MANAGEMENT

21.1 See SOP NE054 for details.

22.0 REFERENCES

22.1 US-EPA SW-846 Test Methods for Solid Waste; Soxhlet Extraction Method 3540C; United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Vol.1B, Cincinnati, OH 45268. December 1996.

22.2 US-EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition Compendium Method TO-4A Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using High Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MS) 3/18/99

22.3 Guide to Environmental Analytical Methods, Genium Publishing Corporation, Schenectady, NY 12304. 1997

23.0 TABLES, DIAGRAMS, FLOWCHARTS and VALIDATION DATA

23.1 Surrogate and Spike Additions for PCBs

23.2 Method Outline Summary

23.1 Table 1. Surrogate and Spike Additions

Fortification Mixture	Concentration	Volume added to Samples
TCMX/DCBP Surrogate mix in hexane	0.05 ug/mL TCMX/ 0.5 ug/mL DCBP	0.500 mL
Aroclor 1016 Spike mix in hexane	1.000 ug/mL	1.000 mL
Aroclor 1221 Spike mix in hexane	1.000 ug/mL	1.000mL
Aroclor 1232 Spike mix in hexane	1.000 ug/mL	1.000 mL
Aroclor 1242 Spike mix in hexane	1.000 ug/mL	1.000 mL
Aroclor 1248 Spike mix in hexane	1.000 ug/mL	1.000 mL
Aroclor 1254 Spike mix in hexane	1.000 ug/mL	1.000 mL
Aroclor 1260 Spike mix in hexane	1.000 ug/mL	1.000 mL

23.2 METHOD OUTLINE FOR PUF EXTRACTION USING SOXHLET TECHNIQUE

1. PREPARE FUME HOOD AND SAMPLES FOR EXTRACTION
2. RINSE GLASSWARE AND LET DRY
3. SET UP SOXHLET EXTRACTOR APPARATUS
4. ADD SURROGATES AND/OR MATRIX SPIKE
5. EXTRACT SAMPLE FOR APPROXIMATELY 18 HOURS +/- 2hours
6. BREAKDOWN SOXHLET EXTRACTOR APPARATUS
7. TRANSFER SOLVENT TO TURBO TUBE
8. SOLVENT REDUCTION, USING THE TURBOVAP EVAPORATION SYSTEM
9. SET TO VOLUME
10. EXTRACT CLEANUP (ACID, FLORISIL, MERCURY)
11. GC SCREENING/ ANALYSIS

STANDARD OPERATING PROCEDURE REVIEW

SOP Name	Review Number	Reviewers	Title	QAO Approval	Effective Date
NE151_07	00	Jill Grygas Christina L. Braidwood Robert E. Wagner	Extraction Supervisor QAO Lab Director	Christina Braidwood	03/29/11
NE151_08	00	Jill Grygas Kelly Sableski Dan Pfalzer	Extraction Supervisor QAA AGM/Lab Director	Christina Braidwood	06/05/12

Attachment J-2

Determination of PCBs Aroclors

STANDARD OPERATING PROCEDURE

DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBS) AROCLORS

Reference Methods: SW-846 EPA METHOD 8082

LOCAL SOP NUMBER:	NE148_08
EFFECTIVE DATE:	05/11/2012
SUPERSEDES:	NE148_07
SOP DOCUMENT NUMBER:	S-NY-O-148-rev.08

APPROVALS

Dan Pfalzer
Assistant General Manager

Date

05/10/2012

Christina L. Braidwood
Quality Manager

Date

PERIODIC REVIEW

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

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1.0 Identification of Test Method

1.1 This Standard Operating Procedure (SOP) is used to determine Polychlorinated Biphenyl (PCB) by gas chromatography with electron capture detection and Total Aroclor Quantification using EPA SW846 Method 8082- Polychlorinated Biphenyl (PCB) Aroclors by Capillary Column GC.

2.0 Applicable Matrix

2.1 This SOP is applicable in the determination and quantification of PCBs as outlined in EPA SW-846 Method 8082. It is applicable to the following matrices: water, soil, sediment, sludge, oil, fuel oil, waste solvent, fish, other aquatic animals, tissue samples, caulk, and air cassette samples including polyurethane foam (PUF) and associated filters for EPA Methods TO-4A and TO-10A.

3.0 Detection Limit

3.1 Detection Limit: Reporting Limits (RLs) and Method Detection Limits (MDLs) vary for each compound.

3.1.1 The following are default Reporting Limits based on the lowest calibration standard and global MDL /Reporting Limits based on MDL studies performed by Pace Analytical Services used for EPA Method 8082. Reporting Limits (PQLs) are based on the Lowest Calibration Standard.

Matrix	Sample Mass/Volume Extracted	Calibration Curve Low Standard	Extract Volume	Global MDL* (All Aroclors)	RL (PQL) (all Aroclors)
Soil/Sediment Solid	10 g	20 ng/ml	25 mL	*	0.050 mg/kg
Water	1 Liter	5 ng/ml	10 mL	*	0.050 ug/L
Biota	10 g (wet weight basis)	20 ng/mL	25 mL	*	0.050 mg/kg
Polyurethane Foam Cassette (TO-4A/TO 10A)	1 PUF	20 ng/ml	5 mL	*	0.100 ug/Puf
Waste Oil	0.5 g	20 ng/ml	25 mL	*	1.00 mg/kg
Wipe	1 Wipe	20 ng/ml	25 mL	*	0.500 ug/wipe

* Global MDL values can be obtained by request from the Quality Control department.

3.2 Individual MDLs and RLs are determined every two years for each instrument with matrix specific MDL studies for each extraction methodology. MDLs must be determined again whenever a major change in instrumentation or extraction methodology occurs.

3.3 MDLs are verified annually by the extraction and analysis of a low level MDL verification check sample. The Aroclor must be observed qualitatively in the MDL verification check sample.

4.0 Scope and application, including components to be analyzed

4.1 This SOP is applicable in the determination and quantification of PCB in the following matrices as outlined in EPA SW-846 Method 8082 : water, soil, sediment, sludge, oil, fuel oil, waste solvent, fish, other aquatic animals, tissue samples, caulk, wipes and air cassette samples including polyurethane foam (PUF) and associated filters for EPA Methods TO-4A and TO-10A.

4.2 In general, samples are extracted, or in the case of oils and waste solvent, diluted with a pesticide grade solvent. Applicable extraction methods for solids and animal tissues include: SW-846 Method 3540 (Soxhlet), SW-846 Method 3545 (Pressurized

Fluid Extraction), or SW-846 Method 3550 (Ultrasonic Extraction). Extraction methods for aqueous samples include SW-846 Method 3510 (Separatory Funnel), and SW-846 Method 3520 (Continuous Liquid Liquid Extraction). The extracts are further processed by concentrating or diluting, depending on the PCB concentration, and carried through a series of clean-up techniques. Applicable cleanup techniques include SW-846 Method 3620 (Florisil Cleanup), SW-846 Method 3660 (Sulfur Cleanup) and SW-846 Method 3665 (Sulfuric Acid/Permanganate Cleanup). The sample is then analyzed by direct injection onto a gas chromatographic system and detected by an electron capture detector.

4.3 This method provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCB by capillary column gas chromatography. Each matrix requires different sample handling or special preparation procedure before analysis can be performed. Each sample matrix will be covered separately in the extraction standard operating procedures.

5.0 Summary of Test Method

5.1 Samples are extracted with a pesticide analytical grade solvent. The extracts are further processed by concentration and a series of clean-up procedures. The sample extracts are then analyzed by injecting onto a gas chromatographic system and with an electron capture detector in series.

5.2 This purpose of this SOP is to provide a detailed written document for quantification of PCB according to SW-846 Method 8082 specification.

5.3 This SOP provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCBs by gas chromatography. Sample extraction and cleanup procedures are described separately in additional laboratory Standard Operating Procedures.

5.4 The following PCB Aroclors can be determined by this method*:

Compound	CAS Number
Aroclor 1016	12674-11-2
Aroclor-1221	11104-28-2
Aroclor-1232	11141-16-5
Aroclor-1242	53469-21-9
Aroclor-1248	12672-29-6
Aroclor-1254	11097-69-1
Aroclor-1260	11096-82-5

*Note: Additional PCB Aroclor mixtures including Aroclor 1262 and Aroclor 1268 may be analyzed by this method with minor modifications

5.5 Extensive knowledge of this SOP and EPA Method 8082 is required. The analysis portion of this method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.

6.0 Definitions

6.1 Accuracy – The nearness of a result or the mean of a set to the true value. Accuracy is assessed by analysis of references samples and percent recoveries.

6.2 Analytical Batch –The basic unit for analytical quality control is the analytical batch, which is defined as samples which are analyzed together with the sample method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar matrices (e.g. water, sediment, soil, etc.).

6.3 Blank – A blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix, however, a universal blank matrix does not exist for solid samples, but sometimes sodium sulfate is used as a blank matrix. The blank is taken through the appropriate steps of the process. A reagent blank is an aliquot of analyte-free water or solvent analyzed with the analytical batch. Field blanks are aliquots of analyte-free water or solvents brought to the field is sealed containers and transported back to the laboratory with the sample containers. Trip blanks and equipment blanks are two specific types of field blanks. Trip blanks are not opened in

the field. They are a check on sample contamination originating from sample transport, shipping and from site conditions. Equipment blanks are opened in the field and the contents are poured appropriately over or through the sample collection device, collected in a sample container, returned to the laboratory as a sample. Equipment blanks are a check on sampling device cleanliness.

6.4 Continuing Calibration Check Standard (CCCS) –The continuing calibration check standard contains all target analytes found in the calibration standards and is used to verify that the initial calibration is prepared correctly and that the instrument system is correctly calibrated. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards

6.5 Calibration Standard (ICAL)– A series of known standard solutions used by the analyst for instrument calibration. Calibration standards are prepared from primary standard and/or stock standard solutions.

6.6 CAS Number – An assigned number used to identify a chemical. CAS stands for Chemical Abstracts Service, an organization that indexes information published in Chemical Abstracts by the American Chemical Society and that provides index guides by which information about particular substances may be located in the abstracts. Sequentially assigned CAS numbers identify specific chemicals, except when followed by an asterisk (*) which signifies a compound (often naturally occurring) of variable composition. The numbers have no chemical significance. The CAS number is a concise, unique means of material identification. (Chemical Abstracts Service, Division of American Chemical Society, Box 3012, Columbus, OH 43210: [614] 447-3600).

6.7 Duplicate– A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

6.10 Environmental Sample – An environmental sample or field sample is a representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination as requested or required.

6.11 Initial Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the analytical detector or method.

6.12 Instrument Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

6.13 Laboratory Control Sample (LCS) – Also known as the Quality Control (QC) Check Standard or Quality Control (QC) Check Sample. The LCS consists of an aliquot or reagent water or other blank matrix to which known quantities of the method analytes are added. The LCS is extracted and analyzed exactly like a field sample, and its purpose is to determine whether the analysis is in control and whether the laboratory is capable of making accurate and precise measurements.

6.14 Laboratory Method Blank – An analytical control consisting of all reagents and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

6.15 Matrix – The predominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

6.16 Matrix Spike – Aliquot of sample (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

6.17 Matrix Spike Duplicate – A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

6.18 Method Detection Limit (MDL) – The minimum constituent concentration that can be measured and reported with 99% confidence that the signal produced is different from the blank in a given matrix. The MDL is determined from a minimum of seven replicate samples, taken through the entire preparation and analysis procedure. The standard deviation, *s*, of those replicates is multiplied by a student's t factor in order to calculate the MDL.

6.19 MSDS – Material Safety Data Sheet. OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs.

6.20 PCB Polychlorinated biphenyls (PCBs) are a class of 209 individual chemical compounds (congeners), in which one to ten chlorine atoms are attached to biphenyl. Use of PCBs has made them a frequent environmental pollutant.

6.21 Precision – The agreement between a set of replicate measurements without assumption of knowledge of the true value. Precision is assessed by means of duplicate/replicate sample analysis.

6.22 Quality Control – Set of measures within a sample analysis methodology to assure that the process is in control.

6.23 Standard Curve – A standard curve is a curve which plots concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by diluting the stock analyte solution in graduated amounts which cover the expected range of the samples being analyzed. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

6.24 Stock Solution – Standard solution which can be diluted to derive the other standards.

6.25 Surrogate – Organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, calibration and check standards, samples (including duplicates and QC reference sample) and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

6.26 Surrogate Standard – A pure compound added to a sample in the laboratory just before processing so that the overall efficiency of a method can be determined.

7.0 Interferences

7.1 Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters exhibit response on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Laboratory method blanks must be thoroughly reviewed for presence of non-target peaks and comparison of samples with blank chromatographic patterns.

7.2 Elemental sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur can be removed through the use of Method 3660.

7.3 Polychloroterphenyls (PCTs), polybrominatedbiphenyls (PBB), polychlorinated naphthalenes (PCN), as well as dioxins can co-elute with PCBs. Carry-over from these compounds, when in high concentration, is common if clean-up procedures are not followed. These materials may be removed through the use of specified clean-up procedures.

7.4 Pesticides can be a source of contamination through breakdown into components such as hexachlorobenzene (HCB). This chlorinated compound can carry-over on the GC column, and contaminate samples. Specified clean-up procedures should be followed to eliminate this as a source of contamination when analyzing PCBs. High concentrations of pesticides can cause carry-over on GC columns.

8.0 Safety

8.1 Safety glasses and disposable gloves must be worn when handling samples and extracts.

8.2 All manipulations of sample extracts should be conducted inside a chemical fume hood. Manipulation of sample extracts outside of a fume hood should be minimized by the analyst.

8.3 Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize themselves with the precautions for handling solvents and chemicals used to process samples. The analyst should refer to the laboratory chemical hygiene plan for further safety information.

8.4 Samples remaining after analysis should either be returned to the customer for disposal or disposed of through the laboratory's disposal plan. Refer to the sample custodian for assistance and also standard operating procedure NE054, disposal of laboratory waste.

9.0 Equipment and Supplies

9.1 Gas Chromatograph: Complete system for high resolution, capillary column capability and all required accessories. Pace Analytical Services, Inc. will use a Varian Models 3400, 3800 and 450 (or equivalent) gas chromatograph (or equivalent), equipped with a Model 1177 split/splitless injector (or equivalent), temperature programmable oven, Varian Model 8200, Varian Model 8400, or LEAP GC pal automatic sampler (or equivalent), and electron capture detector (or equivalent). A data system and integration of detector signal is interfaced to the gas chromatograph.

9.2 Chromatographic Data System: A data system for measuring peak height and peak area. An Empower computer network based workstation (Waters Corporation), will be employed to capture detector response and digitally store the chromatographic, electronic peak integration for precise calculations, database structuring of the analytical information, and archival capabilities.

9.3 Column (Primary Helium Carrier Gas): ZB-1, Phenomenex Cat. No. 7HG-G001-11; 30 m x 0.25mm x 0.25 um ; DB-1, J&W Part No. 122-1032; 30 m x 0.25 mm x 0.25 um ; or equivalent.

9.4 Column (Secondary Helium Carrier Gas): ZB-5, Phenomenex Cat. No. ZB-5-G002-11; 30m x 0.25mm x 0.25 um ; DB-5, J&W Part No. 122-5032; 30m x 0.25 mm x0.25 um; or equivalent.

9.5 Column (Primary Hydrogen Carrier Gas): ZB-1MS, Phenomenex Cat. No 7FD-G011-08; 20m x 0.18mm x 0.18um

9.6 Column (Secondary Hydrogen Carrier Gas): ZB-5, Phenomenex Cat. No 7FD-G002-08; 20m x 0.18mm x 0.18um

9.7 Class A volumetric flasks: 5.0 – 100mL.

9.8 8 dram vials and 4 vials dram for sample extract storage.

9.9 Pasteur pipettes.

9.10 250ml and 100ml beakers, glass.

9.11 Disposable 1.0, 5.0, and 10.0 ml pipettes.

9.12 Hexane, Burdick and Jackson-Pest Grade.

9.13 Acetone, Burdick and Jackson.-Pest Grade

9.14 Toluene, Baker, (Cat.No. 9336-03)

9.15 Methylene Chloride, Burdick and Jackson, (Cat. No. 300-4)

9.16 Ferrules: 0.4mm graphite/vespel, Restek 20229, and ¼" graphite ferrules, Restek 20210 or equivalent.

9.17 Injector septa: Thermolite Septa, Restek 20365 or equivalent.

9.18 Injector liner: Low Pressure Drop Liner w/Glass Wool, Restek 21033 or equivalent.

9.19 SGE Injector Syringe 10.0 µL: SGE 002987 or equivalent.

9.20 Auto sampler vials: Snap vial 12x32mm Clear w/P, Microliter 11-5200.

9.21 Snap Caps: 11mm Natural Snap Cap PTFE, Microliter 11-0051N-B.

10.0 Reagents and Standards

10.1 Analytical Standard Solutions.

10.1.1 Aroclor Stock Standard Solutions

10.1.1.1 Polychlorinated Biphenyls - Neat commercial material for standard preparation. These materials are multi-component mixtures of PCB congeners and are the actual materials that were used in products such as electric power transformers and capacitors. Monsanto was the largest producer of PCB formulations and sold them under the trade name Aroclor. These standards should be compared to PCB reference materials to verify commercial materials. To be used as calibration standards, they must have the same pattern and congener distribution.

10.1.1.2 Stock standards are prepared from individual Aroclor formulations by weighing an exact amount of the neat material to the nearest 0.1 g, and dissolving and diluting to volume in a 100 mL volumetric flask with hexane. See Attachment A, Table 1 for exact weights of each compound.

10.1.1.3 The stock standards are transferred into Boston bottles and stored in a refrigerator at 0-6°C, protected from light.

10.1.1.4 The stock standards are transferred into screw-cap boston bottles and stored in a freezer 0°C , protected from light. Stock standards should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards. Stock PCB standards must be replaced after one year, or sooner if a problem with instrument calibration is detected.

10.2 Calibration Standards

10.2.1 Calibration standards are prepared at five concentration levels using a prepared working standard. See Attachment A, Tables 2 and 3A AND 3B for the preparation and exact concentrations of the working standards. The following five standards make up the initial calibration curve standard set for a High Level curve : 20 ng/mL, 100 ng/mL, 250 ng/mL, 500 ng/mL, 1000 ng/mL. The following five standards make up the initial calibration curve set for a Low Level curve: 5 ng/mL, 10 ng/mL, 20 ng/mL, 50 ng/mL, 100 ng/mL.

10.2.1 The two surrogates Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) are included in the A1254 calibration standards. The standard for TCMX/DCBP is prepared by diluting 1 mL of TCMX/DCBP custom standard solution (ULTRA, cat.#CUS-4911, at 500/5000 ng/mL) into a 1000 mL volumetric flask resulting in a solution of TCMX/DCBP at 0.5/5.0 PPM

10.2.2 Refer to Attachment A, Tables 4A and 4B for instructions on preparation of the calibration standards containing A1254 and the surrogates. Refer to Attachment A, Tables 3A and 3B for instructions on preparing the remaining calibration standards.

10.2.3 Transfer all calibration standards to ASE vials and store in a refrigerator at 0-6°C, protected from light. Calibration standards must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

10.3 Continuing Calibration Standards:

10.3.1 The surrogate compounds Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) are included in all Continuing Calibration Check Standards at a concentration near the mid-point of the surrogate calibration

curve sequence. All continuing calibration standards are prepared independently from calibration standards, by using an alternate source purchased from standard vendors. Refer to Attachment B, Tables 1-3 for instructions on preparation of these standards.

11.0 Sample Collection, Preservation, Shipment and Storage

11.1 Sample Collection and Preservation:

11.1.1 Routine soil, sediment, sludge, solid, caulk, and concentrated liquid samples should be collected in 8 oz clear glass wide-mouth jars, fitted with a Teflon-lined cap. Aqueous samples should be collected in 1 liter amber glass bottles with a Teflon-lined cap. Project specific protocols may require that containers be pre-cleaned to EPA specification protocol A –. Protect samples from light.

11.1.2 All samples must be placed on ice or refrigerated at >0-6°C from the time they are collected until delivery to the lab. Samples that are collected within driving distance of the laboratory and delivered the same day may not have reached temperature acceptance limits. These samples are deemed acceptable if evidence of cooling is present (i.e. they are received with ice in the cooler)

11.2 Sample Shipment:

11.2.1 Sample Shipment is accomplished through a carrier such as Federal Express or United Postal Service for overnight 1-day delivery to the lab. Shipment is normally handled by the field personnel collecting the samples and coordinated with sample receiving department at the lab. Samples can also be picked up by the lab courier service if samples are collected within driving distance to the lab.

11.3 Sample Storage:

11.3.1 The samples must be protected from light and refrigerated at >0-6°C from time of receipt until they are removed from storage for extraction. Remaining sample material will be stored protected from light and refrigerated at >0-6°C. Sample will be disposed of or stored / archived according to project specifications.

11.3.2 Routine soil, sediment, sludge, solid, liquid and concentrated liquid samples are stored in a refrigerator dedicated for this type of sample.

11.4 Sample Extract Storage:

11.4.1 Sample extracts must be protected from light and refrigerated at >0-6°C during the analysis. After analysis is complete, sample extracts will be discarded after 60 days or can be archived in a freezer at less than -20°C for longer periods of time depending on the program requirements.

11.4.2 Field samples, sample extracts, and calibration standards must be stored separately.

11.5 Required Hold Times

11.5.1 Extraction of solid samples by appropriate technique must be completed within fourteen days from sample collection.

11.5.2 Extraction of aqueous samples by appropriate technique must be completed within seven days from sample collection.

11.5.3 Sample extracts must be analyzed within forty days of sample extraction.

12.0 Quality Control

12.1 This section outlines the necessary quality control samples that need to be generated at the time of sample extraction. The results of the quality control measurement samples document the quality of the data generated. The following table lists the Quality Control samples required for capillary gas chromatography analysis of PCBs.

Quality Control Requirements

QC Sample	Frequency
Method Blank	With each sample batch (up to 20 samples)
Lab Control Spike	With each sample batch (up to 20 samples)
Cont Cal Check Std	Analyzed prior to each sample batch and at a frequency or one per ten injections. Each analytical sequence must close with a Continuing Calibration Check Standard (CCCS).
Duplicate Analysis	Field generated sample – analyzed at discretion of client.
Matrix Spike	One matrix spike per 20 field samples or designated sample batch may be performed as specified in the client site plan.
Matrix Spike Duplicate	One matrix spike duplicate per 20 field samples or designated sample batch may be performed as specified in the client site plan.

12.2 Method Blank

12.2.1 With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples and organic-free reagent water blank is processed. The method blank must exhibit PCB levels less than the matrix defined reporting limit (RL). If the method blank exhibits PCB contamination above the reportable RL, the samples associated with the contaminated blank should be re-extracted and analysis repeated. If there is no original sample available for re-extraction then the results should be flagged with a "B" indicating blank contamination. The value measured in the blank is reported for those samples associated with the particular blank out of criteria.

12.3 Laboratory Control Spike

12.3.1 A Laboratory Control Spike (LCS), also referred to as a QC reference check standard, is extracted with each batch of samples at a rate of one per 20 samples. For water sample, spike one liter of laboratory organic free water, extract and analyze. For solid and tissue samples spike 10 grams of sodium sulfate, extract and analyze. For oil samples spike 1 gram of PCB free oil, extract and analyze. An Aroclor is chosen for the LCS analyte, typically based on program requirements or expected sample contamination. Calculate the percent recovery for the PCB spike. If the percent recovery for the LCS is out of criteria, (70%-130%) the analysis is out of the control and the problem should be immediately corrected.

12.3.2 The following are default Laboratory Spikes Concentrations:

Aqueous Samples: .1.0 mL of A1242 @ 0.5 ug/mL (ppm) yielding a final sample concentration of 0.500 ug/L

Solid Samples: 1.0 mL of A1242 @ 12.5 ug/mL (ppm) yielding a final sample concentration of 10 ug/g

Note: Alternate spike concentrations and selection of Aroclors may be applicable based on project specific requirements.

12.4 Duplicate Analysis

12.4.1 Duplicate analysis of the same sample is performed to assess method precision. A duplicate can also be performed as a blind duplicate, so that identification with original sample is withheld. The analysis of a duplicate sample precludes that PCBs are to be found at appreciable levels in samples. If this is not known the analysis of matrix spike/matrix spike duplicates provide more consistent quality control information. The relative percent difference of the two measurements on the sample is calculated on total PCB concentration by the following equation:

RPD = (DUP1-DUP2)/AVG x 100

Where: RPD = Relative Percent Difference
DUP1 = The greater of the measured values
DUP2 = The lesser of the measured values
AVG = Average of the two analysis

The relative percent difference must be less than or equal to 30%.

12.5 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

12.5.1 A matrix spike is to be analyzed at a rate of one matrix spike per every 20 samples. Also matrix spike duplicate or duplicate sample is to be analyzed at a rate of one per every 20 samples. Duplicate samples may be appropriate in place of matrix spike duplicate, for soil and waste samples, where detectable amounts of organics are present.

12.5.2 The following are default Laboratory Matrix Spike Concentrations:

Aqueous Samples: 1.0 mL of A1242 @ 0.5 ug/mL (ppm) yielding a final sample added concentration of 0.500 ug/L

Solid Samples: 1.00 mL of A1242 @ 100 ug/mL (ppm) yielding a final sample added concentration of 10 ug/g

Note: Alternate spike concentrations and selection of Aroclors may be applicable based on project specific requirements.

12.5.3 Analyze one unspiked and one spiked sample. Calculate the percent recovery based on PCB concentration of both samples as follow:

P = A-B/T x 100

Where: P = Percent recovery, %
A = concentration of analyte in the spike sample aliquot
T = Know true values of the spike concentration
B = Background concentration of PCB in the unspiked sample aliquot

12.5.4 Matrix spike recovery information is used to assess the long-term precision and accuracy of the method for each encountered matrix. Matrix spike/matrix spike duplicate results are not used alone to qualify an extraction batch. Generally, percent recovery for MS/MSD samples should be greater than or equal

to 70% and less than or equal to 130% based on the total PCB concentration. If the percent recovery is outside the limits, all calculations should be checked and the data should be narrated to describe possible matrix interference.

12.6 Surrogates

12.6.1 A surrogate compound is added to each sample, matrix spike, matrix spike duplicate, duplicate, method blank, and LCS at time of extraction. The surrogate compounds chosen for this method are Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP). The following are typical surrogate amounts added to normal encountered matrices. These amounts can be adjusted if the PCB background levels are high and the surrogate is being diluted out of analysis range.

12.6.1.1 Soil, sediment, sludge, oil, fuel oil, waste solvent, fish, other aquatic animals, tissue samples: 0.5ml of 0.5ppm TCMX/ 5.0ppm DCBP set to 25ml final extract volume.

12.6.1.2 Water: 1.0ml of 0.05ppm TCMX/ 0.5ppm DCBP set to 10ml final extract volume.

12.6.2 Only one surrogate analyte needs to meet established control limits for the analysis to be valid. The recovery must fall within lab established limits of 60-140% if lab limits are not available for the analysis to be valid. If percent surrogate recovery is not within laboratory established limits for either surrogate, the following steps are required.

12.6.2.1 Review calculations that were used to generated surrogate percent recovery values to make certain there are no errors.

12.6.2.2 Check by GC analysis surrogate solutions used during sample extraction steps to ensure that no problems exist with spiking solutions.

12.6.2.3 Review data for chromatographic interferences.

12.6.2.4 Re-extraction and/or re-analysis of samples may be indicated if problems persist with surrogate recoveries. If the surrogate percent recovery is out of limits on the re-extracted samples, low or high surrogate recovery is due to matrix affects and the data can be reported as estimated. If above steps do not lead to satisfactory results then consult with organics manager to resolve the situation.

12.7 Continuing Calibration Check Standard (CCCS)

12.7.1 The initial CCCS is from an alternative source independent of the calibration check standards. It is prepared at a concentration approximately equal to the midlevel standard. This standard is analyzed after the initial calibration standards, every tenth injection, and at the end an analytical sequence. One check standards must be run with a 24 hour time period. The percent recover must be ±15% of the true value.

12.7.2 If the criterion is exceeded, the analyst should inspect the system to determine the cause and perform maintenance as necessary. The system can then be recalibrated and sample analysis can proceed. Note that all samples which are not bracketed by valid check standards must be re-analyzed when the system is in-control.

12.8 Retention Time

12.8.1 The retention time (RT) windows are established from the Continuing Calibration Check Standard (CCS) peak retention times. The CCS is analyzed three times over a 72-hour period and the standard deviation is calculated from the three retention time measurements. The standard deviation is multiplied by three and this establishes the retention time window for each quantified peak (±3SD). Use the retention time for a peak in the continuing calibration check standard to determine the mid point of the retention time window for the analysis sequence. If the continuing calibration checks fall outside of

these windows update the windows using the previous check standard. If the retention times are still outside the established windows instrument maintenance must be performed and recalibration may be required.

12.8.2 This function is performed in the chromatography software graphically as vertical dropdown retention time markers with retention time window brackets. Besides using the retention time window to assign peaks for quantification, the analyst should also rely on their experience in pattern recognition of multi-response sample analysis.

12.8.3 See attachment E for an example of calculated retention time windows.

12.9 Analytical Sequence Queue:

12.9.1 The following is an example of the order that initial calibration standards, continuing calibration check standards, method blanks, QC samples, and samples are placed in an analytical sequence. A continuing calibration check standard is run after every nine samples in the analytical sequence. All analytical sequences must end with a continuing calibration check standard regardless of the number of samples. Below is an example of an analytical sequence:

Injections	Material Injected
1-2	Hexane Blank
3-47	Initial Calibration Standards
48	Hexane Blank
49-57Continuing Calibration Check Standard	
58-66	Samples analyses, including method blanks, matrix spikes, matrix duplicates, matrix spike duplicates, and QC reference check standard. A maximum of nine samples between continuing calibration check standards.
67	Continuing calibration check standard
68 and higher	repeat inject. 58-66 sequence

12.10 PCB Aroclor Qualitative Identification and Secondary GC Column Confirmation:

12.10.1 Positive identification of PCB Aroclors is based on comparison of retention time of the five selected quantitation peaks and major non-quantitation peaks for the unknown sample with retention time of reference standards (continuing calibration verification standards). Additionally pattern recognition is used for comparison of unknown samples with reference standards for positive identification. Confirmation of Aroclor presence by secondary GC column analysis may be necessary for highly altered/degraded PCB patterns or for programs including PCB air monitoring, US-EPA CLP protocol and other projects as specified in the site sampling and analysis quality assurance plan.

12.10.1.1 Dual Column/Confirmatory Column Analysis by GC:

Inject samples under same operating conditions and analytical run QA/QC parameters on a secondary GC column of dissimilar phase (e.g ZB-1 and ZB-5). Note: If using dual GC column system, samples are injected sequentially through separate injection ports onto both columns. Samples are analyzed and concentration results are reported.

12.10.1.2 Dual Column/Confirmatory Column Laboratory Default by SW-846:

12.10.1.2. 1 Report **highest** concentration of the 2 column results for each individual Aroclor on the merged EDD, Form 1 or Certificate of Analysis (Note: This is appropriate for Aroclor regulated projects. E.g. Air Monitoring for EPA TO-

10A alternative reporting may be based upon total PCB values for PCB- Total regulated projects).

12.10.1.2.2 If **RPD percent** exceeds 40% report the highest concentration result of the two analyses unless observed chromatographic interference or instrumental analysis QA/QC indicates the lower value may be more accurate. P-flag all excursions > 40% and describe interferences or rationale for reporting lower value in Data Narrative.

12.10.1.2.3 If a concentration is above the PQL on one column and below the PQL on the second column, the qualitative presence is not confirmed and the sample is reported as not detected. **Note: If reporting to the MDL is required do the following:**

For reporting the MDL:

a) If one result is greater than the PQL and other result is < PQL (J-flag) Report the **highest** result as confirmed (*unless interference or QC reasons indicate lower value*)

b) If one result is above MDL (J-Flag) and second is Not Detected report the concentration as **not detected**. (Presence not confirmed).

c) If both results are J-Flag values (< PQL) report the **highest** value of the two.

12.10.1.3 USEPA-CLP/ASP Program Protocols

a) Report **Lowest** Value of the 2 column results for each individual Aroclor on the merged EDD, Form 1 or Certificate of Analysis (Note: This is appropriate for Aroclor regulated projects. E.g. Air Monitoring for EPA TO-10A alternative reporting may be based upon total PCB values for PCB- Total regulated projects).

b) If **Percent Difference** (not RPD%) exceeds 25% then P-flag all excursions > 25%. Note any chromatographic interferences present in Case Narrative.

c) If one result is greater than PQL and other result is < PQL (J-Flag) Report the **lowest** result (J-Flagged) value (*confirmed hit*).

d) If one result is above MDL (J-Flag) and second is Not detected, report the concentration as **not detected** (*presence not confirmed*).

13.0 Calibration and Standardization

13.1 Gas chromatographic operation parameters: See Attachment C

13.2 Initial GC Calibration

13.2.1 GC calibration is performed by the external standard calibration procedure. Prior to running samples the system must be calibrated and system performance must be verified.

13.2.2 Establish the gas chromatographic operating parameters outlined in the Procedure section and prepare the calibration standards at the five concentrations outlined in the Reagent and Standard section. Inject

each calibration standard using the GC Autosampler and the parameters outlined in the Procedure section. Note: The same parameters are used for actual samples.

13.2.3 For each Aroclor, 5 peaks are selected to prepare calibration curves. The peaks selected from the multi-component Aroclor formulations were based on maximizing the separation for each Aroclor (i.e., minimizing peak overlap in retention time). Consideration was also given to selecting peaks that normally did not have problems with co-elution with interfering peaks or possible co-elution with organochlorine pesticides. The determined area of the five peaks selected for calibration is processed by the data workstation as a group, combining the area for calculations of the calibration factors. The following table lists the Aroclors that are included in the initial calibration and the peak numbers used.

Aroclor	Peak Numbers
A1016	6, 7, 8, 9, 10
A1221	1, 2, 3, 4, 5
A1232	5, 7, 8, 9, 10
A1242	6, 7, 8, 9, 10
A1248	11, 12, 13, 14, 15
A1254	16, 17, 18, 19, 20
A1260	20, 21, 22, 23, 24

13.2.4 For the initial calibration curve to be considered valid, the percent relative standard deviation of response factors must be less than 20% over the working range if average calibration factor quantitation is used. Note: the % RSD is a useful check for linearity through the origin and is used as a data quality indicator. In general an inverse weighted linear calibration curve with intercept is used for quantitation and is not replaced with the average calibration factor. For linear calibration curve the Correlation Coefficient R must be greater than 0.99.

13.2.5 Our laboratory uses a computer based chromatography software module (Water Corporation, Empower software) interfaced to the gas chromatograph. The workstation processes the detector signal, performs an analog to digital conversion, and stores the digitized chromatograms on the computer hard disk. Integration of peak areas and production of chromatograms is performed in the Empower software. All data analysis will be carried out in Empower including calculating calibration curves/response factors, report generation, and archival of data.

13.2.6 If a re-calibration is performed, the CCCS must be analyzed again and values calculated using the new relative response factors. If the CCCS fails to meet the percent difference criteria after re-calibration, sample analysis must not proceed until the problem is found and corrected (*i.e.,* GC gas leak, autosampler syringe plugged, broken injector liner).

13.4 Retention Time Windows

13.4.1 The GC system should be checked by the analyst to make sure it is functioning properly before establishing retention time windows. Select a calibration standard and inject three times within a 72-hour time period.

13.4.2 For each peak calculate the standard deviation resulting from the variation in the three retention times for that peak.

13.4.3 The retention time window is defined as plus or minus three times the standard deviation of the three retention time determinations.

If the standard deviation of the selected peak is zero, then a default standard deviation of 0.01 minutes is used. If it is the last eluting peak that the zero for the standard deviation, then substitute the standard deviation of the peak eluting before the last peak.

14.0 Procedure

14.1 Sample Extraction and Preparation

14.1.1 The following SOP's detail sample extraction procedures that are utilized in preparing samples for analysis by this analytical method:

SOP NAME	TITLE	EPA Method
NE005	Soxhlet Extraction/ Extract Prep	8082, 3540C
NE017	Fish and Biota Extraction	8082
NE088	PCB Extraction Of Wipe	8082, 3540C
NE111	Waste Dilution EPA 3580 for PCB 8082	8082, 3580A
NE120	Extraction and cleanup of PCB by SW-846 3550B	8082, 3550B
NE140	PCB Screening by GC	3510C, 3520C, 3545
NE141	SW-846 3510C H2O PCB extraction	8082, 608, 3510C
NE143	EPA 3545 extraction for 8082 PCB	8082, 3545
NE144	EPA 3545 extraction of wipe for 8082 PCB	8082, 3545
NE151	PUF Extraction for 8082 analysis	8082, 3540C/ TO-10A
NE158	% Lipid Determination Fish & Biota	3540, 3500, 2500A
NE194	High Level PCB extraction for 8082 by SepFunnel	8082, 3510C

14.2 Gas Chromatographic Procedures

14.2.1 Prescreening of sample extracts: See standard operating procedure NE140 for details on the PCB screening procedures used prior to final analysis by this method. Prescreening is a fast and effective way to determine if re-extracts are required and dilutions for over ranged samples. The GC will be standardized by using Aroclor 1221, Aroclor 1242, and Aroclor 1260. These three Aroclor formulations incorporate most environmental PCBs found in sample extracts and provide a good estimate of PCB amount for final dilution for this determinative method. A three level calibration curve is utilized (0.50ug/ml, 2.5ug/ml, and 5.0ug/ml standards). The concentration of each Aroclor (grouped as Aroclor 1221, Aroclor 1242, and Aroclor 1260 only) in a sample will be calculated based on the extract volume (not the sample weight or volume) to supply solution concentration values that show if the extract needs to be diluted for final capillary GC analysis. If a dilution is necessary, sample extracts are diluted to a solution concentration near 0.500ug/g, so ensuring each sample quantifies in the middle of the calibration curve.

14.2.2 Approximately 1.0ml of the final dilution extract is then transferred into a labeled autosampler vial.

14.2.3 The sequence of the analytical queue is set up in the LIMS as a unique batch file. This file contains the exact order in which standards, instrument blanks, and samples will be analyzed. Once the sample set is uploaded into the Empower acquisition/run screen and saved, the sample set is printed and the samples are loaded into the GC autosampler tray in the order specified by the sample set queue.

14.2.4 The following labeling will be used on the autosampler vial and for the sample set file created for the analytical queue.

14.2.4.1 The initial calibration standard will be labeled as 040516A, 040516B, etc. Substitute the actual date of analysis and the Aroclor used in the file name.

14.2.4.2 The instrument blanks will be labeled 070405B01, B02, B03, etc. Substitute the actual date of analysis in the file name.

14.2.4.3 The continuing calibration check standards will be labeled CS160405A CS160405B, etc. Substitute the actual date of analysis and the Aroclor used in the file name.

14.2.4.4 Samples are labeled with the laboratory identification number on the autosampler vial. In the sample set file the laboratory identification number, along with the client identification, sample weight, set volume and dilution are entered.

14.2.5 At this point the chromatography software can be initiated to start data collection. The gas chromatograph is placed into run mode and sample analysis is performed until the analytical queue is complete.

14.2.6 Peak Identification

14.2.6.1 Target peaks are identified in unknown samples based upon Retention Time (RT). The retention time of an unknown peak must fall within the retention time windows established.

14.2.6.2 Besides using retention time windows to assign peak IDs, the analyst should also rely on their own experience in recognition of multi-response PCB chromatograms. Caution should be exercised when identifying peaks which elute near interferences present in samples and blanks. Comparison of sample chromatograms with method blank and field blank chromatograms is useful in determining chromatographic interferences.

14.2.6.3 This method should be applied with caution when used in determining PCB of interest in unknown sample for which no prior historical information exists. In this case confirmatory column analysis or confirmation by GC/MS analysis may be advised.

14.3 Data Reduction/Reporting

14.3.1 Final peak assignments and quantitation calculations are performed within the software along with the current instrument calibration. The final concentration results are provided in the reporting section of the software. Final concentration results are reviewed by QA department or other approved manager before release to the client.

14.3.2 Data Qualifiers:

Sample Concentration Reports (Certificates of Analysis, Data Package Form 1's and Electronic Data Deliverables (EDDs) are generated using the appropriate data qualifiers as follows:

U – Denotes analyte not detected at concentration greater than or equal to the Practical Quantitation Limit (PQL). Note: PQLs are adjusted for sample weight/volume and dilution factors.

J - Denotes an estimated concentration. The concentration result is greater than or equal to the Method Detection Limit (MDL) but less than the Practical Quantitation Limit (PQL).

P - Indicates relative percent difference between primary and secondary GC column analysis exceeds 40%.

C- Denotes analyte confirmed by secondary GC column analysis.

B - Denotes analyte observed in associated method blank. Analyte concentration should be considered as estimated.

E - Denotes analyte concentration exceeded calibration range of instrument. Sample could not be re-analyzed at secondary dilution due to insufficient sample amount, quick turn-around request, sample matrix interference or hold time excursion. Concentration result should be considered as estimated.

Z - Laboratory Reserved Qualifier (explained in associated Case Narrative)

15.0 Calculations

15.1 Calibration curve calculation:

15.1.1 PCB Solution concentration calculation from initial Calibration by Linear Regression Yi= aXi + b

Xi = Calibrated Solution Concentration (ng/mL)
Yi = total area response of 5 PCB quant. peaks (uV-Sec.)
a = slope
b = intercept

Unknown Solution Conc. X = (Y –b) a

Y = Total area response of PCB Chromatogram (uV-Sec.)
a = slope of ICAL by linear regression
b = intercept of ICAL by linear regression

15.2 Capillary GC: Sample calculations

15.2.1 The concentration of each identified PCB Aroclor in a sample will be calculated based on the sample weight or volume.

15.2.2 The PCB solution concentration of the extract is calculated as follows:

Solution Conc. = (Y –b)/a

Where:

Y = Total area response of PCB Chromatogram (uV-Sec.)
a = slope of ICAL by linear regression
b = intercept of ICAL by linear regression

15.3 Final concentration of samples:

15.3.1 Calculations of final PCB concentrations will vary upon matrix, calculations are as follows:

(1.) Soil/Sediment/Solids:

Final Conc. = (Sol. Conc.) * (V)*DF/ (M)* (%Total Solids) (1/1000) ug/g

Where: Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

(2.) Water:

Final Conc.= (Sol. Conc.) * V*DF/[(Vt)](1/1000) ug/L

Where: Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
Vt= Total Volume Extraction (L)

(3.) Biota Tissue

Final Conc. = (Sol. Conc.) * (V)*DF/(M)(1/1000) ug/g

Where: Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

(4.) PUF Cassette

Final Conc. = (Sol. Conc.) * (V)*DF/ (Va) ng/cubic meter

Where: Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
Va = volume of air sampled (cubic meters)

(5.) Waste Oil

Final Conc. = (Sol. Conc.) * (V)*DF/ (M)*(%Total Solids) (1/1000) ug/g

Where: Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

15.3 The calculated concentration for each PCB aroclor will be compared to its respective sample-specific reporting limit (RL) and method detection limit (MDL). The results with concentrations at or above the MDL but below RL will be reported as detects and flagged as estimated J. The results for peaks with concentrations at or above the RL would be reported as unqualified numeric values.

16.0 Method Performance
Method Performance is Assessed by Initial/Continuing Demonstration of Proficiency Studies and MDL Determinations

16.1 Initial Demonstration of Capability (IDOC) Procedure:

16.1.1 Prepare 4 replicates of a fortified laboratory blank sample (using laboratory reagent water or sodium sulfate) by spiking each sample with 1.0 mL of 0.500 ug/mL Aroclor solution for water samples and 0.100 mL of 100 ug/mL Aroclor 1242 solution for solid samples. The Aroclor type used for spiking should be rotated on a yearly basis. Prepare one method blank sample with the batch. Extract and analyze each aliquot according to procedures beginning in Section 14.0 below.

16.1.2 For each replicate the recovery value of the sample must fall in the range of 70±30 % (or established lab limits) and the percent RSD must be < 20 % for the method performance to be considered acceptable. See Section 23 Attachment G for example IDOP study.

16.1.3 This procedure must be repeated using four fresh samples until satisfactory performance has been demonstrated. The initial demonstration of capability is used primarily to preclude the laboratory from analyzing unknown samples via a new, unfamiliar method prior to obtaining some experience with it. It is expected that as laboratory personnel gain experience with this method the quality of data will improve beyond those required here.

16.2 Continuing Demonstration of Performance Procedure:

16.2.1 Annual continuing demonstration of performance may be satisfied by a repeat Initial Demonstration of Performance, the acceptable analysis of an unknown samples (for example PT test sample), or the acceptable analysis of 4 consecutive Laboratory Control Spike samples. Records of continuing demonstration of performance are maintained by the laboratory Quality Assurance Department.

16.2.2 With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples an organic-free reagent water blank is processed.

16.2.3 The method blank should exhibit PCB levels less than the practical quantification limit or reporting limt (PQL or RL). If the method blank exhibits PCB contamination above the reportable quantitation limit, the samples associated with the contaminated blank should be re-extracted and analysis repeated when appropriate. If there is no original sample available for re-extraction or if the associated sample concentrations greatly exceed the blank concentration, then all positive concentration results for the associated samples should be flagged with a "B" indicating blank contamination and a case narrative describing the situation prepared.

16.2.4 A matrix spike is to be analyzed at a rate of 1 matrix spike per every 20 samples. A duplicate sample may be prepared in lieu of a matrix spike when detectable PCB concentrations are known to be present.

16.3 Method Detection Limit

16.3.1 A method detection limit will be determined for this method whenever major modification to the extraction or analysis procedures are made or at a minimum frequency of every 2 years. A minimum of seven laboratory organic free water samples or sodium sulfate will be prepared and spiked with chlorinated PCB methyl esters mixture, at a low level and taken through all extraction and analytical procedures.

$MDL = S * t_{(n-1, 1-alpha=0.99)}$

Where:

S = Standard deviation of the replicate analyses

n = Number of replicates

$t_{(n-1, 1-alpha=0.99)}$ = Student's t value for the 99% confidence level with n-1

For example: t for 8 replicates = $t_{(7,0.99)} = 2.998$

16.3.2 The determined MDL must be less than the concentration spiked but greater than one tenth (1/10) the spiked concentration. If not, repeat the MDL determination at an appropriate spike concentration for affected analytes.

17.0 Pollution Prevention

17.1 Pollution prevention is practiced in the laboratory by minimizing usage of solvents and chemicals, so that disposal of waste generated is held to the smallest amount possible. This is directly linked to the types of extraction procedures in place at the laboratory to reduce the volumes of solvents used for semi-volatile extraction procedures. Pace Analytical Services employs extraction procedures such as continuous liquid/liquid and solid phase extraction methods to reduce solvent requirements for water extraction protocols and ASE and Soxhlet extractions for solid matrices.

17.2 Pollution prevention also relies on minimizing to the best extent the chemicals and solvents required to perform extraction and analysis procedures. The laboratory personnel strive to purchase chemicals and standards that will be consumed based on anticipated workload. For additional information about laboratory pollution prevention, please refer to laboratory SOP NE168.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 The GC analyst is responsible for generating the data and also is the initial individual to review the data. This would include inspection of the chromatographic data, processing the raw data, producing all required data forms, inspection of calibration curves for compliance, surrogate recovery, laboratory control spike recovery, matrix spike/matrix spike duplicate recovery, and continuing calibration compliance.

18.2 Once the initial review of the data is performed by the analyst, decisions are made at that time to accept the data if all criteria are met or to reject sample data if any of the quality control parameters or limits are out of control. Depending on the situation, samples requiring re-extraction will be notified to the appropriate extraction personnel, sample extracts

requiring re-injection will be queued for analysis, new calibrations may have to be performed, or samples re-analyzed due to failing continuing check standards.

18.3 The analyst may also consult with the quality control officer as to the best form of action to take or if the situation warrants corrective action beyond routine practices. If no recourse is available and the data is to be reported out of criteria, a Case Narrative Report is generated and the deviation is documented and reported to the client. The Case Narrative Report is filed with the data and is also useful for production of case narratives that are issued with the final data reports. If a problem exists that requires follow-up to rectify, a Corrective Action Report (CAR) is issued to document the problem found, steps taken to resolve the problem, and what samples were affected. This CAR form is filed by the quality control officer and reviewed by management to verify that appropriate actions have been taken to correct the problem.

18.4 Please see Table 19.1 below for specific Quality Assurance Acceptance Criteria.

19.0 Corrective Action for Out-Of-Control Data

19.1 The table below outlines the data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

Quality Control Acceptance Criteria and Corrective Action Plan

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	The five point calibration is analyzed initially and when Continuing Calibration Check standard fails criteria.	- %RSD≤20% for the relative response factors for the calibration standards if using average response factor calibration. Correlation Coefficient R must be >0.99 for Linear Regression.	- Re-analyze the initial calibration standard and/or evaluate/correct instrument malfunction to obtain initial calibration and continuing calibration check standards that meet criteria.
Continuing Calibration Check Standard (CCCS)	- Initially analyze a CCCS immediately following an initial calibration. - After the initial CCCS of the sequence, a CCCS must be analyzed after 9 samples. - Analytical sequence must end with analysis of a CCCS.	- Calibration factor for the continuing calibration check must ±15% of the true value. - Retention time of all quantitated peaks must be within RT window (reset with each initial CCCS of a sequence). - All samples must be bracketed by a CCCS that meet all criteria stated above.	- If the reason for the failure of the CCCS appears to be a poor injection (or a degraded standard solution), the CCCS will be re-injected (or re-prepared and re-injected) immediately following the failed CCCS. This can only occur if the instrument is being attended by an analyst. If upon re-injection, the CCCS meets all the acceptance criteria and there is no apparent impact on the sample data the analytical sequence will continue and samples will not be reanalyzed. The associated sample data will be reported. - If CCCS failure was not due to a poor injection (or degraded standard solution) or the instrument was unattended at the time of the CCCS failure, correct system, if necessary, and recalibrate. Initial calibration and CCS criteria must be met before sample analysis may begin. Samples that are not bracketed by complaint CCCSs must be reanalyzed. -If acceptable CCCSs are observed later in the sequence, samples bracketed by acceptable CCCSs will be reported. Samples between the failed CCS and prior/ subsequent complaint CCCS will be re-analyzed.

-Retention Time (RT)	- Use the retention time for peak in the CCSs to determine midpoint of the relative retention time window for the analysis sequence. -Each sample analysis: Rely on RT windows to identify PCB Aroclor to report. Also use pattern recognition and professional judgment for peaks that shift from RT windows, because compound composition may shift RT for GC peaks.	- Each quantitated peak and surrogate peak should be with established windows.	-Inspect chromatographic system for malfunction, correct problem. Perform re-analysis if necessary.
Method Blank	-One per extraction batch of ≤20 samples of the same matrix per day. -Must be analyzed on each instrument used to analyze associated samples. -Must undergo all sample preparative procedures.	- Concentration does not exceed the RL for any PCB Aroclor. - Must meet surrogate criteria of 60-140% recovery.	- Re-analyze method blank to determine if instrument contamination was the cause. If method blank re-analysis passes, then report samples. -If method blank is found to contain PCB contamination above the RL for any PCB Aroclor compound , then re-extract and re-analyze all associated samples. If no sample exists for re-extraction, report data B flagged to indicate method blank contamination.
Laboratory Control Spike (LCS)	- One per extraction batch of ≤20 samples per matrix per day.	-Percent recovery must be within method limits. - Must meet Aroclor spike criteria of 70-130% recovery -Must meet surrogate criteria of 60-140% recovery.	-Re-analyze LCS to determine if instrument was the cause. If LCS passes, then report samples. -If LCS recovery is still out of limits, the re-extract and re-analyze all associated samples. If no sample exists for re-extraction, report data flagged to indicate LCS failed recovery.

Matrix Spike/Matrix Spike Duplicate (MS/MSD)	-Normal method procedure is to extract and analyze a matrix spike sample. One MS per extraction batch of ≤20 samples per matrix per day. -If requested, an MSD can be extracted and analyzed. The MSD would follow the above criteria as for the MS.	- Percent recovery for MS must be within method limits - If MS/MSD is analyzed, relative percent difference (RPD) should be within 30%. - Must meet Aroclor spike criteria of 70-130% recovery -Must meet surrogate criteria of 60-140% (unless original unspiked sample is also outside of criteria)	-Re-analyze MS and/or MSD to determine if instrument was the cause. If MS and/or MSD pass, then report samples. -Check for errors such as calculations and spike preparation. -Check original unspiked sample results and surrogate recovery for indications of matrix effects. -If no errors are found, and the associated LCS is within limits, then sample matrix effects are likely the cause. Note exceedance in case narrative.
Surrogates	-Surrogates are added to all samples and QC samples. Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) solution.	- Percent recovery for the surrogate should be 60-140%.	-Re-analyze the affected sample or QC sample to determine if instrument was the cause. If surrogate passes, then report samples. -Check for errors in surrogate calculation and surrogate solutions. -If no problem is found, then re-extract and re-analyze the sample. -If re-extraction is within limits and sample extract holding time, then report only the re-analysis. -If the re-extraction is within limits, but out of extraction holding time, then report both sets of data. -If the re-extraction produces surrogate recovery still out of limits, then report both sets of data. -If no sample exists for re-extraction, report data flagged to indicate surrogate failed recovery or have a client re-sample.

20.0 Contingencies for Handling Out-Of-Control or Unacceptable Data

20.1 Data that is detected to be out-of-control for any reason, when compared to method acceptance criteria, will addressed in the following manner:

20.1.1 If the problem exists with the gas chromatographic instrumentation, appropriate action will be taken to repair and perform maintenance to bring the instrument back to operation condition. Once the instrumentation is determined to be correctly operating analysis can begin again.

20.1.2 If the problem exists with calibration standard solutions, the analyst will prepare new standards and discard the standard solutions that are suspect. Instrument calibration can be performed and analysis can begin once system is control.

20.1.3 If the problem exists with sample extraction and extract preparation, the extraction step that is producing the out of-control situation will be diagnosed and rectified. Once the troubleshooting procedures correct the problem extraction can once again occur and analysis can continue.

20.1.4 In situations where data is reported under out-of-control conditions, the data will be annotated with data qualifiers and/or appropriate descriptive comments defining the nature of the excursion in the sample case narrative. If warranted, a corrective action report (CAR) will be issued to define the problem, steps to correct the problem, and final resolution.

21.0 Waste Management

21.1 All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste generated during the execution of this method.

21.2 Please refer to standard operating procedures NE089 and NE054 regarding how hazardous waste is handled and disposed of by the laboratory.

22.0 References:

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

22.2 U.S. EPA 40 CFP Part 136, "Guidelines Establishing Test Procedures of the Analysis of Pollutants", July, 1988.

22.3 "Standard Methods for the Examination of Water and Waste Water", 19th Edition 1995, American Public Health Association, American Water Works Association, Water Pollution Control Federation.

22.4 New York State Department of Health, "Environmental Laboratory Approval Program Certification Manual", Wadsworth Center for laboratories and Research, 1996.

22.5 Guide to Environmental Analytical Methods", third edition, Genium Publishing Corporation, 1997.

23.0 Tables, Diagrams, Flowcharts and Validation Data

23.1 Attachment A: PCB Stock Standard/Calibration Standard Preparation

23.2 Attachment B: Continuing Calibration Check Standard Preparation

23.3 Attachment C: GC Operating Parameters

23.4 Attachment D: Chromatograms

23.5 Attachment E: Example Retention Time Window Study

Attachment A-PCB Stock Standards Prep Table:

Table 1

PCB Stock Standard Preparation Table

PCB Formulation	Supplier	Catalog #	Standard weight(mg)	Conc. (PPM)
A1016	Monsanto Neat Archive	NA	100.0	1000.0
A1221	Monsanto Neat Archive	NA	100.0	1000.0
A1232	Monsanto Neat Archive	NA	100.0	1000.0
A1242	Monsanto Neat Archive	NA	100.0	1000.0
A1248	AccuStandard	C-248N-50mg	100.0	1000.0
A1254	Monsanto Neat Archive	NA	100.0	1000.0
A1260	Monsanto Neat Archive	NA	100.0	1000.0
TCMX/DCBP (Surrogate)	Ultra Scientific	CUS-4911*	0.5/5.0	500/5000

Unless otherwise noted hexane is the solution used to make all dilutions. *Custom Order

Table 2
PCB Calibration Standard Preparation Table (High Level Calibration Curve)

Initial Volume (mL)	Initial Conc. (ug/mL)	Final Volume (mL)	Final Concentration (PPM)					
			A1016	A1221	A1232	A1242	A1248	A1260
5.0	(10.0)	50.0	1.000	1.000	1.000	1.000	1.000	1.000
2.5	(10.0)	50.0	0.500	0.500	0.500	0.500	0.500	0.500
1.25	(10.0)	50.0	0.250	0.250	0.250	0.250	0.250	0.250
1.00	(10.0)	50.0	0.200	0.200	0.200	0.200	0.200	0.200
0.500	(10.0)	50.0	0.100	0.100	0.100	0.100	0.100	0.100
5.0	(0.200)	50.0	0.020	0.020	0.020	0.020	0.020	0.020

Actual Concentration, see Table 1 for actual working standard concentrations for each Aroclor.
See Table 3 for A1254 Standard Preparation (high level)

Table 2A
PCB Calibration Standard Preparation Table (Low Level Calibration Curve)

Init. Volume (mL)	Initial Conc. (ug/ml)	Final Volume (mL)	Final Concentration (PPM)					
			A1016	A1221	A1232	A1242	A1248	A1260
0.5	(10.0)	50.0	0.100	0.100	0.100	0.100	0.100	0.100
2.5	(1.0)	50.0	0.050	0.050	0.050	0.050	0.050	0.050
1.0	(1.0)	50.0	0.020	0.020	0.020	0.020	0.020	0.020
1.0	(0.500)	50.0	0.010	0.010	0.010	0.010	0.010	0.010
0.50	(0.500)	50.0	0.005	0.005	0.005	0.005	0.005	0.005

Actual Concentration, see Tables 1 and 2 for actual working standard concentrations for each Aroclor.
See Table 3A for A1254 Standard Preparation (low level)

Table 3
PCB A1254 Calibration Standard Preparation Table (for High Level Curve)

Initial Volume (mL) A1254	Initial Conc. (ug/mL) A1254	Initial Volume (mL) 0.5/5.0 -PPM Surrogate	Final Volume (mL)	Final Concentration (PPM)		
				A1254	TCMX	DCBP
5.0	10.0	0	50	1.000	0	0
2.5	10.0	0	50	0.500	0	0
10.0	10.00	4.0	100	1.000	0.020	0.200
25.0*	1.000		50	0.500	0.010	0.100
1.25	10.0	0.800	50	0.250	0.008	0.080
0.500	10.0	0.500	50	0.100	0.005	0.050
1.000**	1.000	0.200	50	0.020	0.002	0.020

*This initial volume is of the A1254 1.000 ppm calibration standard WITH surrogates.
**This initial volume is of the A1254 1.000 ppm secondary stock solution WITHOUT surrogates.

Table 3A
PCB A1254, TCMX and DCBP Calibration Standard Preparation Table (for Low Level Curve)

Initial Volume A1254 (mL)	Initial Conc. A1254 (ug/mL)	Initial Volume (mL) 0.5/5.0 -PPM Surrogate	Final Volume (mL)	Final Concentration (PPM)		
				A1254	TCMX	DCBP
5.00	1.000	0.80	50	0.100	0.00800	0.0800
2.50	1.000	0.50	50	0.050	0.00500	0.0500
1.0	1.000	0.40	50	0.020	0.00400	0.0400
1.0	0.500	0.250	50	0.010	0.00250	0.0250
0.50	0.500	0.100	50	0.005	0.00100	0.0100

ATTACHMENT B: PCB Continuing Calibration Standards

Table 1
PCB Continuing Calibration Working Standards
prepared from 1000 PPM Stock Standards

PCB	Stock Source	Initial Volume (mL)	Final Volume (mL)	Concentration (PPM)
A1016	Chem Service Cat # F107AS	1.0	100	10.0
A1221	Chem Service Cat # F108AS	1.0	100	10.0
A1232	Chem Service Cat# F113AS	1.0	100	10.0
A1242	Chem Service Cat# F109AS	1.0	100	10.0
A1248	Chem Service Cat# F110AS	1.0	100	10.0
A1254	Chem Service Cat# F111AS	1.0	100	10.0
A1260	Chem Service Cat# F112AS	1.0	100	10.0

ATTACHMENT B cont'd

Table 2
PCB Continuing Calibration Standards (High Level)
prepared from 10 PPM CCV Working Standards and all contain surrogates

PCB	Surr. Volume* (mL)	Initial Volume (mL)	Final Volume (mL)	Surrogate Concentration TCMX/DCBP (PPM)	Aroclor Concentration (PPM)
A1016	2.0	5.0	100	0.010/0.100	0.500
A1221	2.0	5.0	100	0.010/0.100	0.500
A1232	2.0	5.0	100	0.010/0.100	0.500
A1242	2.0	5.0	100	0.010/0.100	0.500
A1248	2.0	5.0	100	0.010/0.100	0.500
A1254	2.0	5.0	100	0.010/0.100	0.500
A1260	2.0	5.0	100	0.010/0.100	0.500

*Surrogate stock solution 0.500 PPM TCMX and 5.0 PPM DCBP

Table 3
PCB Continuing Calibration Standards (low Level)
prepared from 10.0 PPM CCV Working Standards and all contain surrogates.

PCB	Surr. Volume* (mL)	Initial Volume (mL)	Final Volume (mL)	Surrogate Concentration TCMX/DCBP (PPM)	Aroclor Concentration (PPM)
A1016	1.0	0.500	100	0.005/0.050	0.050
A1221	1.0	0.500	100	0.005/0.050	0.050
A1232	.1.0	0.500	100	0.005/0.050	0.050
A1242	.1.0	0.500	100	0.005/0.050	0.050
A1248	.1.0	0.500	100	0.005/0.050	0.050
A1254	1.0	0.500	100	0.005/0.050	0.050
A1260	1.0	0.500	100	0.005/0.050	0.050

*Surrogate stock solution 0.500 PPM TCMX and 5.0 PPM DCBP

Attachment C: GC Operating Parameters

GC #:	**GC-21 8082 High Level Method(GEHR inclusive, parameters)**
Method:	Method 2
Column:	**ZB-1 Front**
	ZB-5 Middle
Date:	01/18/2012
Analyst:	JKA
File Name:	S:\Lab Data\PCB\GC Parameters\[GC21_Parameters.xls]8082 M2

Sample Delivery: SEE LEAP PARAMETERS

Column Oven:

Step	Temp (°C)	Rate (°C/min)	Hold (min)	Total (min)
Initial	140	-------	2.00	2.00
2	200	10	0.00	8.00
3	245	5	13.5	30.5

Stabilization Time (min): 0.20

Injector: Front CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	30

Injector: Middle CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	30

Flow/PSI(Front EFC, Type 1):

Carrier Gas: Helium

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	30.0*	-------	20	20

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 5.0

Flow/PSI(Front EFC, Type 1):

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	30.0*	-------	20	20

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 5.0

Detector:

Front ECD

ECD Oven Power: ON
Temperature (°C) 300
Electronics: ON
Range: 1

Time	Range	Autozero
Initial	1	YES

Middle ECD

ECD Oven Power: ON
Temperature (°C) 300
Electronics: ON
Range: 1

Time	Range	Autozero
Initial	1	YES

Front ECD Adjustment	Front	Middle
Time Constant:	Fast	Fast
Cell Current:	CAP	CAP
Contact Potential (mV):	-650 *	-380 *
Date of last adjustment	12/15/2011	12/15/2011

Front ECD Adjustments

Make-up Flow (mL/min) 35 *

Middle ECD Adjustments

Make-up Flow (mL/min): 35 *

*values may change with use

Analog Output

Detectors:	Front: ECD	Attenuation	1
	Middle: ECD	Attenuation	1
	Rear: None		

GC-10 8082 High Level Method - Hydrogen

GC #: 10
Method: Hydrogen 8082
GC Method #: 2
Date: 02/09/2012
Analyst: MTH
File Name:
Column: Front ZB-1 MS 20m 0.18 0.18
Middle ZB-5 20m 0.18 0.18

Sample Delivery: SEE LEAP PARAMETERS

Column Oven:

Step	Temp (°C)	Rate (°C/min)	Hold (min)	Total (min)
Initial	150		1.41	1.41
	290	17.5	0.65	10.04

Stabilization Time (min): 0.5

Injector: Front CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	30

Flow/PSI(Front EFC, Type 1):
Carrier Gas Hydrogen

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	30 *		10.00	10.00

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 5.0

Injector: Middle CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	30

Flow/PSI(Front EFC, Type 1):

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	30 *		10.00	10.00

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 5.0

Detector:

Front ECD

ECD Oven Power: ON
Temperature (°C) 300
Electronics: ON
Range: 1

Time	Range	Autozero
Initial	1	YES

Middle ECD

ECD Oven Power: **ON**
Temperature (°C) 300
Electronics: ON
Range:

Time	Range	Autozero
Initial	1	YES

Front ECD Adjustment	Front	Middle
Time Constant:	Fast	Fast
Cell Current:	CAP	CAP
Contact Potential (mV):	250 *	365 *
Date of last adjustment	01/23/2012	01/23/2012
Make-Up Flow (ml/min):	35.0	35.0

*values may change with use

Analog Output

Detectors:
Front: ECD Attenuation 1
Middle: ECD Attenuation 1
Rear: None

Leap GC Pal Parameters					
Sample injection Methods					
Method	**GC Dual**	**GC Duals**		**Method**	**GC Inj s**
Cylcle	GC Dual	GC Dual		**Cylcle**	GC Inj S
Syringe	10 ul	10 ul		**Syringe**	10 ul
1. Sample Vol	1.0 ul	1.0 ul		**1. Sample Vol**	1.0 ul
1. Air Vol	1.0 ul	1.0 ul		**Solvent Plug**	200 nl
1. Inject to	GC Inj 1	GC Inj 1		**Slv Source**	Standard
Inj Time Diff	0s	0s		**Int Standard**	0 nl
2. Sample Offs	1	0		**Std Source**	Standard
2. Sample Vol	1.0 ul	1.0 ul		**Air Gap (s)**	1.0 ul
2. Air Vol	1.0 ul	1.0 ul		**1. Air Vol Ndl**	1.1 ul
2. Inject to	GC Inj 2	GC Inj 2		**Pre Cln Slv 1**	2
Pre Cln Slv 1	2	2		**Pre Cln Slv 2**	2
Pre Cln Slv 2	2	2		**Fill Speed**	5.0 ul/s
Pre Cln Sp 1	0	0		**Pull Up Delay**	1.0 s
Int Cln Slv 1	2	2		**Inject to**	GC Inj 1
Int Cln Slv 2	2	2		**Inject Speed**	5.0 ul/s
Pst Cln Slv 1	2	2		**Pre Inj Del**	0 ms
Pst Cln Slv 2	2	2		**Pst Inj Del**	0 ms
Fill Volume	10 ul	10 ul		**Pst Cln Slv 1**	2
Fill Speed	2.5 ul/s	2.5 ul/s		**Pst Cln Slv 2**	2
Fill Stroke	0	0			
Pull Up Delay	500ms	500ms			
Inject Speed	10 ul/s	10 ul/s			
Pre Inj Del	0 ms	0 ms			
Pst Inj Del	0 ms	0 ms			

ATTACHMENT D ZB5 Chromatograms (Helium Carrier Gas)

FIGURE 1. A1016 @ 0.500PPM PLOT

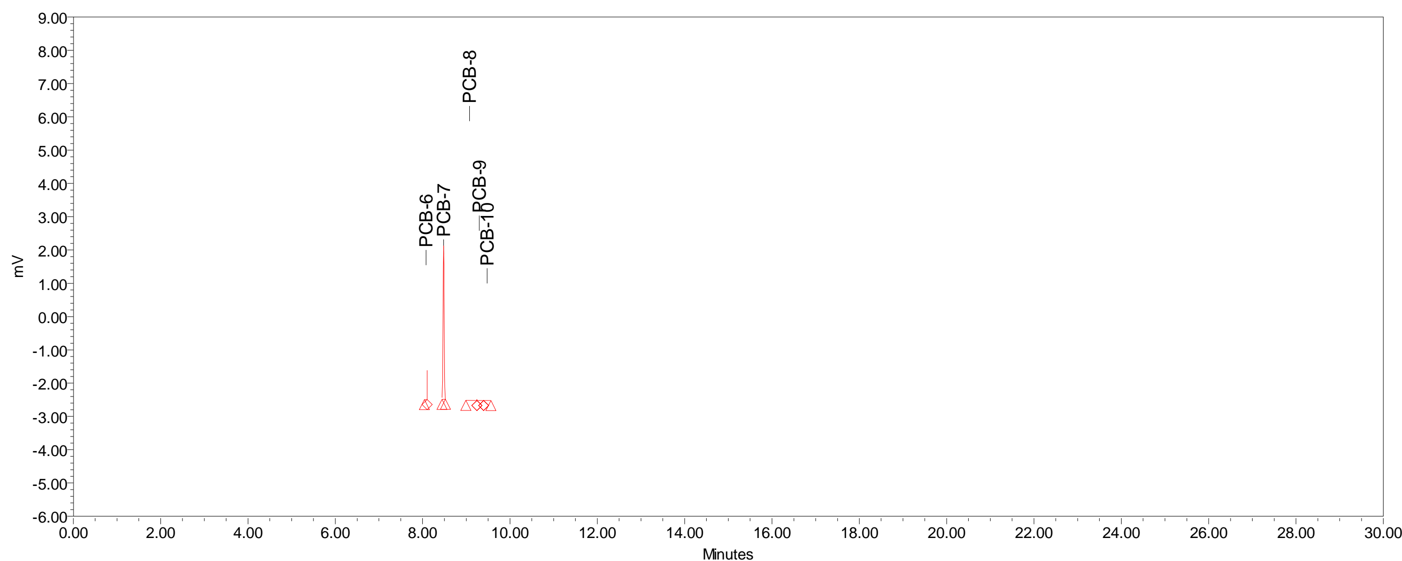


FIGURE 2. A1221 @ 0.500PPM PLOT

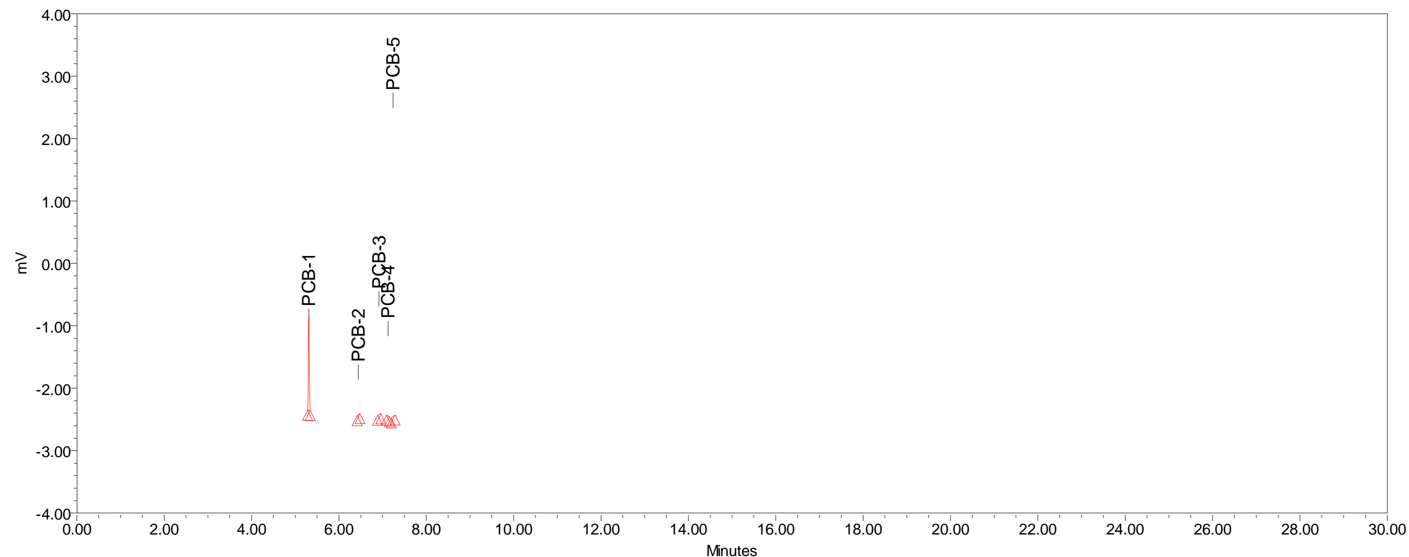


FIGURE 3. A1232 @ 0.500PPM PLOT

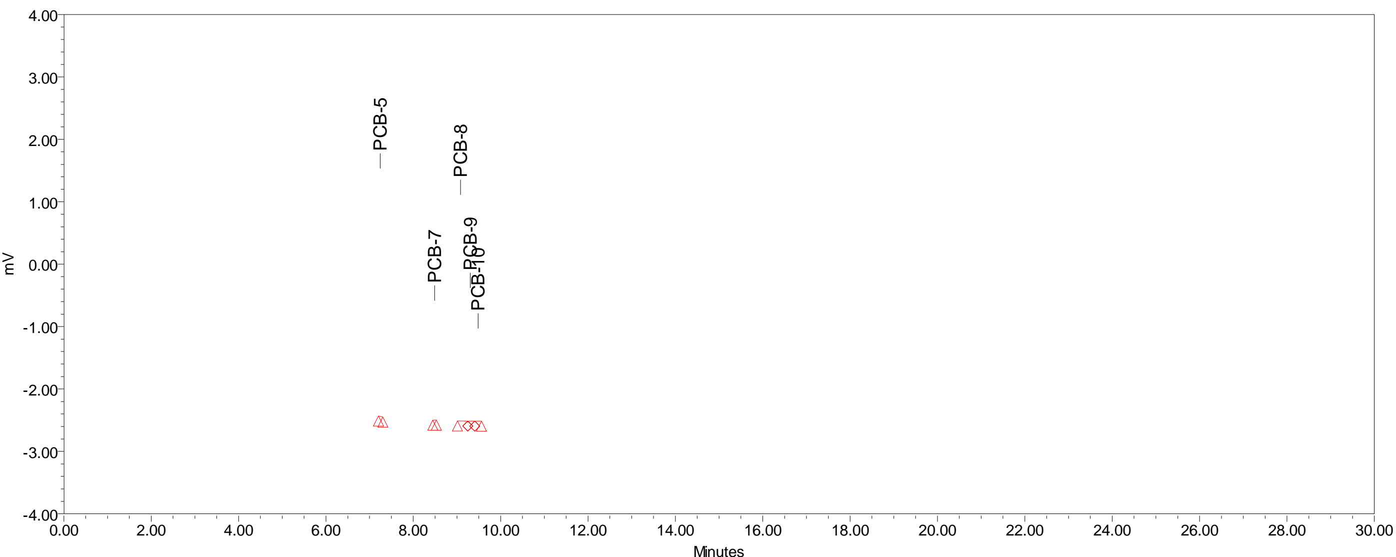


FIGURE 4. A1242 @ 0.500PPM PLOT

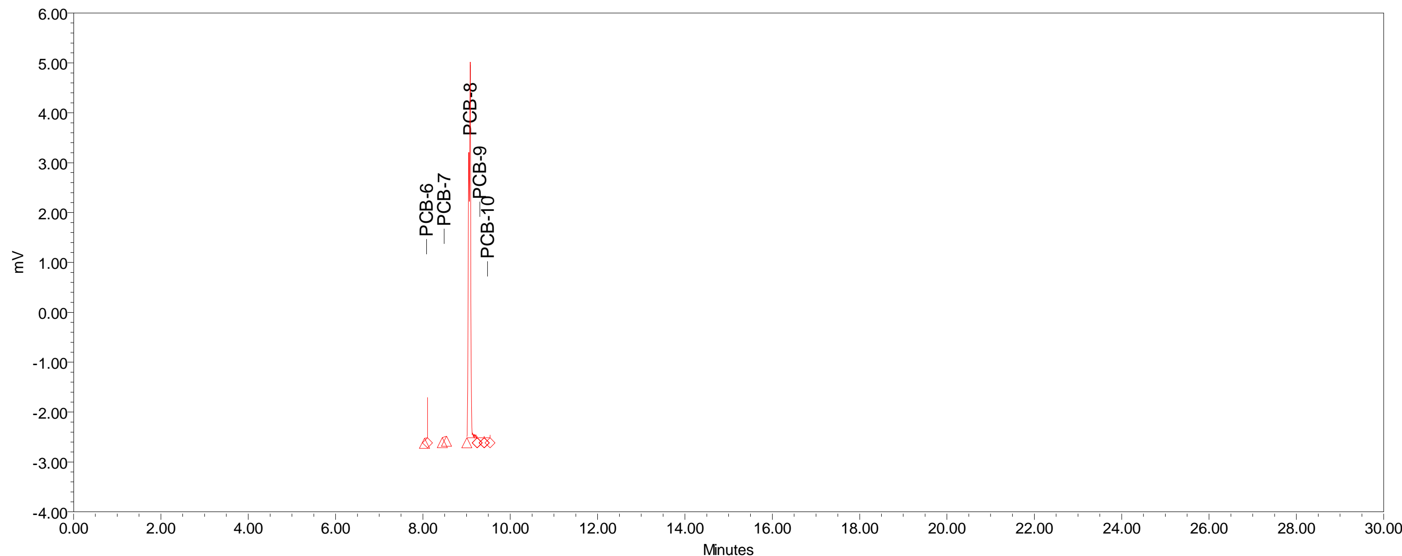


FIGURE 5. A1248 @ 0.500PPM PLOT

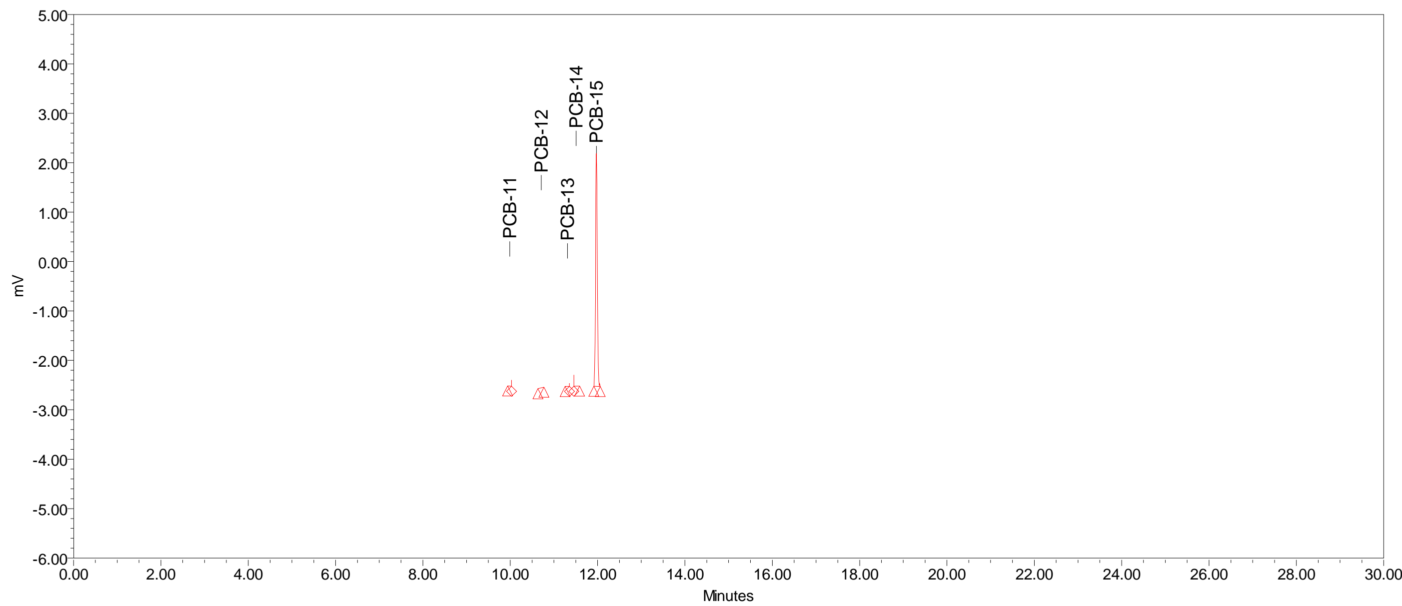


FIGURE 6. A1254 @ 0.500PPM w/ TCMX & DCBP @ 10/100PPB PLOT

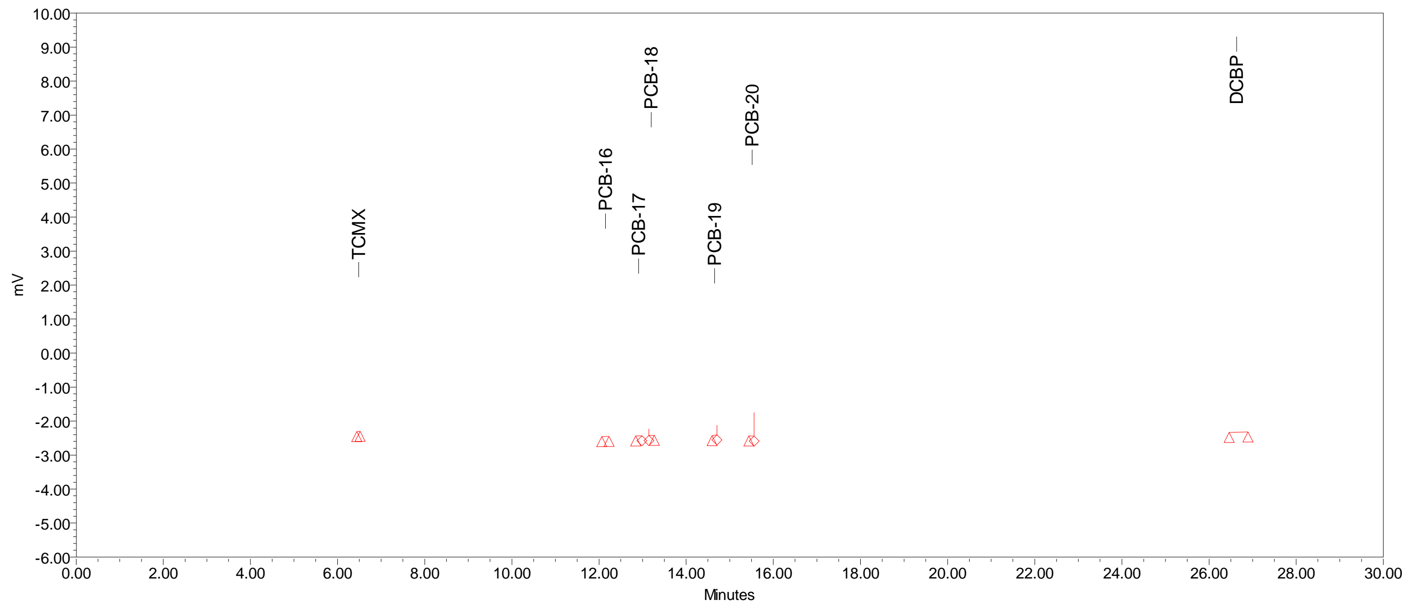
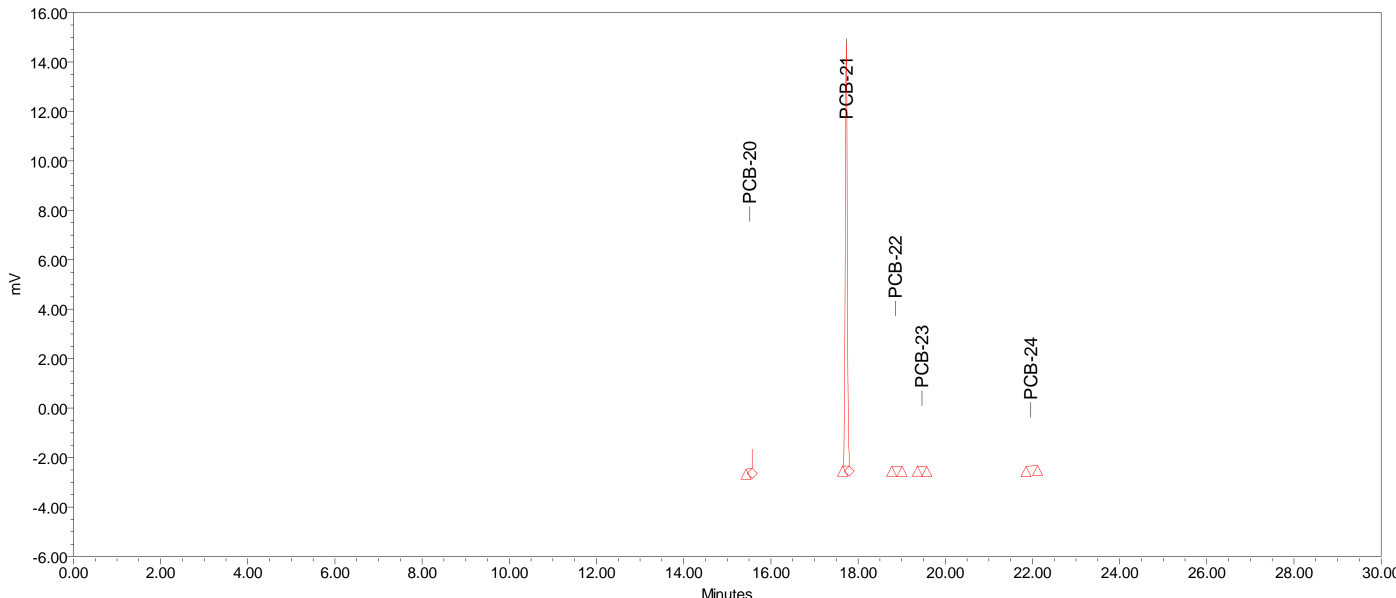


FIGURE 7. A1260 @ 0.500PPM PLOT



ATTACHMENT D cont' ZB1 Chromatograms (Helium Carrier Gas)

FIGURE 1. A1016 @ 0.500PPM PLOT

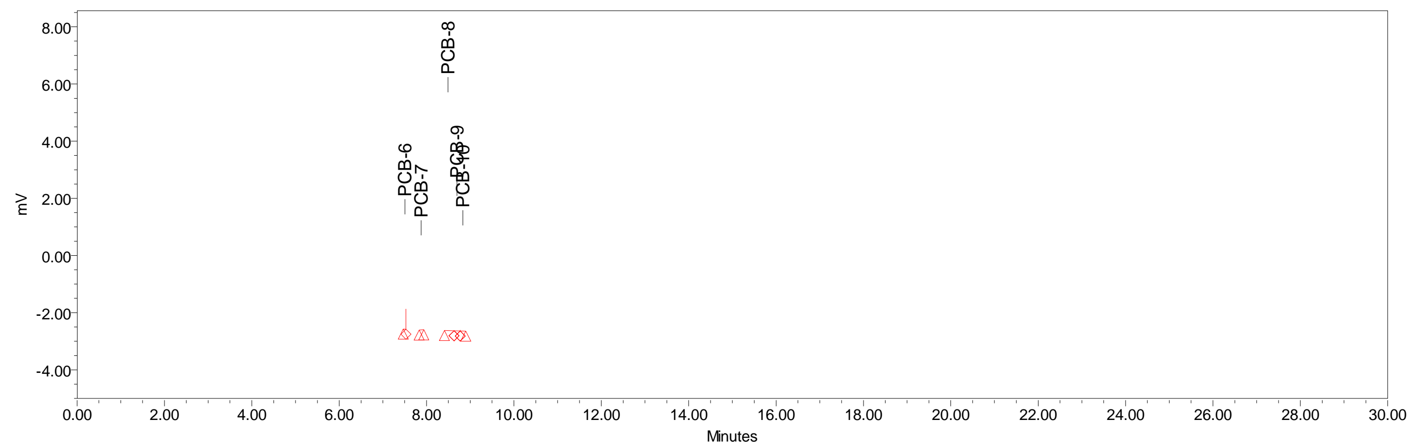


FIGURE 2. A1221 @ 0.500PPM PLOT

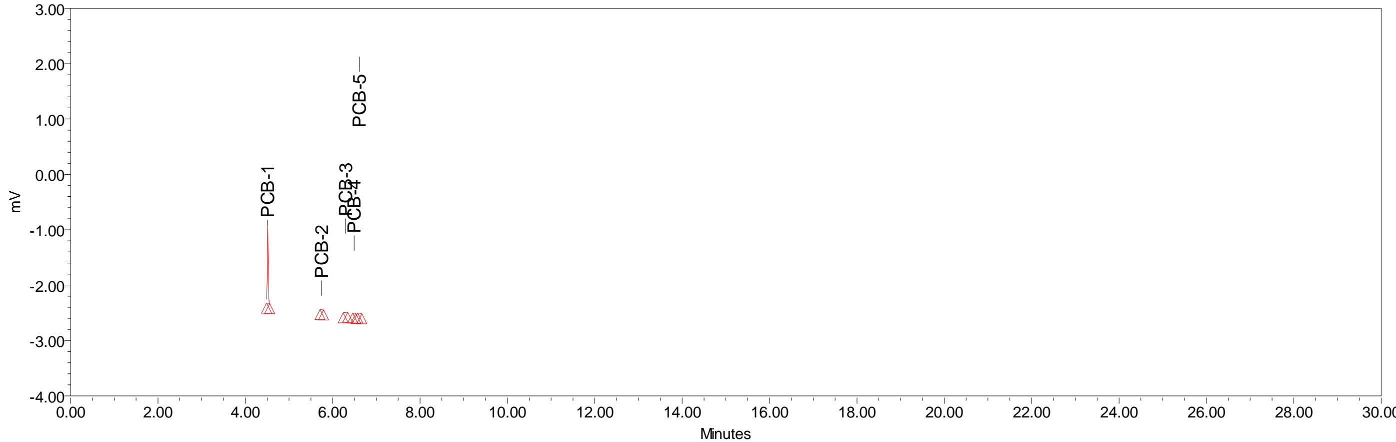


FIGURE 3. A1232 @ 0.500PPM PLOT

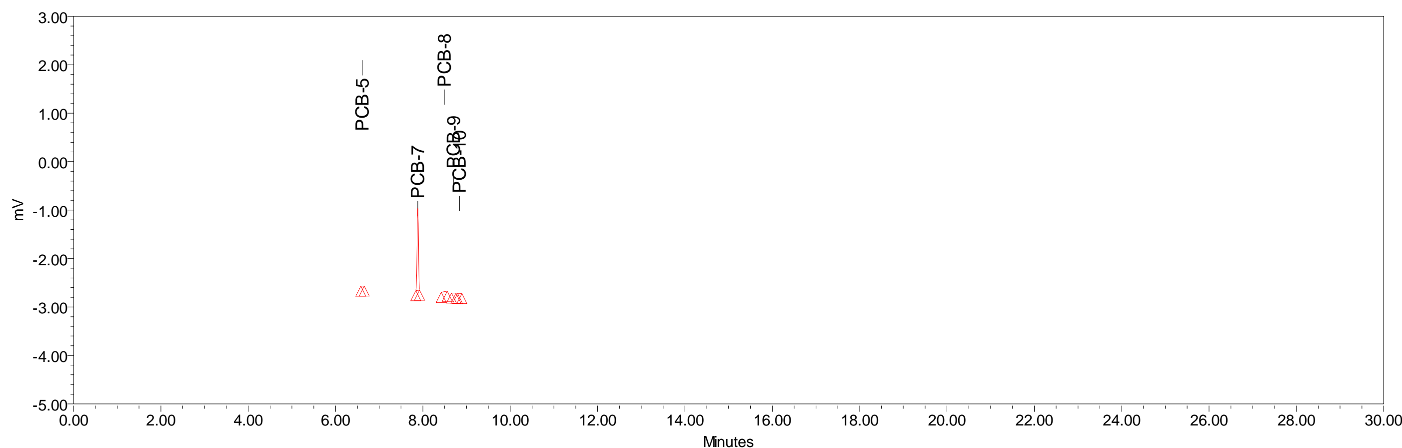


FIGURE 4. A1242 @ 0.500PPM PLOT

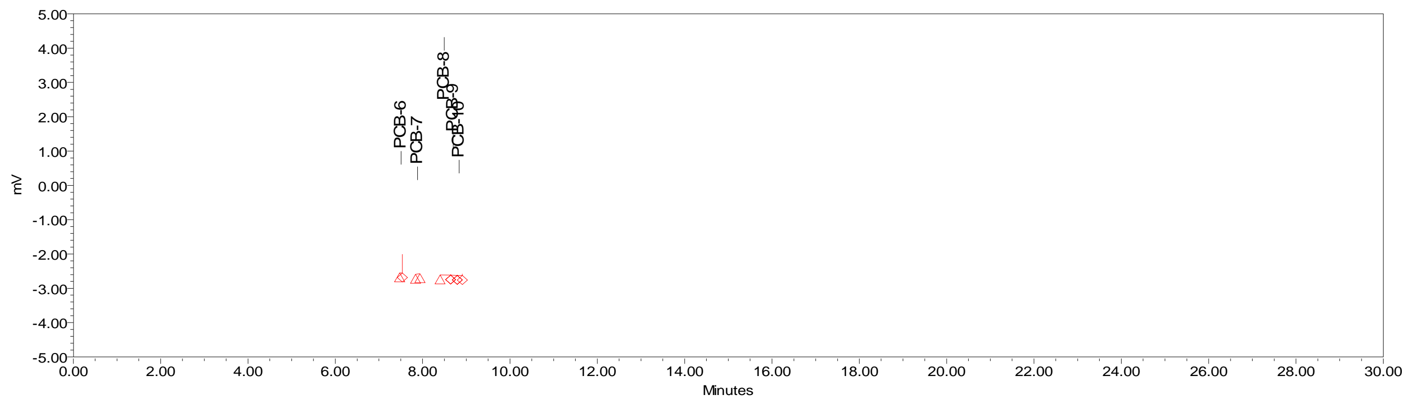


FIGURE 5. A1248 @ 0.500PPM PLOT

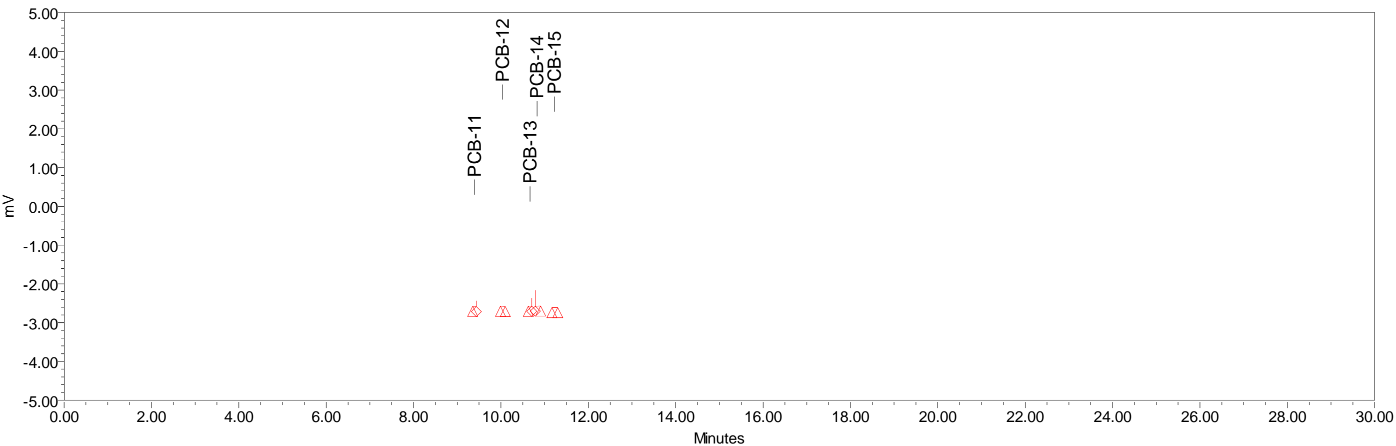


FIGURE 6. A1254 @ 0.500PPM w/ TCMX & DCBP @ 10/100PPB PLOT

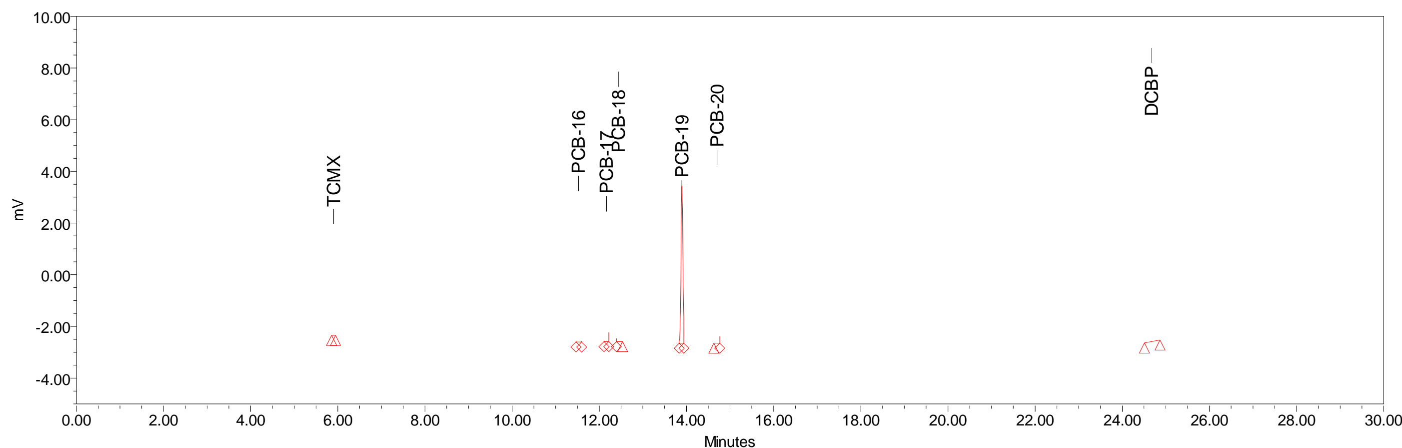
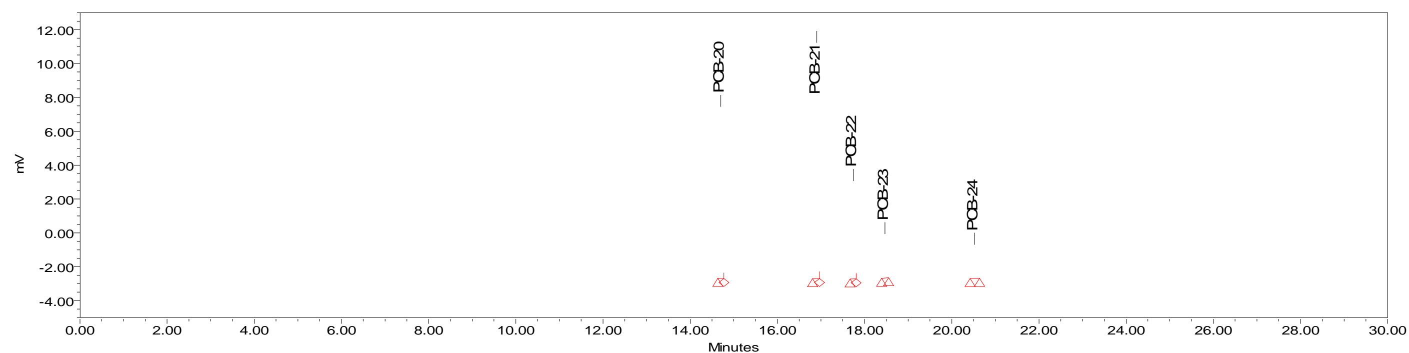


FIGURE 7. A1260 @ 0.500PPM PLOT



ATTACHMENT D cont' ZB-1MS Chromatograms (Hydrogen Carrier Gas)

FIGURE 1. A1016 @ 0.500PPM PLOT

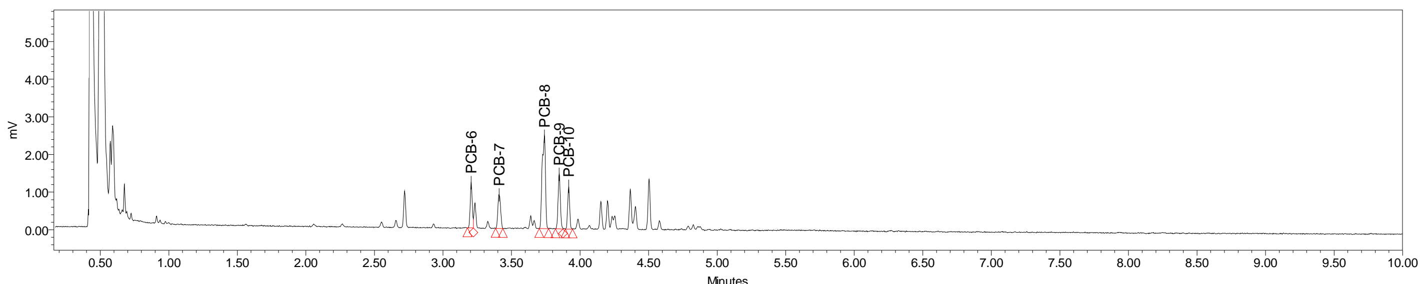


FIGURE 2. A1221 @ 0.500PPM PLOT

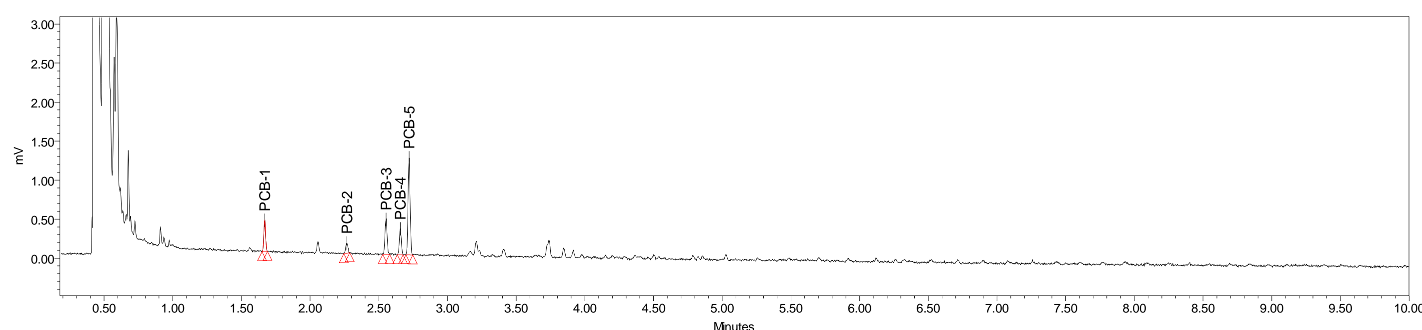


FIGURE 3. A1232 @ 0.500PPM PLOT

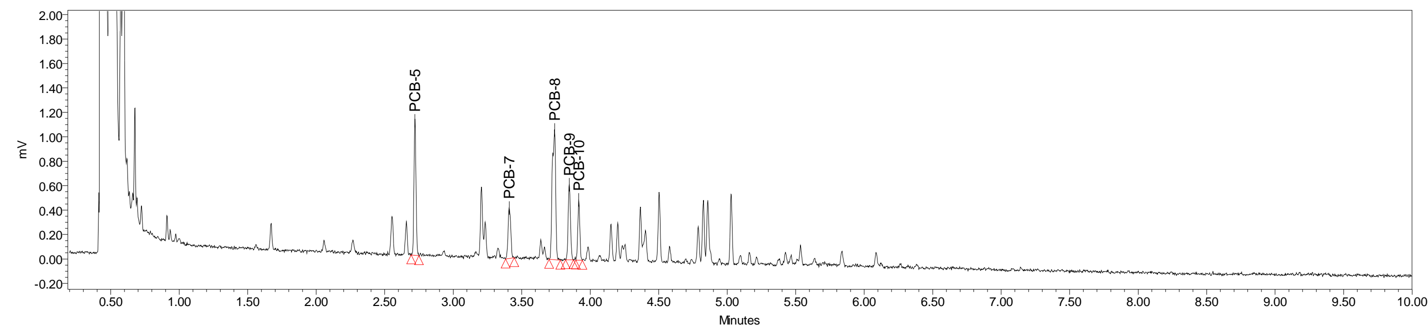


FIGURE 4. A1242 @ 0.500PPM PLOT

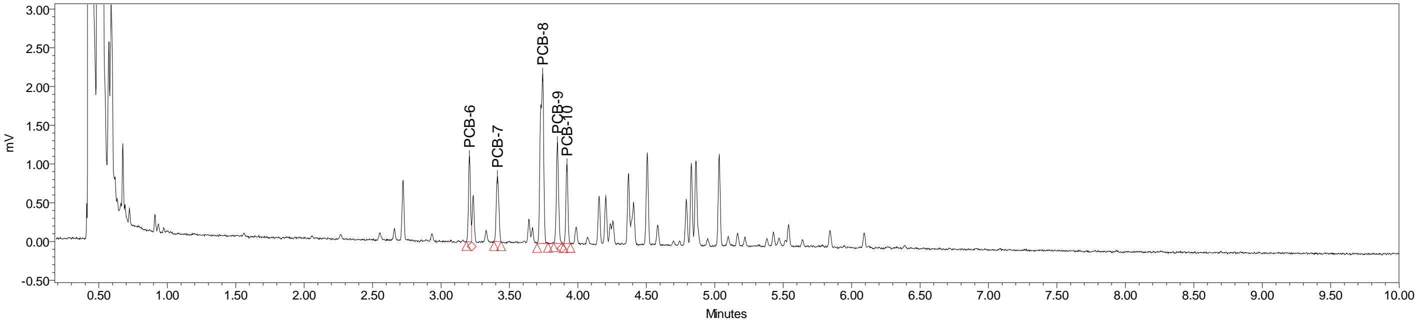


FIGURE 5. A1248 @ 0.500PPM PLOT

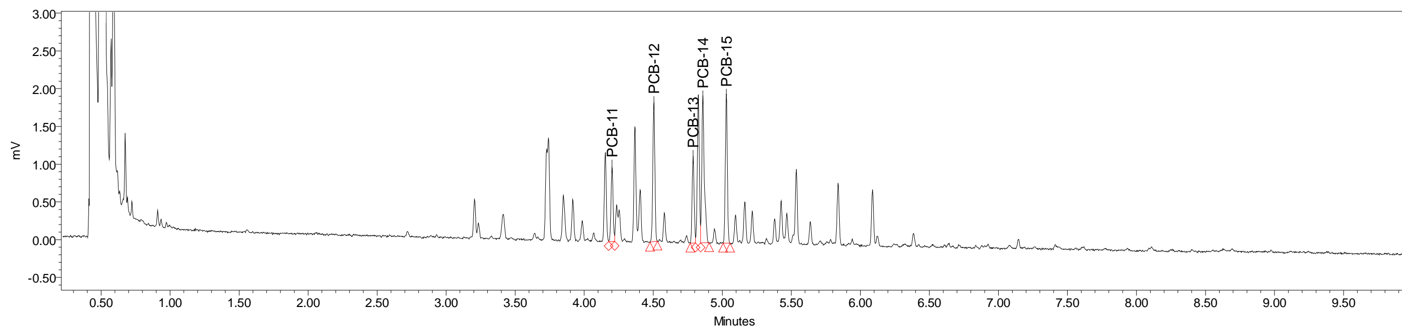


FIGURE 6. A1254 @ 0.500PPM w/ TCMX & DCBP @ 10/100PPB PLOT

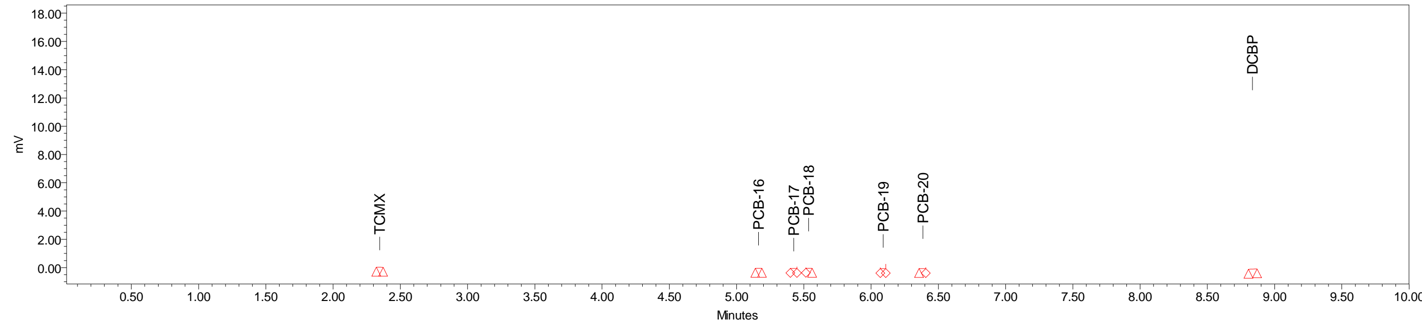
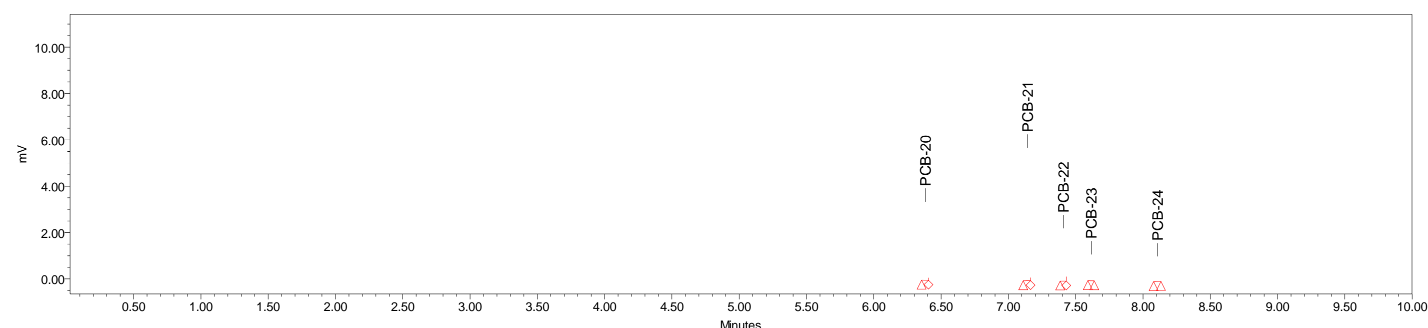


FIGURE 7. A1260 @ 0.500PPM PLOT



Attachment E Retention Time Windows: ZB-1 Column (Helium)

Pace Analytical Services, Inc
Retention Time Window Study

Retention Time Window Study

Compound: Aroclor 1016	Analysis: RT Window Study
Matrix: Solvent	Instrument: GC21F
Extraction: NA	GC Column: Phenomenex, Zebron ZB-1, 30 m, 0.25 mm ID, 0.25 μm
Method: EPA 8082/608	Detector: ECD
	Hour Span: 510

Analyte	Peak	CS160424A 500 PPB GC21F-1767-5 04-24-12 15:40 (R.T. min.)	CS160409A 500 PPB GC21F-1757-3 04-09-12 09:13 (R.T. min.)	CS160403A 500 PPB GC21F-1753-4 04-03-12 09:27 (R.T. min.)	Standard Deviation	RSD (%)	Window +/- min
Aroclor 1016	06	7.52	7.52	7.52	0.000	0.000	0.01
Aroclor 1016	07	7.89	7.90	7.90	0.006	0.073	0.017
Aroclor 1016	08	8.51	8.52	8.52	0.006	0.068	0.017
Aroclor 1016	09	8.72	8.72	8.73	0.006	0.066	0.017
Aroclor 1016	10	8.86	8.86	8.86	0.000	0.000	0.01
TCMX	NA	5.91	5.91	5.91	0.000	0.000	0.01
DCBP	NA	24.97	24.96	24.95	0.010	0.040	0.030

Gas Chromatography Analyst: Jared Acker Date: 04/27/2012

QA/QC Officer: Christina Braidwood Date: 04/27/2012

Lab Director: Dan Pfalzer Date: 04/27/2012

Print Date: 4/27/2012

Attachment E cont' Retention Time Windows: ZB-5 Column (Helium)

Pace Analytical Services, Inc
Retention Time Window Study

Retention Time Window Study

Compound: Aroclor 1016	Analysis: RT Window Study
Matrix: Solvent	Instrument: GC21B
Extraction: NA	GC Column: Phenomenex, Zebron ZB-5, 30 m, 0.25 mm ID, 0.25 µm
Method: EPA 8082/608	Detector: ECD
	Hour Span: 510

Analyte	Peak	CS160424A 500 PPB GC21B-1706-5 04-24-12 15:40 (R.T. min.)	CS160409A 500 PPB GC21B-1696-3 04-09-12 09:13 (R.T. min.)	CS160403A 500 PPB GC21B-1692-4 04-03-12 09:27 (R.T. min.)	Standard Deviation	RSD (%)	Window +/- min
Aroclor 1016	06	8.12	8.13	8.12	0.006	0.071	0.017
Aroclor 1016	07	8.53	8.53	8.53	0.000	0.000	0.01
Aroclor 1016	08	9.14	9.15	9.14	0.006	0.063	0.017
Aroclor 1016	09	9.37	9.37	9.37	0.000	0.000	0.01
Aroclor 1016	10	9.55	9.55	9.55	0.000	0.000	0.01
TCMX	NA	6.50	6.51	6.51	0.006	0.089	0.017
DCBP	NA	27.34	27.34	27.32	0.012	0.042	0.035

Gas Chromatography Analyst: Jared Acker Date: 04/27/2012

QA/QC Officer: Christina Braidwood Date: 04/27/2012

Lab Director: Dan Pfalzer Date: 04/27/2012

Print Date: 4/27/2012

Attachment E cont' Retention Time Windows: ZB-1MS Column (Hydrogen)

Pace Analytical Services, Inc

Retention Time Window Study

Retention Time Window Study

Compound: Aroclor 1016	Analysis: RT Window Study
Matrix: Solvent	Instrument: GC10F
Extraction: NA	GC Column: Phenomenex, Zebron ZB-1MS, 20 m, 0.18 mm ID, 0.18 µm
Method: EPA 8082/608	Detector: ECD
	Hour Span: 247

Analyte	Peak	CS160423A 500 PPB GC10F-344-3 04-23-12 08:29 (R.T. min.)	CS160416A 500 PPB GC10F-339-3 04-16-12 09:31 (R.T. min.)	CS160412A 500 PPB GC10F-337-55 04-13-12 01:16 (R.T. min.)	Standard Deviation	RSD (%)	Window +/- min
Aroclor 1016	06	3.21	3.20	3.20	0.006	0.180	0.017
Aroclor 1016	07	3.41	3.41	3.41	0.000	0.000	0.01
Aroclor 1016	08	3.74	3.74	3.73	0.006	0.155	0.017
Aroclor 1016	09	3.85	3.85	3.84	0.006	0.150	0.017
Aroclor 1016	10	3.92	3.91	3.91	0.006	0.148	0.017
TCMX	NA	2.35	2.35	2.34	0.006	0.246	0.017
DCBP	NA	8.83	8.82	8.82	0.006	0.065	0.017

Gas Chromatography Analyst: Jared Acker Date: 04/27/2012

QA/QC Officer: Christina Braidwood Date: 04/27/2012

Lab Director: Dan Pfalzer Date: 04/27/2012

Print Date: 4/27/2012

Attachment E cont' Retention Time Windows: ZB-5 Column (Hydrogen)

Pace Analytical Services, Inc
Retention Time Window Study

Retention Time Window Study

Compound: Aroclor 1016	Analysis: RT Window Study
Matrix: Solvent	Instrument: GC10B
Extraction: NA	GC Column: Phenomenex, Zebron ZB-5, 20 m, 0.18 mm ID, 0.18 µm
Method: EPA 8082/608	Detector: ECD
	Hour Span: 247

Analyte	Peak	CS160423A 500 PPB GC10B-315-3 04-23-12 08:29 (R.T. min.)	CS160416A 500 PPB GC10B-310-3 04-18-12 09:31 (R.T. min.)	CS160412A 500 PPB GC10B-308-55 04-13-12 01:17 (R.T. min.)	Standard Deviation	RSD (%)	Window +/- min
Aroclor 1016	06	3.75	3.74	3.74	0.006	0.154	0.017
Aroclor 1016	07	3.93	3.93	3.93	0.000	0.000	0.01
Aroclor 1016	08	4.20	4.20	4.20	0.000	0.000	0.01
Aroclor 1016	09	4.30	4.30	4.30	0.000	0.000	0.01
Aroclor 1016	10	4.38	4.37	4.37	0.006	0.132	0.017
TCMX	NA	3.02	3.02	3.02	0.000	0.000	0.01
DCBP	NA	9.06	9.05	9.05	0.006	0.064	0.017

Gas Chromatography Analyst: Jared Acker Date: 04/27/2012

QA/QC Officer: Christina Braidwood Date: 04/27/2012

Lab Director: Dan Pfalzer Date: 04/27/2012

Print Date: 4/27/2012

STANDARD OPERATING PROCEDURE REVIEW

SOP Name	Review Number	Reviewers	Title	QAO Approval	Effective Date
NE148_06	01	Kari Lantiegne Robert E. Wagner	Lab Analyst Lab Director	Christina Braidwood	02/19/09
NE148_07	00	Tom Herold Robert E. Wagner	Lab Analyst Lab Director	Christina Braidwood	03/11/11
NE148_08	00	Inga Hotaling Dan Pfalzer	GC Supervisor Lab Director	Christina Braidwood	05/11/12

Appendix K

Radioisotope Analysis of Cesium-137 and Beryllium -7 in Sediments

Radioisotope Analysis of Cesium-137 and Beryllium-7 in Sediments

I. Background

Select core samples will be geo-chronologically dated to determine the sediment depositional pattern for the area. The dating techniques used for this project have been successfully utilized by the United States Geological Survey (USGS) in a number of similar applications. Two radioisotopes, Cesium-137 (Cs-137) and Beryllium-7 (Be-7), will be used for this project.

Cs-137 is present in sediments due to fallout activity from open-air nuclear testing that occurred during the 1950s and 1960s. Cs-137 usually can be identified in sediments after 1955, when open-air nuclear testing was initiated. Measurable Cs-137 concentrations are expected to represent deposition from 1955 to present. Sediments deposited after 1963 are expected to have lower concentrations of Cs-137 due to restrictions on open-air nuclear testing. Thus, both 1963 and 1955 horizons may be detectable. A sudden transition of Cs-137 values in sediment from measurable to non-detectable (or low) Cs-137 levels as the sediment depth increases can be interpreted as the 1954 Ahorizon@ in the sediment bed.

To verify that sampling procedures did not disturb the sediment profile, Be-7 may be analyzed. Be-7 is naturally occurring in sediments and may be analyzed in surface sediment sections to confirm recent deposition. Due to the short half-life of this isotope, Be-7 detected in samples is representative of deposition occurring within approximately one year. The identification of recent deposition at the surface of a core increases the confidence that the sample core was collected and delivered to the laboratory intact.

The Adating@ analyses are designed to provide an approximate measure of the rate of historic sediment deposition, and will serve to provide a projected sediment deposition rate.

II. Principle, Scope, and Application

This procedure presents the methods for determining gamma emitting radioisotopes by lithium-drifted germanium and high purity germanium detectors with high resolution spectrometry in sediment. The method can be summarized as follows:

- Collect sediment cores following the procedures in Appendix F;
- Section cores into samples as specified in Appendix F;
- Dry and crush samples;
- Weigh into planchet or other standard counting geometry container; and
- Analyze by gamma spectrometry.

Each sample to be assayed is prepared and counted in general geometries such as 300-mL or 150-mL bottles, or 2-inch planchet geometries. Calibration checks will be performed on 2-inch diameter standards.

Gamma spectrometry is the basis of this analysis. The 661.6 kilectron volts (KeV) emission is measured to quantify Cs-137. The 477.6 KeV emission is measured to quantify Be-7.

Samples are counted on large (>55-cc) germanium detectors connected to Nuclear Data 6620 data acquisition and data computation systems. All resultant spectra are stored on magnetic tape.

The analysis of each sample consists of calculating the specific activities of all detected radionuclides or the detection limits.

III. Range of Measurement

The range of measurement is expected to be 0 to 50 picoCuries per gram (pCi/gm). The working range of the detection system is at least 0 to 5,000 pCi.

IV. Limit of Detection

The limit of detection is approximately 0.1 pCi/gm at counting time based on the analysis of a minimum 10-gram sample with an overnight count. With larger samples (in 150-mL or 300-mL bottles) similar detection limits can be obtained with shorter counting intervals.

V. Interferences

A high-resolution germanium/lithium detector is adequate to avoid interferences from other radionuclides.

VI. Procedures

Step 1 - Dry a 10-gram to 300-gram aliquot in an oven for at least one hour at 103 to 105°C. Cool the sample in a desiccator and weigh. Repeat the cycle of drying at 103 to 105°C, cooling, desiccating, and weighing until a constant weight is obtained or until loss of weight is less than 4 percent of the previous weight.

Step 2 - Crush the dried sample using a mortar and pestle.

Step 3 - Transfer the crushed sample into a pre-tared 2-inch diameter stainless steel planchet or into a 150 to 300-mL plastic bottle.

Step 4 - Reweigh the sample and pre-tared sample container and record the weight of the crushed sample.

Step 5 - Analyze the samples using the high resolution germanium detector apparatus. Utilize the attached computer system following the manufacturers recommended procedures for data acquisition and reduction.

VII. Quality Control

Standards Calibration

Mixed gamma ray standards traceable to the National Bureau of Standards are used to calibrate the various standard sample geometries described above. Each standard has been counted on each germanium detector and an efficiency versus energy curve was determined for each geometry for the energy range of approximately 50 KeV to 2 MeV.

Each standard lists the manufacturer of the nuclides, the certificate numbers, nuclides, and activities of the nuclides at a reference time. The counting of the standards are recorded in a log containing the sequential listing of every sample (including backgrounds and standards) by detector number and sample number. The start date, time and duration of count are recorded. The magnetic tape number and storage location are also recorded. The magnetic tapes will be made available to ARCADIS prior to destruction.

The primary precaution in preparing standards is to maintain the activity level below that which would create more than a one percent Adead-time@ effect in the electronics (approximately 1,000 counts per minute). The temperature of the room is maintained at a constant temperature with thermostatically controlled air conditioning and heating units.

GE(Li) Detector Calibration

Upon preparation of calibration standards for each geometry, all Ge(Li) detectors are calibrated for efficiency with each standard once per year. Efficiencies for other energies are calculated by a computer using a curve-fitting program using the above energy calibrations as reference points. The energy calibration is performed by adjusting electronic discriminators and amplifier gains so that each channel of a 2048 channel pulse height analyzer represents one KeV of energy.

Each standard in solution is diluted in the matrix recommended by the manufacturer of the standard. Each individual counting standard is prepared by volumetric aliquoting of an appropriate activity size.

System Calibration Check

Once a week, the 2-inch diameter standard is counted on each detector for energy, efficiency, calibration, and resolution. The efficiencies of the appropriate energies are calculated and plotted on a control chart. The length of count is present to a time sufficient to obtain counting statistics of 5% or better.

VIII. Data Reduction

Results are calculated as follows:

$$A = \frac{(N/dt-B)}{2.22\,(E)(V)(D)(I)}$$

$$E = \frac{2\,[(N/dt+B)/dt]^{2}}{2.22\,(E)(V)(D)(I)}$$

$$L.T. = \frac{4.66\,(B/dt)^{2}}{2.22(E)(V)(D)(I)}$$

where:

A	=	activity, pCi/gm
E	=	2 sigma counting error, pCi/gm
L.T.	=	detection limit, pCi/gm
I	=	branching intensity of nuclide (fractional abundance)
N	=	number of counts in the peak region
dt	=	counting interval, minutes
2.22	=	dpm/pCi
E	=	counter efficiency in the peak region
V	=	sample mass, grams
D	=	radioactive decay factor from collection to mid-count time
B	=	background count rate in the peak region cpm. This is derived from the sample spectrum as the average of count rates at the borders of the peak region.

Corrective Actions

When a detector is determined to be out of control, the laboratory manager or the person designated by the laboratory manager will evaluate the detector and the electronic system to determine the cause of the problem. The detector will be labeled Anot to be used to analyze samples@ while designated out of control. Corrective action must be documented in the maintenance log kept in the laboratory. If a sample (or samples) had been counted during a time period for which the detector was judged out of control, the sample (or samples) are counted a second time on a detector which is within control limits.

References

Teledyne, Inc. Methods:

PRO-042-5 Determination of Gamma Emitting Radioisotopes.

PRO-042-44 Calibration of Ge(Li) Gamma Ray Spectrometers.

IWL-0032-395 Quality Assurance Manual; Environmental Analysis Department Compliance With 0CFR50, Appendix B, Compliance With Regulatory Guide 4.15.

IWL-0032-365 Quality Control Internal Controls and Audits Environmental Analysis Department.

Appendix L

Handling, Packing, and Shipping Procedures

Handling, Packing, and Shipping Procedures

I. Chain-of-Custody Procedures

Step 1 - Prior to collecting samples, complete the chain-of-custody (COC) form (Attachment L-1) header information by filling in the project number, project name, and the name(s) of the sampling technician(s). Please note that it is important that COC information is printed legibly using indelible ink.

Step 2 - After sample collection, enter the individual sample information by filling in the following COC fields:

1 STA. NO. - Indicates the station number or location that the sample was collected from. Appropriate values for this field include well locations, grid points, or soil boring identification numbers (e.g., MW-3, X-20, SB-30, etc.).

2 Date - Indicates the date that the sample was collected. The date format to be followed should be mm/dd/yyyy (e.g., 03/07/2000).

3 Time - Indicates the time at which the sample was collected. The time value should be presented using the military format. For example, 3:15 P.M. should be entered as 15:15.

4 Comp - This field should be marked with an “X” if the sample was collected as a composite.

5 Grab - This field should be marked with an “X” if the sample was collected as an individual grab sample.

6 Station Location - This field should represent the complete sample name. Although, in some instances it may be similar to the STA. NO. field. An example of a complete sample name is SB-3 (0.5-1.0), where the 0.5-1.0 represents the depth interval in feet from where the sample was collected. Please note that it is very important that the use of hyphens in sample names and the depth units (i.e., feet or inches) remain consistent for all samples entered on the COC form. Sample names may also use the abbreviations “MS/MSD,” “FB,” “TB,” and “DUP” as prefixes or suffixes to indicate that the sample is a matrix spike/matrix spike duplicate, field blank, trip blank, or field duplicate, respectively.

7 Number of Containers - This field represents the number of containers that were collected at the sampling location to be submitted for analysis.

8. Analytical Parameters - The analytical parameters that the samples are being analyzed for should be written legibly on the diagonal lines to the right of the "number of containers" column. The analytical parameters should be chosen from those presented in Table 5. As much detail as possible should be presented to allow the analytical laboratory to properly analyze the samples. For example, polychlorinated biphenyl (PCB) analyses may be represented by entering "PCBs" or "Method 8082." Multiple methods and/or analytical parameters may be combined for each column (e.g., PCBs/VOCs/SVOCs or 8082/8260/8270). These columns should also be used to present project-specific parameter lists (i.e., Appendix IX+3 minus herbs and pests). QA/QC information may also be entered in a separate column for each parameter (e.g., PCBs - MS/MSD) to identify a sample that the laboratory is to use for a specific QA/QC requirement. Each sample that requires a particular parameter analysis will be identified by placing an "X" in the appropriate analytical parameter column.

9 Remarks - The remarks field should be used to communicate special analytical requirements to the laboratory. These requirements may be on a per sample basis such as "extract and hold sample until notified" or may be used to inform the laboratory of special reporting requirements for the entire SDG. Reporting requirements that should be specified in the remarks column include: 1) turnaround time; 2) contact and address where data reports should be sent; 3) name of laboratory project manager; and 4) type of sample preservation that was utilized.

10 Relinquished By - This field should contain the signature of the sampling technician that relinquished custody of the samples to the shipping courier or the analytical laboratory.

11 Date - Indicates the date that the samples were relinquished. The date format should be mm/dd/yyyy (e.g., 03/07/2000).

12 Time - Indicates the time that the samples were relinquished. The time value should be presented using the military format. For example, 3:15 P.M. should be entered as 15:15.

13 Received By - This field should contain the signature of the sample courier or laboratory representative that received the samples from the sampling technician.

Step 3 - Complete as many COC forms as necessary to properly document the collection and transfer of the samples to the analytical laboratory.

Step 4 - Upon completion of the COC forms, forward two copies to the analytical laboratory and retain one for the field records. The field records copy should also be sent to Penny Rabasco, ARCADIS (413) 494-4317.

II. Handling

Step 1 - After completing the sample collection procedures, record the following information in the field notebook with indelible ink:

- project number and site name;
- sample identification code and other sample identification information, if appropriate;
- sampling method;
- date;
- name of sampler(s);
- time;
- location (project reference); and
- any other comments.

Step 2 - Fill in sample label (Attachment L-2) with the following information in indelible ink:

- sample type (e.g., surface water);
- project number and site name;
- sample identification code and other sample identification information, if applicable;
- analysis required;
- date;
- time sampled;
- initials of sampling personnel;
- sample type (composite or discrete);
- tissue preparation procedure (biota; e.g. fillets, whole body), if applicable; and
- preservative added, if applicable.

Step 3 - Cover the label with clear packing tape to secure the label onto the container. Step 4 - Check the caps on the sample containers to ensure that they are tightly sealed. Step 5 - Wrap the sample container cap with clear packing tape to prevent it from becoming loose. Step 6 - Place a signed custody seal label (Attachment L-3) over the cap such that the cap cannot be removed without breaking the custody seal. Alternatively, if shipping several containers in a cooler, custody seal evidence tape may be placed on the shipping container as described below.

III. Packing

Step 1 - Using duct tape, secure the outside and inside of the drain plug at the bottom of the cooler that is used for sample transport.

Step 2 - Place each container or package in individual polyethylene bags (resealable-type) and seal. If a cooler temperature blank is supplied by the laboratory, it should be packaged following the same procedures as the samples. If the laboratory did not include a temperature blank, do not add one since the sample temperature will be determined by the laboratory using a calibrated infrared thermometer.

Step 3 - Place 1 to 2 inches of cushioning material (i.e., vermiculite) at the bottom of the cooler.

Step 4 - Place the sealed sample containers upright in the cooler.

Step 5 - Package ice or blue ice in small resealable-type plastic bags and place loosely in the cooler. Do not pack ice so tightly that it may prevent addition of sufficient cushioning material. Samples placed on ice will be cooled to and maintained at a temperature of approximately 4°C.

Step 6 - Fill the remaining space in the cooler with cushioning material.

Step 7 - Place the completed COC forms (Attachment L-1) in a large resealable-type bag and tape the bag to the inside of the cooler lid.

Step 8 - Close the lid of the cooler and fasten with packing tape.

Step 9 - Wrap strapping tape around both ends of the cooler.

Step 10 - Mark the cooler on the outside with the following information: shipping address, return address, “Fragile” labels (Attachment L-4) on the top and on one side, and arrows indicating “This Side Up” (Attachment L-4) on two adjacent sides.

Step 11 - Place custody seal evidence tape (Attachment L-4) over front right and back left of the cooler lid and cover with clear plastic tape.

Note: Procedure numbers 2, 3, 5, and 6 may be modified in cases where laboratories provide customized shipping coolers. These coolers are designed so the sample bottles and ice packs fit snugly within preformed Styrofoam cushioning and insulating packing material.

IV. Shipping

All samples will be delivered by an express carrier within 48 hours of sample collection. Alternatively, a laboratory courier may be used for sample pickup. If parameters with short holding times are being analyzed [i.e., VOCs (EnCore® Sampler), nitrate, ortho-phosphate (dissolved), and BOD], sampling personnel will take precautions to assure that the maximum holding times for these parameters will not be exceeded.

The following COC procedures will apply to sample shipping:

- Relinquish the sample containers to the laboratory via express carrier or laboratory courier. Alternatively, samples may be taken to the sample handling area at the GE Pittsfield laboratory where they will be packaged for transport. The signed and dated forms should be included in the cooler. The express carrier will not be required to sign the COC forms.

- When the samples are received by the laboratory, the laboratory personnel shall complete the COC by recording the data and time of receipt of samples, measure and record the internal temperature of the shipping container, and then check the sample identification numbers on the containers to ensure that they correspond to the COC forms.

Attachment L-1

Chain of Custody Form

ID#: 11057

CHAIN OF CUSTODY & LABORATORY ANALYSIS REQUEST FORM

Page ___ of ___

Lab Work Order #

Send Results to:	
Contact & Company Name:	Telephone:
Address:	Fax:
City State Zip	E-mail Address:
Project Name/Location (City, State):	Project #:
Sampler's Printed Name:	Sampler's Signature:

Preservative							
Filtered (✓)							
# of Containers							
Container Information							

PARAMETER ANALYSIS & METHOD

Keys

Preservation Key:
A. H_2SO_4
B. HCL
C. HNO_3
D. NaOH
E. None
F. Other: ______
G. Other: ______
H. Other: ______

Container Information Key:
1. 40 ml Vial
2. 1 L Amber
3. 250 ml Plastic
4. 500 ml Plastic
5. Encore
6. 2 oz. Glass
7. 4 oz. Glass
8. 8 oz. Glass
9. Other: ______
10. Other: ______

Matrix Key:
SO - Soil, SE - Sediment, NL - NAPL/Oil
W - Water, SL - Sludge, SW - Sample Wipe
T - Tissue, A - Air, Other: ______

Sample ID	Collection Date	Collection Time	Type (✓) Comp	Type (✓) Grab	Matrix								REMARKS

Special Instructions/Comments:

☐ **Special QA/QC Instructions(✓):**

Laboratory Information and Receipt		Relinquished By	Received By	Relinquished By	Laboratory Received By
Lab Name:	Cooler Custody Seal (✓)	Printed Name:	Printed Name:	Printed Name:	Printed Name:
☐ Cooler packed with ice (✓)	☐ Intact ☐ Not Intact	Signature:	Signature:	Signature:	Signature:
Specify Turnaround Requirements:	Sample Receipt:	Firm:	Firm/Courier:	Firm/Courier:	Firm:
Shipping Tracking #:	Condition/Cooler Temp: ______	Date/Time:	Date/Time:	Date/Time:	Date/Time:

Distribution: **WHITE – Laboratory returns with results** **YELLOW – Lab copy** **PINK – Retained by BBL**

Attachment L-2

Sample Label

ARCADIS

Project #		Date
Sample I.D.		
Sample Type ☐ Soil/Sediment ☐ Water	Collection Mode ☐ Composite ☐ Grab	Time
Analysis		
Sampler(s)	Preservative	

Attachment L-3

Custody Seal Label

CUSTODY SEAL

SEALED BY

____

DATE __________ **TIME** __________

6723 Towpath Road, P.O. Box 66 • Syracuse, New York 13214-0066 • Tel 315.446.9120

Attachment L-4

Shipping Container Labels

fragile
HANDLE
WITH CARE

Appendix M

Shipment of Department of Transportation Hazardous Materials

Shipment of Department of Transportation Hazardous Materials

I. Introduction

Selected materials collected and shipped to analytical laboratories during this project may be subject to the requirements of the United States Department of Transportation (USDOT) Hazardous Materials Regulations (HMR) and the International Air Transport Association (IATA) Dangerous Goods Regulations (DGR).

II. Shipment Summary

The sample shipment procedure consists of 11 steps. A summary of the 11 steps for shipping samples is described below.

- Determination of Proper Shipping Name and Material Classification for the Material.
- Identify Shipping Name and Shipping Requirements in List of Dangerous Goods.
- Determine Mode of Transport and Carrier.
- Determine Operator-/Carrier-Specific Requirements.
- Define Quantity Limitations for Materials to be Shipped.
- Identify Packing.
- Select Packaging Components and Package Material.
- Pack Samples and Verify Packaging Restrictions, Specifications, and Quantities.
- Implement Marking and Labeling Requirements for Package.
- Complete Shipper's Declaration for Dangerous Goods.
- Record Acceptance of Shipment by Dangerous Goods Transporter.

III. Sample Shipping Procedure

Shipment procedures consist of 11 steps. A summary of the 11 steps for shipping of materials is described below.

Step 1 - Determination of Proper Shipping Name and Material Classification for the Material: Based on available information and characteristics of the material, a determination of the classification of the material into Dangerous Goods/Hazardous Materials Class must be made. Classification into one or more of the following classes shall be made:

Class 1 Explosives;

Class 2 Gases;

Class 3 Flammable Liquids

Class 4 Flammable Solids; substances liable to spontaneous combustion; substances which, in contact with water, emit flammable gases.

Class 5 Oxidizing Substances and Organic Peroxides;

Class 6 Toxic and Infectious Substances;

Class 7 Radioactive Material;

Class 8 Corrosives; and

Class 9 Miscellaneous Dangerous Goods (includes Polychlorinated Biphenyls).

Step 2 - Identify Shipping Name and Shipping Requirements in List of Dangerous Goods Hazardous Materials Table: Based on the classification of the material and the proper shipping name for the material, the specific entry in the List of Dangerous Goods (Section 4 of the IATA DGR) or USDOT Hazardous Materials Table (HMR 172.101) can be located and the specific shipping requirements for the sample can be identified.

Step 3 - Determine Mode of Transport and Carrier: In order to ensure compliance with specific modal and operator requirements, the selected means of transport and the carrier must be identified.

Step 4 - Determine Operator-/Carrier-Specific Requirements: Section 2 of the IATA DGR and USDOT HMR will be reviewed to determine carrier-specific requirements (i.e., Federal Express, Delta Airlines, etc.) and the List of Dangerous Goods/USDOT Hazardous Materials Table will be reviewed for model-specific restrictions (i.e., cargo aircraft, passenger aircraft, etc.).

Step 5 - Define Quantity Limitations for Materials to be Shipped: The List of Dangerous Goods (Section 4 of the IATA DGR) or USDOT Hazardous Materials Table (HMR 172.101) entry shall be reviewed for the material being shipped and specific quantity limitations for the material (inner packaging limit/outer packaging limit) shall be identified.

Step 6 - Identify Packing Procedure: The List of Dangerous Goods/USDOT Hazardous Materials Table (HMR 172.101) shall be reviewed for the material and specific packing instructions for the material shall be identified.

Step 7 - Select Packaging Components and Package Material: Corresponding numbered packing instructions in Section 5 of the IATA DGR provide acceptable packaging configurations for each dangerous good. USDOT HMR (173) provides acceptable packaging configurations for hazardous materials to be shipped via domestic ground transportation.

Step 8 - Pack Material and Verify Packaging Restrictions, Specifications, and Quantities: Pack material in appropriate inner and outer packaging in accordance with numbered packing instruction in Section 5 of the IATA DGR or USDOT HMR (173). Verify that packaging restrictions, specifications, and maximum package quantities meet the requirements of the packing instruction.

Step 9 - Implement Marking and Labeling Requirements for Package: Prior to shipping, the completed package must be marked and labeled in accordance with Section 7 of the IATA DGR or USDOT HMR. Markings and labels may include, but not be limited to: the shipper's name/identification, proper shipping name, UN identification number, hazard class, subsidiary hazards, and package orientation.

Step 10- Complete Shipper's Declaration for Dangerous Goods/Hazardous Materials Shipping Papers: An executed Shipper's Declaration for Dangerous Goods/Hazardous Shipping Papers and/or carrier-specific airbill (for air transport) must be presented at consignment of shipment. The Shipper's Declaration for Dangerous Goods may include, but not be limited to: transport details, shipper's name/identification, nature and quantity of dangerous goods, proper shipping name, UN identification number, hazard class, packing group, subsidiary hazards, packing instruction number, type of packing, authorization, emergency contact number, and additional handling information.

Step 11- Record Acceptance of Shipment by Dangerous Goods Transporter: Upon consignment of the shipment to a dangerous goods carrier, a completed copy of the Declaration for Dangerous Goods/Hazardous Materials Shipping Papers will be maintained by the shipper and copies provided to any emergency contacts identified on the declaration.

Appendix N

Photoionization Detector Field Screening Procedures

Photoionization Detector Field Screening Procedures

I. Introduction

Field screening with a photoionization detector (PID) is a procedure to measure relative concentrations of volatile organic and inorganic compounds. Field screening can be conducted in the head space of soil samples (as described below) with the PID. The characteristics of these instruments are found in Attachment N-1.

II. Materials

- MiniRAE 2000 or equivalent
- PID;
- sample jars;
- aluminum foil; and
- field notebook.

III. Procedures

Soil samples will be field screened upon collection with the PID for a relative measure of the total volatile organic concentration. PID readings will be recorded in the field notebook or the boring logs, whichever is appropriate.

The recommended procedures for conducting analytical screening of soils utilizing a portable PID are taken from the Massachusetts DEP document entitled "Management Procedures for Excavated Soils Contaminated with Virgin Petroleum Oils Policy #WSC-89-001," June 30, 1989. The procedures follow:

Step 1 - Half-fill two clean glass jars with the sample (if sufficient quantities of soil are available) to be analyzed. Quickly cover each open top with one or two sheets of clean aluminum foil and subsequently apply screw caps to tightly seal the jars. Sixteen-ounce (approximately 500 mL) soil or "mason"-type jars are preferred; jars less than 8 ounce (approximately 250 mL) total capacity may not be used.

Step 2 - Allow headspace development for at least 10 minutes. <u>Vigorously shake jars for 15 seconds both at the beginning and end of the headspace development period.</u> Where ambient temperatures are below 32°F (0°C), headspace development should be within a heated building.

Step 3 - Subsequent to headspace development, remove screw lid to expose the foil seal. Quickly puncture foil seal with instrument sampling probe to a point about one-half of the headspace depth. Exercise care to avoid contact with water droplets or soil particulates.

Step 4 - Following probe insertion through foil seal, record the highest meter response for each sample as the jar headspace concentration. Using the foil seal/probe insertion method, maximum response should occur between 2 and 5 seconds. Erratic meter response may occur at high organic vapor concentrations or conditions of elevated headspace moisture, in which case headspace data should be recorded and erratic meter response noted.

Step 5 - The headspace screening data from both jar samples should be recorded and compared; generally, replicate values should be consistent to plus or minus 20%. It should be noted that in some cases (e.g., 6-inch increment soil borings), sufficient quantities of sample may not be available to perform duplicate screenings. A single screening will be considered sufficient for this case.

Step 6 - PID field instruments shall be operated and calibrated to yield “total organic vapors” in ppm (v/v) as benzene. PID instruments must be operated with at least a 10.0 eV (±) lamp source. Operation, maintenance, and calibration shall be performed in accordance with the manufacturer’s specifications presented in Attachment N-1. For jar headspace analysis, instrument calibration shall be checked/adjusted at least twice per day at the beginning and end of each day of use. Calibration will exceed twice per day if conditions and/or manufacturer’s specifications dictate.

Step 7 - Instrumentation with digital (LED/LCD) displays may not be able to discern maximum headspace response unless equipped with a “maximum hold” feature or strip-chart recorder.

Attachment N-1

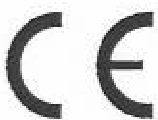
MiniRAE 2000 Operation and Maintenance Manual

MiniRAE 2000

Portable VOC Monitor
PGM-7600

OPERATION AND MAINTENANCE MANUAL

(Document No. 011-4001-[illegible])
Rev. A



RAE SYSTEMS INC.
680 West Maude Avenue, #[illegible]
Sunnyvale, CA 94086

December 1998

2. OPERATION OF MINIRAE 2000

The MiniRAE 2000 Portable VOC Monitor is a compact Monitor designed as a broadband VOC gas monitor and datalogger for work in a hazardous environment. It gives real time measurements and activates alarm signals whenever the exposure exceeds preset limits. Prior to factory shipment the MiniRAE 2000 is preset with default alarm limits and the sensor is pre-calibrated with standard calibration gas. After the monitor is fully charged, it is ready for immediate operation.

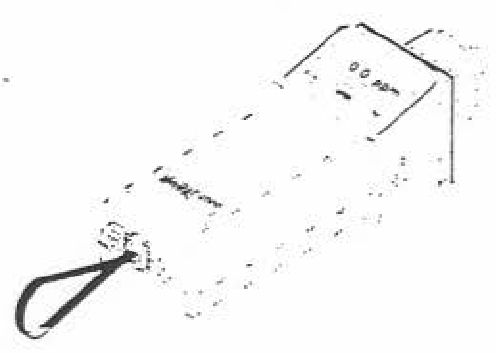


Figure 2-1 MiniRAE 2000

2.1 Physical Description

The main components of the MiniRAE 2000 Portable VOC monitor include:

- Three keys for user to interact with the monitor: 1 operation key and 2 programming keys for normal operation or programming of the monitor
- LCD display with back light for direct readout and calculated measurements
- Buzzer and red LED's for alarm signaling whenever the exposures exceed preset limits
- Wrist strap
- Charge contact for plugging directly to the charging station
- Gas entry and exit ports
- Serial communication port for PC interface
- External alarm and analog output port
- Protective rubber cover

2.2 Keys and Display

Figure 2.2 shows the LCD display and the keypad on the front panel of the monitor. The function of the 3 keys during normal operation are summarized below:

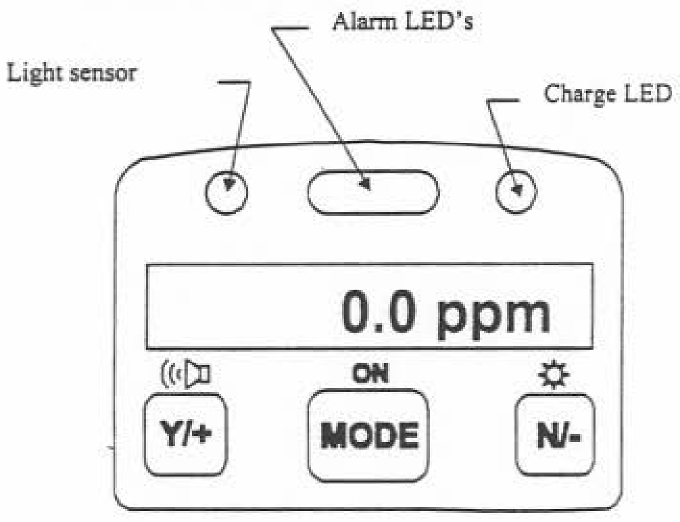


Figure 2.2 LCD display and Keypad

Key	Function in Normal Operation
[MODE]	-Turn on/off the power* and step through menu items
[N/-]	-Toggle on/off the back light, negative acknowledge/decrement value
[Y/+]	-Start measurement, positive acknowledge/increment value

* Pressing and holding **[MODE]** key for 5 seconds turns off the power to the monitor. Monitor will beep once per

second and display countdown timer during power-down sequence. Press **[MODE]** key momentarily to step through menu items. To save time, press any key during message scrolling to skip to the end of the message.

2.3 Power On/Off

To turn on the MiniRAE 2000 portable VOC monitor, press **[MODE]** key for one second and release. The audio buzzer will beep once and the air pump will turn on. The display will show "ON !.." and then " Ver n.nn" to indicate the unit's current firmware version number. Next displayed are the serial number, the model number, Operating mode, current date and time, unit internal temperature, gas selected, high low, STEL, TWA/AVG alarm limits, battery voltage, and shut off voltage . Also displayed are internal mode settings such as User mode, Alarm mode, datalog time remaining and log periods in the respective order.

To turn off the MiniRAE 2000 portable VOC monitor, press and hold the **[MODE]** key for 5 seconds. The monitor will beep once per second during the power-down sequence with a count down timer showing the number of remaining seconds . The message "Off !.." flashes on the LCD display and the display will go blank indicating that the monitor is turned off.

Data protection during power off

When the monitor is turned off, all the current real time data including last measured value are erased. However, the datalog data is preserved in non-volatile memory. Even if the battery is disconnected, the datalog data will not be lost. While the power is off, the real time clock will continue to operate until the battery is completely drained (usually in 4-5 days without any charging.). If the battery

is completely drained or is disconnected from the monitor for more than 30 minutes, the real time clock will be lost. In this case, the user needs to enter the real time clock information again, as described in Section 4, or send the PC clock during configuration through the PC communication.

2.4 Operation

The **MiniRAE 2000** VOC monitor has two operation modes: **Survey** and **Hygiene** mode. The **Survey mode** allows the user to manually start and stop the monitoring/measuring operation and display certain exposure values. In the **Hygiene mode,** the monitor runs continuously after the monitor is turned on.

2.4.1 Survey mode

After the monitor is turned on, it runs through the start up menu. Then a message **"Ready.."** is displayed (see figure below). At this point, the user has two options; 1) step through the operation menu, or 2) take a measurement.

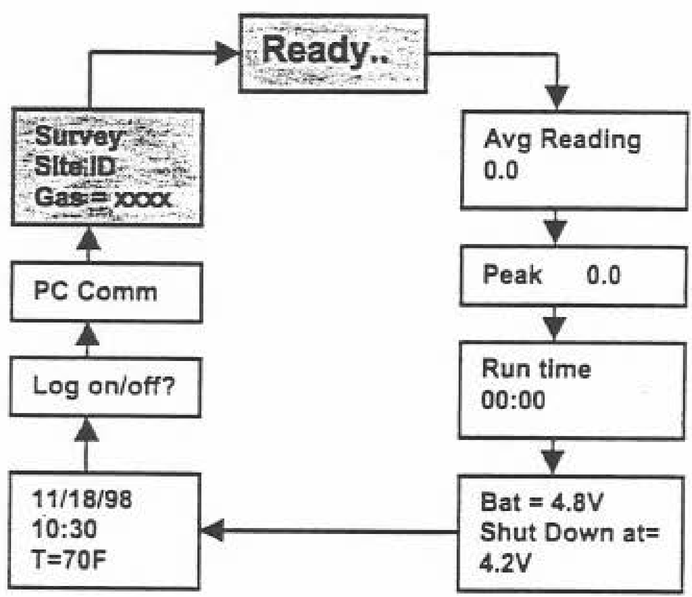
Press the **[MODE]** key to cycle through the idle operation menu. The PID sensor and pump are turned off during this idle operation.

Main operation menu displays include:

- **"Ready..."**
- **Avg reading**
- **Peak reading**
- **Run time**
- **Current battery voltage and shutdown voltage**
- **Date, time and temperature**
- **Log on/off?**
- **PC communication?**

- **Survey, Site ID and Gas Name**

The displays are arranged in a "round robin" order: To choose a specific display, press the **[MODE]** key one or more times until the desired display appears.



Note: To get back to "Ready" mode from any of the above display, press **[MODE]** key repeatedly until the "Ready" message appear.

More details on the Main Operation Menu:

- READY: The monitor is ready to take a measurement. Press the **[MODE]** key to advance to the next menu. Or Press **[Y/+]** key to start a measurement. (Read **"Taking a Measurement"** on page 2-11 for details)

- **AVERAGE READING:** Running average since the start of the measurement.
- **PEAK READING:** The highest instantaneous reading since the start of the measurement.
- **RUN TIME:** The current measurement has been last.
- **CURRENT BATTERY VOLTAGE and SHUT DOWN VOLTAGE:** The present battery voltage is displayed.

Note: A fully charged battery pack should show 4.8 volts or higher. When the battery voltage falls below 4.4 volts, a flashing "Bat" will appear as a warning message. There are about 20-30 minutes of run time left before the monitor turns off automatically, when the battery voltage falls below 4.2 volts.

- **DATE, TIME, TEMPERATURE:** This menu displays the current date (month/day/year), time (24-hour format), and internal unit temperature in degrees Fahrenheit.
- **LOG ON/OFF?** Allows the user to start datalogging of the current measurement. A superscript "L" flashes in the ppm measurement display when datalogging is on.

Note: Before datalogging can be turned on, this function must be enabled as described in Section 4.6.4.

- **PC COMMUNICATION:** Allows the user to upload data from the MiniRAE 2000 to a Personal Computer (PC) or send/receive configuration information between a PC and the MiniRAE 2000. Connect the monitor to a serial port of a PC, and start the MiniRAE 2000 application software. Press the **[Y/+]** key and the LCD

displays "pause monitor, ok?". Press the **[Y/+]** key one more time, the display shows "Comm...". The monitor is now ready to receive commands from the PC.

- **CURRENT OPERATING MODE:** The monitor displays the current operating mode e.g.; "Survey", the site ID, gas name and then returns to "Ready.."

To choose a specific display, press the **[MODE]** key one or more times until the desired display appears.

Taking a measurement:

There are two ways to start a measurement. 1) Operating in Hygiene mode. 2) Manually start and stop measurement in Survey mode. To start an measurement in Hygiene mode, please refer to Section 4.7.1 on "Change Op mode". To start a measurement in Survey mode, the MiniRAE 2000 monitor must first be in the "Ready..." mode. This is the mode that the monitor normally powers up.

Measurement phases:

- **Ready**
- **Start measurement**
- **Measurement Display and datalogging**
- **Stop measurement**

Ready

Unit is ready to start a sample.

Start Measurement

Press the **[Y/+]** key to start the measurement cycle.

Display will show the site ID and then the gas selected for measurement. The pump will start and the reading will be displayed.

Measurement Display and Datalog

Instantaneous readings of the gas concentration in parts per million (ppm) are updated every second. A flashing superscript "L" is displayed when datalogging is on. Datalog information is saved only after one full datalog period is completed (see Section 4.6.5)

Stop Measurement

Press the **[MODE]** key and the display shows " STOP?". Press **[N/-]** key to continue measurement and **[Y/+]** key to stop the measurement and datalog event. The pump stops automatically when measurement is stopped.

Automatic Increment of Site ID

Every time a measurement is taken, the site ID will be incremented by one automatically in Survey mode.

Variable Alarm Signal

During Survey mode operation, if the measurement exceeds the low limit, the buzzer and flashing alarm will be activated. The frequency of the alarm is proportional to the measurement value. When the measurement value is lightly about the low alarm, the buzzer and LED will beep and flash once a second. When the measurement value reaches the high alarm limit, the buzzer and LED will beep and flash 7 times per second.

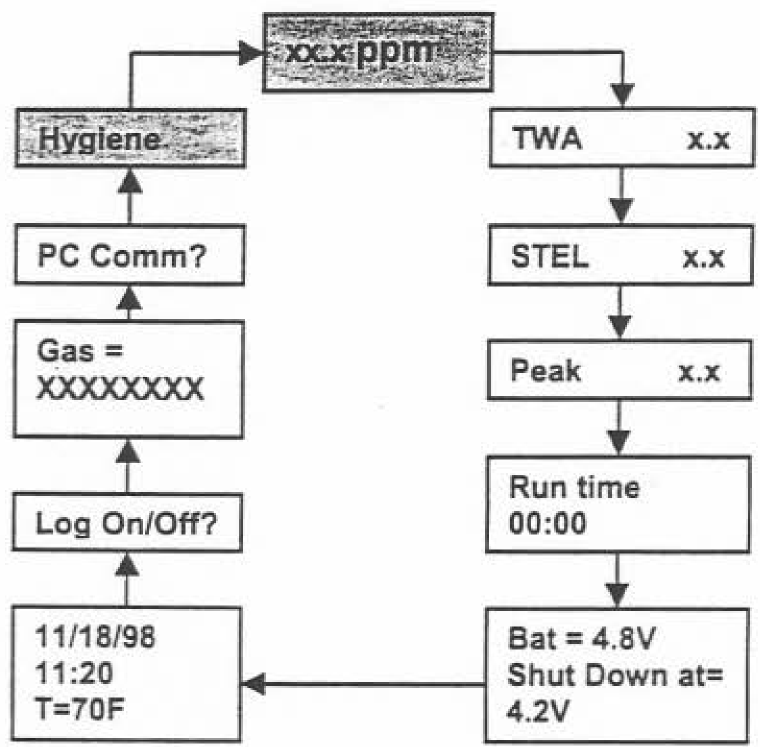
2.4.2 Hygiene Mode

In Hygiene Mode, the unit will continuously taking measurements, once the power is turned on. After the initial start-up sequence displaying the current monitor settings, the LCD displays the instantaneous readings.

The Hygiene operation menu displays include:

- **Real time readings in ppm**
- **Current TWA/Avg, STEL and Peak values (see Section 4.6.6.)**
- **Run time**
- **Current battery voltage and shut down voltage**
- **Date, time and temperature**
- **Log on/off?**
- **Gas name**
- **PC communication?**
- **Hygiene**

Detailed description of most of these displays are the same as Section 2.4.1.



To choose a specific display, press the **[MODE]** key one or more times until the desired display appears.

Note: To get back to instantaneous reading from any of the above display, press **[MODE]** key repeatedly until the "XX.X ppm" display appears.

2.5 Alarm Signals

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings: Low, High, TWA and STEL). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn the user of the alarm condition.

In addition, the MiniRAE 2000 will alarm if one of the following conditions occurs: battery voltage falls below a pre-set voltage level (4.4 V), failure of UV lamp, pump stall, or when the datalog memory is full. When the low battery alarm occurs, there will be approximately 20-30 minutes of operating time remaining. When the battery voltage falls below 4.2 V, the monitor will turn off automatically.

Alarm Signal Summary :

Condition	Alarm Signal
Gas exceeds "High Alarm" limit	3 beeps/flashes per second
Gas exceeds "Low Alarm" limit	2 beeps/flashes per second
Gas exceeds "TWA" limit	1 Beeps/flashes per seconds
Gas exceeds "STEL" limit	1 Beeps/flashes per seconds
Pump failure	3 beeps/flashes per second plus "Pump" message on LCD
PID lamp failure	3 beeps/flashes per second plus "Lamp" message on LCD
Low battery	1 flash per second, 1 beep per minute plus "Bat" message on LCD
Memory full	1 flash per second plus "Mem" message on LCD

Alarm Signal Testing:
Under normal non-alarm conditions, it is possible to test the MiniRAE 2000 LED and buzzer in Special Diagnostic Mode. (See Section 8 for details)

2.6 Preset Alarm Limits and Calibration

The MiniRAE 2000 portable VOC monitor is factory calibrated with standard calibration gas, and is programmed with default alarm limits. There are 102 gases setting stored in library. Some examples of calibration and alarm limits are shown below. Refer to Section 4 for programming procedures for selecting a different gas, perform a calibration or set new alarm limits.

Factory Calibration and Preset Alarm Limits

Cal Gas	Cal Span	unit	Low	High	TWA	STEL
Isobutylene	100	ppm	50	100	100	250
Hexane, n-	100	ppm	500	750	500	750
Xylene, m-	100	ppm	100	150	100	150
Benzene	5	ppm	2	5	5	2
Styrene	50	ppm	20	40	20	40
Toluene	100	ppm	50	100	50	100
Vinyl Chloride	10	ppm	5	10	5	10
Custom	100	ppm	50	100	50	100

2.7 Integrated Sampling Pump

The MiniRAE 2000 portable VOC monitor includes an integrated sampling pump. This is a diaphragm type pump that provides 450-550 cc per minute flow rate. Connecting a Teflon or metal tubing with 1/8 inch inside diameter to the gas inlet port of the MiniRAE 2000, this pump can pull in air samples from 200 feet away horizontally, or 90 feet vertically, at about 3 feet per second flow speed.

The pump turns on when a measurement is started, and turns off when the sample is manually stopped in Survey mode or when the unit is turned off from Hygiene Mode.

If liquid or other objects are pulled into the inlet port filter, causing the pump to stall, the monitor will detect the obstruction and shut down the pump immediately. The alarm will be activated and a flashing error message "Pump" will be also displayed on the LCD display.

The user needs to acknowledge the pump shut off condition by clearing the obstruction and pressing the **[Y/+]** key to re-start the pump.

The pump stall threshold is set in the special Diagnostic Mode (Section 8).

4.4 Calibrate and Select Gas

In the first menu of the programming mode, the user can perform functions such as calibration of the MiniRAE 2000 Monitor, select default cal memories, and modify cal memories. See Table 4.4.

Table 4.4

Calibrate/Select Gas Sub-Menu
Fresh Air Cal ?
Span Cal ?
Select Cal Memory ?
Change Span Value?
Modify Cal Memory ?*
Change Correction Factor ?*

*** Sub-menus not available for Cal mem #0.**

Calibrating the MiniRAE 2000 monitor is a two-point process using "fresh air " and the standard reference gas (also known as span gas). First a "Fresh air" calibration, which contains no detectable VOC (0.0 ppm), is used to set the zero point for the sensor. Then a standard reference gas that contains a known concentration of a given gas is used to set the second point of reference.

Note: The span value must be set prior to calibrating for fresh air or span.

In addition to calibrations, the first menu allows the user to store calibrations for up to 8 different measurement gases.

The default gas selections are as follows:
Cal Memory #0......Isobutylene
Cal Memory #1......Hexane
Cal Memory #2......Xylene
Cal Memory #3......Benzene
Cal Memory #4......Styrene
Cal Memory #5......Toluene
Cal Memory #6......Vinyl Chloride
Cal Memory #7......Custom?

With the exception of memory 0, the other 7 cal memories may be modified to one of 102 preprogrammed chemicals or to a user-defined custom gas. The user may calibrate with either isobutylene or the gas selected. If Isobutylene is used, the library correction factor is applied before the readings are displayed. If the selected measurement gas has been used to calibrate, no correction factor is applied.

To change a default gas to a library or custom gas , first go to Select Cal Memory (Section 4.4.3) and then proceed to Modify Cal Memory (Section 4.4.5) to enter the desired gas.

4.4.1 Fresh Air Calibration

This procedure determines the zero point of the sensor calibration curve. To perform a fresh air calibration, use the calibration adapter to connect the MiniRAE 2000 to a "fresh" air source such as from a cylinder or Tedlar bag (option accessory). The "fresh" air is clean dry air without any organic impurities. If such an air cylinder is not available, any clean ambient air without detectable contaminant or a charcoal filter can be used.

1. The first sub-menu shows: "Fresh air Cal ?"
2. Make sure that the MiniRAE 2000 is connected to one of the "fresh" air sources described above.
3. Press the **[Y/+]** key, the display shows "zero in progress" followed by "wait.." and a countdown timer.
4. After about 15 seconds pause, the display will show the message "zeroed... reading = X.X ppm...". Press any key or wait about 20 seconds, the monitor will return back to "Fresh air Calibration?" submenu.

Note: The charcoal filter has a check box so that user can mark off a box each time the filter has been used. The charcoal filter should be replaced after 4 calibrations.

4.4.2 Span Calibration

This procedure determines the second point of the sensor calibration curve for the sensor. A cylinder of standard reference gas (span gas) fitted with a 500cc/min. flow-limiting regulator or a flow-matching regulator is needed to perform this procedure. Alternatively, the span gas can first be filled into a Tedlar Bag. Connect the calibration adapter to the inlet port of the MiniRAE 2000 Monitor, and connect the tube to the regulator or Tedlar bag.

Before executing a span calibration, make sure the span value has been set correctly (see next sub-menu).

1. Make sure the monitor is connected to one of the span gas sources described above.
2. Press the **[Y/+]** key at the "Span Cal?" to start the calibration. The display shows the gas name and the span value of the corresponding gas.
3. The display shows " Apply gas now!". Turn on the valve of the span gas supply.
4. Display shows " wait.... 30" with a count down timer showing the number of remaining seconds while the monitor performs the calibration.
5. To abort the calibration, press any key during the count down. The display shows "Aborted!" and return to "Span Cal?" sub-menu.
6. When the count down timer reaches 0, the display shows the calibrated value.

Note: The reading should be very close to the span gas value.

7. During calibration, the monitor waits for an increased signal before starting the countdown timer. If a minimal response of 30 raw counts is not obtained after 35 seconds, the monitor displays "No Gas!". Check the span gas valve is open and for lamp or sensor failure before trying again.
8. The calibration can be started manually by pressing any key while the "Apply gas now!" is displayed.
9. After a span calibration is completed, the display will show the message "Span Cal Done! Turn Off Gas"
10. Turn off the flow of gas. Disconnect the calibration adapter and Tedlar bag from the MiniRAE 2000 Monitor.
11. Press any key and it returns back to "Span Gas Cal?".

4.4.3 Select Cal Memory

This function allows the user to select one of eight different memories for calibration and gas selection. The default gas selections are listed in Section 4.4

1. "Select Cal Memory?" is the third sub-menu item in the Calibration sub-menu. Pressing the **[Y/+]** key, the display will show "Gas =" gas name followed by "Mem # x?"
2. Press **[N/-]** to scroll through all the memory numbers and the gas selections respectively. Press **[Y/+]** to accept the displayed Cal Memory number.
3. After the **[Y/+]** key is pressed, the display shows "Save?". Press **[Y/+]** key to save and proceed. Press **[N/-]** to discard the entry and advance to the next sub-menu.
4. If the gas of a newly selected Cal Memory number is not calibrated, the display shows " Not Cal'ed" to warn the user, that a correction factor will be applied if the memory is not calibrated.
5. If the gas of a newly selected cal memory number has been calibrated previously, the display shows "Last calibrated xx/xx/xx".

4.4.4 Change Span Value

This function allows the user to change the span values of the calibration gases.

1. "Change Span Value?" is the fourth sub-menu item in the Calibration sub-menu
2. Press **[Y/+]**, display shows the gas name and the span value. A cursor will blink at the first digit of the Span value. To modify the span gas value, go to step 3. Otherwise, press and hold the **[MODE]** key for 1 second to accept the previously stored span gas value and move to the next sub-menu.
3. Starting from the left-most digit of the span gas value, use the **[Y/+]** or **[N/-]** key to change the digit value and press **[MODE]** key momentarily to advance to next digit. Repeat this process until all digits are entered. Press and hold the **[MODE]** for 1 second to exit.
4. The display shows "Save?". To accept the new value, press the **[Y/+]** key. Press the **[N/-]** key or the **[MODE]** key to discard the change and move to the next sub-menu.

4.4.5 Modify Cal Memory

If the current cal memory number selected is not memory 0, users will be prompted whether to modify the settings of the selected cal memory. Press **[Y/+]** to modify the cal memory and **[N/-]** to go to the next sub-menu.

Once **[Y/+]** is pressed the LCD display will show the current memory number, current Gas selected and prompt user for acceptance of current gas selected.

1. Press **[N/-]** to modify the gas selection if desired. Or press **[Y/+]** key to skip the change of gas selection, and proceed to the next sub-menu.

2. After pressing **[N/-]**, display shows "Copy gas from library?". Press **[Y/+]** to accept or **[N/-]** for the next sub-menu, "Enter Custom gas?"

3. In the "Copy gas from library" submenu, use **[Y/+]** and **[N/-]** keys to scroll through the selections in the library. Press **[MODE]** key momentarily to select the gas. The display shows "Save?". Press **[Y/+]** to save or **[N/-]** to discard the changes and proceed to next sub-menu.

4. In the Custom gas sub-menu, the user can enter the gas name. Press the **[Y/+]** or **[N/-]** key to cycle through all 26 letters and 10 numerals. Press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move to the next digit to the right. Repeat this process until all digits (up to 8 digits) of the custom gas name is entered.

Press and hold the **[MODE]** key for 1 second to exit the name entry mode. The display will show "save?". Press **[Y/+]** to save the entry, or **[N/-]** to discard the changes.

Appendix O

Temperature, Turbidity, Specific Conductivity, pH, Oxidation/Reduction Potential, and Dissolved Oxygen Field Measurement Procedures

Temperature, Turbidity, Specific Conductivity, pH, Oxidation/Reduction Potential, and Dissolved Oxygen Field Measurement Procedures

I. Introduction

Hydrochemical parameters such as specific conductance, pH, temperature, turbidity, oxidation/reduction potential (ORP), and dissolved oxygen (DO) of groundwater or surface water are measured in the field. The pH and conductivity of the ground/surface water will be recorded using a portable meter with temperature compensating pH and conductivity electrodes. If the portable meter does not have a temperature display, the temperature will be taken with a glass, digital, or bimetal thermometer. The pH, specific conductivity, and DO meters will be calibrated twice-daily (at a minimum) in the field. Calibration will occur before use and at the end of the day, and according to the manufacturer's instructions and the procedures specified herein (which include USEPA analytical methods). Additional calibration may be performed if conditions and/or manufacturer's specifications dictate. All calibration data should be recorded and filed with the project field records.

II. Materials

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

- two water quality (temperature/pH/specific conductivity/ORP/turbidity/DO) meters (one for back up) and flow-through measurement cells. Several brands may be utilized, including:
  - YSI 6-Series Multi-Parameter Instrument;
  - Hydrolab Series 3 or Series 4a Multiprobe and Display; and/or
  - Horiba U-10 (for bailing procedures) or U-22 (for flow-through measurement cells) Water Quality Monitoring System.
- appropriate calibration standards;
- thermometer;
- 500-mL glass container;
- cleaning equipment (as required in Appendix W);
- fine screw driver;
- extra batteries;
- field notebook; and
- appropriate log forms.

III. Procedures

A. Calibration

The detailed procedure for the calibration of field instruments used to measure water quality is outlined below. This procedure is generally derived from the USEPA Region I Standard Operating Procedure (SOP) entitled *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductivity, oxidation/reduction potential [ORP], and turbidity)* dated June 3, 1998 and revised January 19, 2010, which is included as Attachment O-1.

- *Temperature:* Perform annual accuracy check according to procedures outlined in Attachment O-1, Steps 1 through 3, as needed.

- *pH*: Perform a two-point calibration according to procedures outlined in Attachment O-1, Steps 1 through 8 and 11 through 12. Note that if pH values observed during field activities are outside the initial calibration range, re-calibration will be required. Alternatively, a three-point calibration may be performed according to procedures outlined in Attachment O-1, Steps 1 through 12.

- *DO*: Perform a saturated air calibration according to procedures outlined in Attachment O-1, Steps 1 through 10. The DO probe's membrane and electrolyte solution should be replaced prior to the sampling period if the instrument has been inactive for an extended time period, or as an initial response if erratic measurements are observed.

- *Conductivity/Specific Conductivity*: Perform calibration according to procedures outlined in Attachment O-1, Steps 1 through 7.

- *ORP:* Perform calibration according to procedures outlined in Attachment O-1, Steps 1 through 7. If possible, plot values of millivolt versus temperature for the calibration standard on graph paper to aid in interpolation of temperature-corrected millivolt values. These values are usually found on the label of the calibration standard and may vary between solutions. Therefore, the values should be checked for each bottle of calibration solution utilized, and new interpolation graphs should be prepared if necessary.

- Turbidity: Perform calibration according to procedures outlined in Attachment O-1, Steps 1 through 5. If erratic readings are observed, clean detector according to manufacturer's instructions as an initial response.

B. Field Measurement

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

Step 1 - The pH and conductivity will be obtained using an appropriate temperature-compensating combination meter. If the combination meter does not have a temperature display, the temperature will be obtained using a thermometer. The DO will be obtained using a DO meter. The pH/specific conductivity/DO meters will be field calibrated in accordance with manufacturer's instructions during each day of use as specified above.

Step 2 - Obtain a small quantity of the water sample, place it in a clean glass container, agitate, and then discard. Refill the container. Rinse the pH, specific conductivity, DO, turbidity, and temperature probes with distilled water. Submerse probes into the container containing the water. Alternatively, the same readings may be obtained directly from the water source, with the probe submersed in the water body (e.g., river channel, lake) at the approximate mid-depth location. In either instance, maintain the probe in the submersed position for approximately 1 minute, allowing the selected parameter readings to stabilize, and then record the measurements on the appropriate forms.

Step 3 - Clean the probe and cable with a non-phosphate soap and water wash, followed by a distilled/deionized water rinse. Store the probe and cable in a clean container.

Attachment O-1

Calibration of Field Instruments

STANDARD OPERATING PROCEDURE
CALIBRATION OF FIELD INSTRUMENTS
(temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity)

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1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to provide a framework for calibrating field instruments used to measure water quality parameters for groundwater and surface water. Water quality parameters include temperature, pH, dissolved oxygen, specific conductance, oxidation/reduction potential [ORP], and turbidity. This SOP supplements, but does not replace, EPA analytical methods listed in 40 CFR 136 and 40 CFR 141 for temperature, dissolved oxygen, conductivity/specific conductance, pH and turbidity.

This SOP is written for instruments that measure temperature, pH, dissolved oxygen, specific conductance, turbidity, and/or oxidation/reduction potential [ORP] and the probe readings for pH, dissolved oxygen, and specific conductance are automatically corrected for temperature.

For groundwater monitoring, the instrument must be equipped with a flow-through-cell and the display/logger or computer display screen needs to be large enough to simultaneously contain the readouts of each probe in the instrument. Turbidity is measured using a separate instrument. It must not be measured in a flow-through-cell because the flow-through-cell acts as a sediment trap. This procedure is applicable for use with the *EPA Region 1 Low Stress (low flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells.*

2.0 HEALTH AND SAFETY WARNINGS

Read all labels on the standards and note any warnings on the labels. Wear appropriate personal protection equipment (e.g., gloves, eye shields, etc.) when handling the standards. If necessary, consult the Material Safety Data Sheets (MSDS) for additional safety information on the chemicals in the standards.

3.0 GENERAL

All monitoring instruments must be calibrated before they are used to measure environmental samples. For instrument probes that rely on the temperature sensor (pH, dissolved oxygen, specific conductance, and oxidation/reduction potential [ORP]), each temperature sensor needs to be checked for accuracy against a thermometer that is traceable to the National Institute of Standards and Technology (NIST). Before any instrument is calibrated or used to perform environmental measurements, the instrument must stabilize (warm-up) according to manufacturer's instructions and must have no air bubbles lodged between the probe and probe guard.

Most projects will require at least two standards to bracket the expected measurement range. This means that one standard is less than the expected value and one is higher. When an environmental sample measurement falls outside the calibration range, the instrument must be re-calibrated to bracket the new range before continuing measurements. Otherwise, the measurements that are outside the calibration range will need to be qualified.

This SOP requires that the manufacturer's instruction manual (including the instrument specifications) accompany the instrument into the field.

4.0 FREQUENCY OF CALIBRATION

At a minimum, the instrument is calibrated prior to use on the day the measurements are to be performed. A post calibration check at the end of the day is performed to determine if the instrument drifted out of calibration. Some projects may require more frequent calibration checks throughout the day in addition to the check at the end of the day. For these checks, the instrument can be recalibrated during the day if the instrument drifted out of calibration and only the data measured prior to the check would need to be qualified. The calibration/post calibration data information is recorded in Table 1.

Instruments (e.g., sonde) that monitor continuously over a period of time are calibrated before deployment. When these instruments are recovered, the calibration is checked to determine if any of them drifted out of calibration.

Some instruments lose their calibration criteria when they are turned off. Those instruments can either be left on all day (battery dependent) or calibrated at each sampling location. If they are calibrated at each sampling location, a post calibration check is not needed.

Ideally, the temperature of the standards should be close to the temperature of the ambient water that is being measured.

5.0 CALIBRATION PROCEDURES

Prior to calibration, all instrument probes and cable connections must be cleaned and the battery checked according to the manufacturer's instructions. Failure to perform these steps (proper maintenance) can lead to erratic measurements.

If a multi-probe instrument is to be used, program the instrument to display the parameters to be measured (e.g., temperature, pH, percent dissolved oxygen, mg/L dissolved oxygen, specific conductance, and ORP).

The volume of the calibration solutions must be sufficient to cover both the probe and temperature sensor (see manufacturer's instructions for the volume to be used).

Check the expiration date of the standards. Do not use expired standards.

All standards are stored according to manufacturer instructions.

5.1 TEMPERATURE

Most instrument manuals state there is no calibration of the temperature sensor, but the temperature sensor must be checked to determine its accuracy. This accuracy check is performed at least once per year and the accuracy check date/information is kept with the instrument. If the accuracy check date/information is not included with the instrument or the last check was over a year, the temperature sensor accuracy needs to be checked at the beginning of the sampling event. If the instrument contains multiple temperature sensors, each sensor must be checked. This procedure is not normally perform in the field. If the instrument is obtained from a rental company, the rental company should performed the calibration check and include with the instrument documentation that it was performed.

Verification Procedure

1. Fill a container with water and adjust the water temperature to below the water body's temperature to be measured. Use ice or warm water to adjust the temperature.

2. Place a thermometer that is traceable to the National Institute of Standards and Technology (NIST) and the instrument's temperature sensor into the water. Wait for both temperature readings to stabilize.

3. Compare the two measurements. The instrument's temperature sensor must agree with the reference thermometer measurement within the accuracy of the sensor (e.g., $\pm 0.2\,^{\circ}C$). If the measurements do not agree, the instrument may not be working properly and the manufacturer needs to be consulted.

4. Adjust the water temperature to a temperature higher than the water body to be measured.

5. Compare the two measurements. The instrument's temperature sensor must agree with the reference thermometer measurement within the accuracy of the sensor (e.g.,

±0.2°C). If the measurements do not agree, the instrument may not be working properly and the manufacturer needs to be consulted.

5.2 pH (electrometric)

The pH of a sample is determined electrometrically using a glass electrode.

Choose the appropriate buffered standards that will bracket the expected values at the sampling locations. If the water body's pH is unknown, then three standards are needed for the calibration: one close to seven, one at least two pH units below seven, and the other at least two pH units above seven. Instruments that will not accept three standards will need to be re-calibrated if the water sample's pH is outside the initial calibration range described by the two standards.

<u>Calibration Procedure</u>

1. Allow the buffered standards to equilibrate to the ambient temperature.

2. Fill calibration containers with the buffered standards so each standard will cover the pH probe and temperature sensor.

3. Remove probe from its storage container, rinse with deionized water, and remove excess water.

4. Select measurement mode. Immerse probe into the initial standard (e.g., pH 7).

5. Wait until the readings stabilize. If the reading does not change within 30 seconds, select calibration mode and then select "pH". Enter the buffered standard value into instrument.

6. Remove probe from the initial standard, rinse with deionized water, and remove excess water.

7. Immerse probe into the second standard (e.g., pH 4). Repeat step 5.

8. Remove probe from the second standard, rinse with deionized water, and remove excess water. If instrument only accepts two standards, the calibration is complete. Go to step 11. Otherwise continue.

9. Immerse probe in third buffered standard (e.g., pH 10) and repeat step 5.

10. Remove probe from the third standard, rinse with deionized water, and remove excess water.

11. Select measurement mode, if not already selected. To ensure that the initial calibration standard (e.g., pH 7) has not changed, immerse the probe into the initial standard. Wait for the readings to stabilize. The reading should read the initial standard value within the manufacturer's specifications. If not, re-calibrate the instrument. If re-calibration does not help, the calibration range may be too great. Reduce calibration range by using standards that are closer together.

12. The calibration is complete. Rinse the probe with deionized water and store the probe according to manufacturer's instructions.

13. Record the calibration information on Table 1.

5.3 DISSOLVED OXYGEN

Dissolved oxygen (DO) content in water is measured using a membrane electrode. To insure proper operation, the DO probe's membrane and electrolyte should be replaced prior to calibration for the sampling event. The new membrane may need to be conditioned before it is used; consult manufacturer's manual on how the conditioning is to be performed. Failure to perform this step may lead to erratic measurements. Before performing the calibration/measurements, inspect the membrane for air bubbles and nicks.

Note: some manufacturers require an altitude correction instead of a barometric correction. In that case, enter the altitude correction according to the manufacturer's directions in Step 5 and then proceed to Step 6.

Note: some instruments have a built-in barometer. Follow the manufacturer's instructions for entering the barometric value in step 5.

<u>Calibration Procedure</u>

1. Gently dry the temperature sensor and remove any water droplets from the DO probe's sensor membrane according to manufacturer's instructions. Note that the evaporation of moisture on the temperature sensor or DO probe may influence the readings during calibration.

2. Create a 100 percent water-saturated air environment by placing a wet sponge or a

wet paper towel on the bottom of the DO calibration container. Place the DO probe into the calibration container. The probe is loosely fitted into the calibration container to prevent the escape of moisture evaporating from the sponge or paper towel while maintaining ambient pressure (see manufacturer's instructions). Note that the probe and the temperature sensor must not come in contact with these wet items.

3. Allow the confined air to become saturated with water vapor (saturation occurs in approximately 10 to 15 minutes). During this time, turn on the instrument to allow the DO probe to warm-up. Select the measurement mode. Check the temperature readings. Readings must stabilize before continuing to the next step.

4. Select calibration mode; then select "DO %".

5. Enter the local barometric pressure (usually in mm of mercury) for the sampling location into the instrument. This measurement must be determined from an on-site barometer. Do not use barometric pressure obtained from the local weather services unless the pressure is corrected for the elevation of the sampling location. [Note: inches of mercury times 25.4 mm/inch equals mm of mercury or consult Oxygen Solubility at Indicated Pressure chart attached to the SOP for conversion at selected pressures].

6. The instrument should indicate that the calibration is in progress. After calibration, the instrument should display percent saturated DO.

7. Select measurement mode and set the display to read DO mg/L and temperature. Compare the DO mg/L reading to the Oxygen Solubility at Indicated Pressure chart attached to the SOP. The numbers should agree. If they do not agree within the accuracy of the instrument (usually ± 0.2 mg/L), repeat calibration. If this does not work, change the membrane and electrolyte solution.

8. Remove the probe from the container and place it into a 0.0 mg/L DO solution (see footnote). Check temperature readings. They must stabilize before continuing.

9. Wait until the "mg/L DO" readings have stabilized. The instrument should read less than 0.5 mg/L (assuming an accuracy of ± 0.2 mg/L). If the instrument reads above 0.5 mg/L or reads negative, it will be necessary to clean the probe, and change the membrane and electrolyte solution. If this does not work, try a new 0.0 mg/L DO solution. If these changes do not work, contact the manufacturer. Note: some projects and instruments may have different accuracy requirements. The 0.5 mg/L

value may need to be adjusted based on the accuracy requirements of the project or instrument.

10. After the calibration has been completed, rinse the probe with tap or deionized water and store the probe according to manufacturer's instructions. It is important that all of the 0.0 mg/L DO solution be rinsed off the probe so as not to effect the measurement of environmental samples.

11. Record calibration information on Table 1.

Note: You can either purchase the 0.0 mg/L DO solution from a vendor or prepare the solution yourself. To prepare a 0.0 mg/L DO solution, follow the procedure stated in Standard Methods (Method 4500-O G). The method basically states to add excess sodium sulfite (until no more dissolves) and a trace amount of cobalt chloride (read warning on the label before use) to water. This solution is prepared prior to the sampling event. Note: this solution can be made without cobalt chloride, but the probe will take longer to respond to the low DO concentration.

5.4 SPECIFIC CONDUCTANCE

Conductivity is used to measure the ability of an aqueous solution to carry an electrical current. Specific conductance is the conductivity value corrected to 25°C.

Most instruments are calibrated against a single standard which is near the specific conductance of the environmental samples. The standard can be either below or above the specific conductance of the environmental samples. A second standard is used to check the linearity of the instrument in the range of measurements.

When performing specific conductance measurement on groundwater or surface water and the measurement is outside the initial calibration range defined by the two standards, the instrument will need to be re-calibrated using the appropriate standards.

Specific Conductance Calibration Procedure

1. Allow the calibration standards to equilibrate to the ambient temperature.

2. Fill calibration containers with the standards so each standard will cover the probe and temperature sensor. Remove probe from its storage container, rinse the probe with deionized water or a small amount of the standard (discard the rinsate), and place the

probe into the standard.

3. Select measurement mode. Wait until the probe temperature has stabilized.

4. Select calibration mode, then specific conductance. Enter the specific conductance standard value. Make sure that the units on the standard are the same as the units used by the instrument. If not, convert the units on the standard to the units used by the instrument.

5. Select measurement mode. The reading should remain within manufacturer's specifications. If it does not, re-calibrate. If readings continue to change after re-calibration, consult manufacturer or replace calibration solution.

6. Remove probe from the standard, rinse the probe with deionized water or a small amount of the second standard (discard the rinsate), and place the probe into the second standard. The second standard will serve to verify the linearity of the instrument. Read the specific conductance value from the instrument and compare the value to the specific conductance on the standard. The two values should agree within the specifications of the instrument. If they do not agree, re-calibrate. If readings do not compare, then the second standard may be outside the linear range of the instrument. Use a standard that is closer to the first standard and repeat the verification. If values still do not compare, try cleaning the probe or consult the manufacturer.

7. After the calibration has been completed, rinse the probe with deionized water and store the probe according to manufacturer's instructions.

8. Record the calibration information on Table 1.

Note: for projects where specific conductance is not a critical measurement it may be possible to calibrate with one standard in the range of the expected measurement.

5.5 OXIDATION/REDUCTION POTENTIAL (ORP)

The oxidation/reduction potential is the electrometric difference measured in a solution between an inert indicator electrode and a suitable reference electrode. The electrometric difference is measured in millivolts and is temperature dependent.

Calibration or Verification Procedure

1. Allow the calibration standard (a Zobell solution: read the warning on the label before use) to equilibrate to ambient temperature.

2. Remove the probe from its storage container and place it into the standard.

3. Select measurement mode.

4. Wait for the probe temperature to stabilize, and then read the temperature.

5. If the instrument is to be calibrated, do Steps 6 and 7. If the instrument calibration is to be verified, then go to Step 8.

6. Look up the millivolt (mv) value at this temperature from the millivolt versus temperature correction table usually found on the standard bottle or on the standard instruction sheet. You may need to interpolate millivolt value between temperatures. Select "calibration mode", then "ORP". Enter the temperature-corrected ORP value into the instrument.

7. Select measurement mode. The readings should remain unchanged within manufacturer's specifications. If they change, re-calibrate. If readings continue to change after re-calibration, try a new Zobell solution or consult manufacturer. Go to Step 9.

8. If the instrument instruction manual states that the instrument is factory calibrated, then verify the factory calibration against the Zobell solution. If they do not agree within the specifications of the instrument, try a new Zobell solution. If it does not agree, the instrument will need to be re-calibrated by the manufacturer.

9. After the calibration has been completed, rinse the probe with deionized water and store the probe according to manufacturer's instructions.

10. Record the calibration information on Table 1.

5.6 TURBIDITY

The turbidity method is based upon a comparison of intensity of light scattered by a sample under defined conditions with the intensity of light scattered by a standard reference suspension. A

turbidimeter is a nephelometer with a visible light source for illuminating the sample and one or more photo-electric detectors placed ninety degrees to the path of the light source. Note: the below calibration procedure is for a turbidimeter which the sample is placed into a cuvette.

Some instruments will only accept one standard. For those instruments, the second, third, etc., standards will serve as check points.

Calibration Procedures

1. Allow the calibration standards to equilibrate at the ambient temperature. The use of commercially available polymer primary standards (AMCO-AEPA-1) is preferred; however, the standards can be prepared using Formazin (read the warning on the label before use) according to the EPA analytical Method 180.1. Other standards may be used if they can be shown that they are equivalent to the previously mentioned standards.

2. If the standard cuvette is not sealed, rinse a cuvette with deionized water. Shake the cuvette to remove as much water as possible. Do not wipe dry the inside of the cuvette because lint from the wipe may remain in the cuvette. Add the standard to the cuvette.

3. Before performing the calibration procedure, make sure the cuvettes are not scratched and the outside surfaces are dry and free from fingerprints and dust. If the cuvette is scratched or dirty, discard or clean the cuvette respectively. Note: some manufacturers require the cuvette to be orientated in the instrument in a particular direction for accurate reading.

4. Select a low value standard such as a zero or 0.02 NTU and calibrate according to manufacturer's instructions. Note: a zero standard (approximately 0 NTU) can be prepared by passing distilled water through a 0.45 micron pore size membrane filter.

5. Select a high standard and calibrate according to manufacturer's instructions or verify the calibration if instrument will not accept a second standard. In verifying, the instrument should read the standard value to within the specifications of the instrument. If the instrument has range of scales, check each range that will be used during the sampling event with a standard that falls within that range.

6. Record the calibration information on Table 1.

6.0 POST CALIBRATION CHECK

After the initial calibration is performed, the instrument's calibration may drift during the measurement period. As a result, you need to determine the amount of drift that occurred after collecting the measurements. This is performed by placing the instrument in measurement mode (not calibration mode) and placing the probe in one or more of the standards used during the initial calibration; for turbidity place the standard in a cuvette and then into the turbidimeter. Wait for the instrument to stabilize and record the measurement (Table 1). Compare the measurement value to the initial calibration value. This difference in value is then compared to the drift criteria or post calibration criteria described in the quality assurance project plan or the sampling and analysis plan for the project. If the check value is outside the criteria, then the measurement data will need to be qualified.

For the dissolved oxygen calibration check, follow the calibration instructions steps one through three while the instrument is in measurement mode. Record dissolved oxygen value (mg/L), temperature, and barometric pressure. Compare the measurement value to the Oxygen Solubility at Indicated Pressure chart attached to this SOP. The value should be within the criteria specified for the project. If measurement value drifted outside the criteria, the data will need to be qualified.

If the quality assurance project plan or the sampling and analysis plan do not list the drift criteria or the post-calibration criteria, use the criteria below.

Measurement	**Post Calibration Criteria**
Dissolved Oxygen	± 0.5 mg/L of sat. value* < 0.5 mg/L for the 0 mg/L solution, but not a negative value
Specific Conductance	±5% of standard or ± 10 μS/cm (whichever is greater)
pH	± 0.3 pH unit with pH 7 buffer*
Turbidity	± 5% of standard
ORP	± 10 mv*

Note: * Table 8.1, USEPA Region 1 *YSI 6-Series Sondes and Data Logger SOP*, January 30, 2007, revision 9.

7.0 DATA MANAGEMENT AND RECORDS MANAGEMENT

All calibration records must be documented in the project's log book or on a calibration log sheet. At a minimum, include the instrument manufacturer, model number, instrument identification number (when more than one instrument of the same model is used), the standards used to calibrate the instruments (including source), the calibration date, the instrument readings, the post calibration check, and the name of the person(s) who performed the calibration. An example of a calibration log sheet is shown in Table 1.

8.0 References

Standard Methods for the Examination of Water and Wastewater, 20th edition, 1998.

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983.

Turbidity - Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993.

USEPA Region 1 YSI 6-Series Sondes and Data Logger SOP, January 30, 2007, revision 9.

USGS Guidelines and Standard Procedures for Continuous Water-Quality Monitors: Station Operation, Record Computation, and Data Reporting, Techniques and Methods 1-D3.

TABLE 1
INSTRUMENT CALIBRATION LOG

Project Name__________________ Date___________________
Weather____________

Calibrated by__________________ Instrument_____________
Serial Number___________

Parameters	Morning Calibration	Morning Temperature	End of Day Calibration Check*	End of Day Temperature
Specific Conductance Standard #1				
Specific Conductance Standard #2				
pH (7)				
pH (4)				
pH (10)				
ORP Zobel solution				
Dissolved Oxygen 100% water saturated air mg/L				
Dissolved Oxygen Zero Dissolved Oxygen Solution mg/L				
Barometric Pressure mm Hg		NA		NA
Turbidity Standard #1				
Turbidity Standard #2				
Turbidity Standard #3				

* For each Parameter, chose one standard as your check standard. If possible, choose the one that is closest to the ambient measurement value.

Oxygen Solubility at Indicated Pressure

Temp.	Pressure (Hg)							
	760	755	750	745	740	735	730	mm
°C	29.92	29.72	29.53	29.33	29.13	28.94	28.74	in
0	14.57	14.47	14.38	14.28	14.18	14.09	13.99	mg/l
1	14.17	14.08	13.98	13.89	13.79	13.70	13.61	
2	13.79	13.70	13.61	13.52	13.42	13.33	13.24	
3	13.43	13.34	13.25	13.16	13.07	12-98	12.90	
4	13.08	12.99	12.91	12.82	12.73	12.65	12.56	
5	12.74	12.66	12.57	12.49	12.40	12.32	12.23	
6	12.42	12.34	12.26	12.17	12.09	12.01	11.93	
7	12.11	12.03	11.95	11.87	11.79	11.71	11.63	
8	11.81	11.73	11.65	11.57	11.50	11.42	11.34	
9	11.53	11.45	11.38	11.30	11.22	11.15	11.07	
10	11.28	11.19	11.11	11.04	10.96	10.89	10.81	
11	10.99	10.92	10.84	10.77	10.70	10.62	10.55	
12	10.74	10.67	10.60	10.53	10.45	10.38	10.31	
13	10.50	10.43	10.36	10.29	10.22	10.15	10.08	
14	10.27	10.20	10.13	10.06	10.00	9.93	9.86	
15	10.05	9.98	9.92	9.85	9.78	9.71	9.65	
16	9.83	9.76	9.70	9.63	9.57	9.50	9.43	
17	9.63	9.57	9.50	9.44	9.37	9.31	9.24	
18	9.43	9.37	9.30	9.24	9.18	9.11	9.05	
19	9.24	9.18	9.12	9.05	8.99	8.93	8.87	
20	9.06	9.00	8.94	8.88	8.82	8.75	8.69	
21	8.88	8.82	8.76	8.70	8.64	8.58	8.52	
22	8.71	8.65	8.59	8.53	8.47	8.42	8.36	
23	8.55	8.49	8.43	8.38	8.32	8.26	8.20	
24	8.39	8.33	8.28	8.22	8.16	8.11	8.05	
25	8.24	8.18	8.13	8.07	8.02	7.96	7.90	
26	8.09	8.03	7.98	7.92	7.87	7.81	7.76	
27	7.95	7.90	7.84	7.79	7.73	7.68	7.62	
28	7.81	7.76	7.70	7.65	7.60	7.54	7.49	
29	7.68	7.63	7.57	7.52	7.47	7.42	7.36	
30	7.55	7.50	7.45	7.39	7.34	7.29	7.24	
31	7.42	7.37	7.32	7.27	7.22	7.16	7.11	
32	7.30	7.25	7.20	7.15	7.10	7.05	7.00	
33	7.08	7.13	7.08	7.03	6.98	6.93	6.88	
34	7.07	7.02	6.97	6.92	6.87	6.82	6.78	
35	6.95	6.90	6.85	6.80	6.76	6.71	6.66	
36	6.84	6.79	6.76	6.70	6.65	6.60	6.55	
37	6.73	6.68	6.64	6.59	6.54	6.49	6.45	
38	6.63	6.58	6.54	6.49	6.44	6.40	6.35	
39	6.52	6.47	6.43	6.38	6.35	6.29	6.24	
40	6.42	6.37	6.33	6.28	6.24	6.19	6.15	
41	6.32	6.27	6.23	6.18	6.14	6.09	6.05	
42	6.22	6.18	6.13	6.09	6.04	6.00	5.95	
43	6.13	6.09	6.04	6.00	5.95	5.91	5.87	
44	6.03	5.99	5.94	5.90	5.86	5.81	5.77	
45	5.94	5.90	5.85	5.81	5.77	5.72	5.68	

(Continued)

Source: Draft EPA Handbook of Methods for Acid Deposition Studies, Field Operations for Surface Water Chemistry, EPA/600/4-89/020, August 1989.

Oxygen Solubility at Indicated Pressure (continued)

Temp.	Pressure (Hg)							
	725	720	715	710	705	700	695	690 mm
°C	28.54	28.35	28.15	27.95	27.76	27.56	27.36	27.17 in
0	13.89	13.80	13.70	13.61	13.51	13.41	13.32	13.22 mg/l
1	13.51	13.42	13.33	13.23	13.14	13.04	12.95	12.86
2	13.15	13.06	12.07	12.88	12.79	12.69	12.60	12.51
3	12.81	12.72	12.63	12.54	12.45	12.36	12.27	12.18
4	12.47	12.39	12.30	12.21	12.13	12.04	11.95	11.87
5	12.15	12.06	11.98	11.89	11.81	11.73	11.64	11.56
6	11.84	11.73	11.68	11.60	11.51	11.43	11.35	11.27
7	11.55	11.47	11.39	11.31	11.22	11.14	11.06	10.98
8	11.26	11.18	11.10	11.02	10.95	10.87	10.79	10.71
9	10.99	10.92	10.84	10.76	10.69	10.61	10.53	10.46
10	10.74	10.66	10.59	10.51	10.44	10.36	10.29	10.21
11	10.48	10.40	10.33	10.28	10.18	10.11	10.04	9.96
12	10.24	10.17	10.10	10.02	9.95	9.88	9.81	9.46
13	10.01	9.94	9.87	9.80	9.73	9.66	9.59	9.52
14	9.79	9.72	9.65	9.68	9.51	9.45	9.38	9.31
15	9.58	9.51	9.44	9.58	9.31	9.24	9.18	9.11
16	9.37	9.30	9.24	9.17	9.11	9.04	8.97	8.91
17	9.18	9.11	9.05	8.98	8.92	8.85	8.79	8.73
18	8.99	8.92	8.86	8.80	8.73	8.67	8.61	8.54
19	8.81	8.74	8.68	8.62	8.56	8.49	8.43	8.37
20	8.63	8.57	8.51	8.45	8.39	8.33	8.27	8.21
21	8.46	8.40	8.34	8.28	8.22	8.16	8.10	8.04
22	8.30	8.24	8.18	8.12	8.06	8.00	7.95	7.89
23	8.15	8.09	8.03	7.97	7.91	7.86	7.80	7.74
24	7.99	7.94	7.88	7.82	7.76	7.71	7.65	7.59
25	7.85	7.79	7.74	7.68	7.60	7.57	7.51	7.46
26	7.70	7.65	7.59	7.54	7.48	7.43	7.37	7.32
27	7.57	7.52	7.46	7.41	7.35	7.30	7.25	7.19
28	7.44	7.38	7.33	7.28	7.22	7.17	7.12	7.06
29	7.31	7.26	7.21	7.15	7.10	7.05	7.00	6.94
30	7.19	7.14	7.08	7.03	6.98	6.93	6.88	6.82
31	7.06	7.01	6.96	6.91	6.86	6.81	6.76	6.70
32	6.95	6.90	6.85	6.80	6.70	6.70	6.64	6.59
33	6.83	6.78	6.73	6.68	6.83	6.58	6.53	6.48
34	6.73	6.68	6.63	6.58	6.53	6.48	6.43	6.38
35	6.61	6.56	6.51	6.47	6.42	6.37	6.36	6.27
36	6.51	6.46	6.41	6.36	6.31	6.27	6.22	6.17
37	6.40	6.35	6.31	6.26	6.21	6.16	6.12	6.07
38	6.30	6.26	6.21	6.16	6.12	6.07	6.02	5.98
39	6.26	6.15	6.11	6.06	6.01	5.97	5.92	5.87
40	6.10	6.06	6.01	5.96	5.92	5.86	5.83	5.78
41	6.00	5.96	5.91	5.87	5.82	5.78	5.73	5.69
42	5.91	5.86	5.82	5.77	5.73	5.69	5.64	5.60
43	5.82	5.78	5.73	5.69	5.65	5.60	5.56	5.51
44	5.72	5.68	5.64	5.59	5.55	5.51	5.46	5.42
45	5.64	5.59	5.55	5.51	5.47	5.42	5.38	5.34

Source: Draft EPA Handbook of Methods for Acid Deposition Studies, Field Operations for Surface Water Chemistry, EPA/600/4-89/020, August 1989.

Appendix P

In-situ Hydraulic Conductivity Test Procedures

In-situ Hydraulic Conductivity Test Procedures

I. Introduction

In-situ hydraulic conductivity tests may be conducted at selected wells. The tests can be used to evaluate the integrity of the well screen of cased wells and for all types of well construction to determine the responsiveness of the well to change in static water levels; however, more importantly, estimates of the hydraulic conductivities can be calculated. In-situ hydraulic conductivity testing can be performed by the falling head test by placing a slug into the well or by the rising head test by withdrawing a known volume of water with a bailer. The following procedures are consistent for both tests, with the exception that the falling head test uses a slug and the rising head test uses a bailer.

II. Materials

- Photoionization detector (PID) to measure headspace vapors;
- Stopwatch;
- Polypropylene rope;
- Bailer or slug;
- Appropriate field logs/forms;
- Water level probe;
- Masking tape;
- Engineer=s rule;
- Waterproof marker;
- Appropriate cleaning materials (as required in Appendix W);
- Field notebook; and
- Health and safety equipment (as required by the Health and Safety Plan).

III. Procedures

Step 1 - Identify site and well number on the In-Situ Hydraulic Conductivity Test Log (Attachment P-1) and/or field notebook along with other appropriate information collected during the in-situ hydraulic conductivity test.

Step 2 - Open the well cover while standing upwind of the well; Remove well cap. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in the field log. If the well headspace reading is less than 5 PID units, proceed, else screen the air within the breathing zone. If the PID reading is above 5 PID units, move upwind from the well for five minutes to allow the volatiles to dissipate. Repeat the breathing zone test. If the reading is still above 5 PID units, put on appropriate respiratory protection in accordance with the requirements of the Health and Safety Plan. Record all PID readings. For wells which are part of the regular weekly monitoring program and prior PID measurements have not resulted in a breathing zone reading above 5 PID units, PID measurements will be taken monthly.

Step 3 - While the water level recording probe is still at static water level, place masking tape on the water level recorder cable from reference point to 5 feet above the reference point.

Step 4 - Using a waterproof pen, mark the masking tape where static water level is reached from the reference point. Label the mark AS@ for static water level.

Step 5 - Remove the cable and probe from the well and place it in the plastic sheeting.

Step 6 - Measure out a length of rope 10 feet greater than the depth to static water level.

Step 7 - Clean the slug or bailer and the rope according to the cleaning protocol (Appendix W) and place on a plastic sheet near the well.

Step 8 - Secure one end of the rope to the slug or bailer and the other end to the well casing using a bowline knot.

Step 9 - Assign one person responsible for lowering the slug or bailer into the well and recording times in the log. Assign another person responsible for lowering the water level probe into the well and finding water levels.

Step 10 - Slowly lower the slug or bailer into the well unit until it is just below the water level. Set the water level probe into the well to monitor the water level until it returns to initial conditions.

Step 11 - Set stop watch.

Step 12*- When both people are ready, remove the slug or bailer from the water and start the stop watch at the same time.

Step 13 - Measure water level at approximately 5-second intervals. Where the water level is found, mark the tape at the reference point and record the time.

Step 14 - After 3 minutes, measure water levels at approximate 15-second intervals for 5 minutes and then at 1-minute intervals for 10 minutes. When readings start to stabilize, they may be taken at longer time increments until the water level reaches static level.

Step 15 - When test is completed, changes in water levels will be measured to the nearest hundredth from the masking tape and recorded with its corresponding change in time reading.

Step 16 - Remove the masking tape from the water level probe cable and clean the probe as specified in Appendix W.

*Steps 12-14 may be modified if a pressure transducer and automatic data logger are utilized.

Attachment P-1

In-situ Hydraulic Conductivity Test

IN SITU HYDRAULIC CONDUCTIVITY TEST

DATE STARTED ____________________
DATE FINISHED ____________________
SHEET ____ OF ____
PROJECT ____________________
JOB NO. ____________________
LOCATION ____________________

OBSERVATION WELL NO. ____________________
DATUM ____________________
FT. ABOVE GROUND LEVEL ____________________
TEST NO. ____________________
STATIC WATER LEVEL BEFORE TEST ____________________

CLOCK TIME	WATER LEVEL READINGS	△	CLOCK TIME	WATER LEVEL READINGS	△	CLOCK TIME	WATER LEVEL READINGS	△

Appendix Q

Water Level/Oil Thickness
Measurement Procedures

Water Level/Oil Thickness Measurement Procedures

I. Introduction

Monitoring well water levels and oil thicknesses will be determined, as appropriate, to develop piezometric maps and to monitor plume migration. The water levels and oil thickness will be obtained using an Oil/Water Interface Probe. The operating and maintenance instruction manual for the probe should be reviewed prior to commencement of work to assure safe and accurate operation. Standard procedures for determining water levels and oil thicknesses in monitoring wells are presented in this Appendix.

II. Materials

- Photoionization detector (PID) to measure headspace vapors;
- Health and safety equipment (as required by the Health and Safety Plan);
- Cleaning equipment (as required in Appendix W);
- Oil/Water interface probe and instruction manual;
- Plastic sheeting;
- Measuring tape;
- Watch (record time and day);
- Field notebook;
- Absorbent pads;
- Appropriate log forms; and
- Monitoring well keys.

III. Procedures

Step 1 - Identify site and well number on Water Level/Oil Thickness Monitoring Field Log (Attachment Q-1) and/or field notebook along with other appropriate information collected during water level measurement.

Step 2 - Don personal protective equipment (as required by the Health and Safety Plan).

Step 3 - Clean the oil/water interface probe and cable in accordance with the cleaning procedures in Appendix W.

Step 4 - Place a piece of plastic sheeting adjacent to the well to use as a clean work area. Cut a hole in the center of sheeting and place the sheet around the well.

Step 5 - If oil is present in the well, place absorbent pads on plastic sheet beside the well to absorb oil which may be present when the oil/water interface probe is removed from the well.

Step 6 - Unlock and open the well cover while standing upwind of the well. Remove well cap. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in the field log. If the well headspace reading is less than 5 PID units, proceed; if the well headspace reading is greater than 5 ppm, screen the air within the breathing zone. If the PID reading in the breathing zone is below 5 PID units, proceed. If the PID reading is above 5 PID units, move upwind from the well for five minutes to allow the volatiles to dissipate. Repeat the breathing zone test. If the reading is still above 5 PID units, don appropriate respiratory protection in accordance with the requirements of the Health and Safety Plan. Record all PID readings. For wells which are part of the regular weekly monitoring program and prior PID measurements have not resulted in a breathing zone reading above 5 PID units, PID measurements will be taken monthly.

Step 7 - Locate a measuring reference point on the well casing. If one is not found, initiate a reference point by notching the inner and outer casings with a hacksaw or by using a waterproof marker. All down-hole measurements will be taken from the reference points. The acronym TIC will designate the top of inner casing and the acronym TOC will designate the top of the outer casing. If a well has both inner and outer casings, use the top of the inner casing as the reference point.

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

Step 8 - Measure to the nearest hundredth of a foot and record the height of the inner and outer casing from reference point to ground level.

Step 9 - Record the inside diameter of the well casing on the field log.

Step 10 - Lower the oil/water interface probe into the well to determine the existence of any light immiscible layer. Carefully record the depths of the air/light phase and light phase/water interfaces (to the nearest 0.01 feet) to determine the thickness of the light phase immiscible layer (if present). If no light phase immiscible layer is present, record the depth of the air/water interface.

Step 11 - For wells in which DNAPL is to be monitored, lower the oil/water interface probe to the bottom of the well and carefully record the dense phase/water interface (if present) and the depth at which the bottom of the well is encountered. The probe will emit a different reading (whether audible or visual) to discern between oil and water interfaces. Record all interface and well depth measurements in the field book to the nearest 0.01 feet. The well depth will be determined to evaluate any silt accumulation or blockage in the well.

Step 12 - Remove cable or tape and probe from the well.

Step 13 - Between wells, when obtaining water level/oil thickness measurements at more than one location, clean the instrument with a non-phosphate soap and water wash followed by a distilled/deionized water rinse. Use an appropriate-solvent rinse, if necessary, to remove oil deposits.

Step 14 - Close the well when all activities are completed.

Step 15 - Collect all PPE and other wastes generated for disposal (see Section IV below).

Step 16 - Certain activities (i.e., monitoring programs at Lyman Street Area and East Street Area 2-South) require staff gauge readings of the Housatonic River during monitoring events. If required, obtain the reading to the nearest 0.01 feet for the Housatonic River staff gauges adjacent to the Lyman Street parking lot and/or Building 64-X Oil/Water Separator in East Street Area 2-South.

IV. Disposal Methods

Materials generated during water level/oil thickness measurement procedures, including disposable equipment, will be disposed of in appropriate containers. Containerized waste will be disposed of by GE consistent with its on-going disposal practices.

Attachment Q-1

Water Level/Oil Thickness Monitoring Field Log

Attachment Q-1

WATER LEVEL/OIL THICKNESS
MONITORING FIELD LOG
WELL No.

Date: ______________________________ Time: ______________________________

Project: ____
Project No.: ____

Weather Conditions: ____
Temerature: ____
Field Personnel: ____

Photoionization Detector Readings: Within Well - _ _ _ _ _ _ _ _ _ _ ppm
Breathing Zone (initial) - _ _ _ _ _ _ _ _ ppm

I. Well Information

	Inner Casing	Outer Casing
Ground to Top of Casing Reference Point		
Inside Diameter of Casing		

II. Phase Thickness Information

	Feet
Reference Mark to Top of LNAPL	
Reference Mark to Water	
Reference Mark to DNAPL (if applicable)	
Bottom of Well (if applicable)	

Appendix R

NAPL Recovery Procedures

NAPL Recovery Procedures

I. Introduction

Non-aqueous phase liquid (NAPL) encountered while performing water level/NAPL thickness measurement procedures will be manually recovered when quantities exceed amounts determined by General Electric (GE) and approved by the United States Environmental Protection Agency (USEPA).

GE has developed site-wide criteria for NAPL monitoring and manual recovery requirements, standard procedures for assessment of new NAPL occurrences, and the feasibility of the installation of new recovery systems.

II. Materials

The following equipment and materials will be available, as required, during manual NAPL recovery efforts:

- Photoionization detector (PID);
- Health and safety equipment (as required by the Health and Safety Plan [HASP]);
- Cleaning equipment (as required in Appendix W);
- Plastic sheeting;
- Field notebook or appropriate log forms;
- Absorbent pads;
- Peristaltic pump and tubing;
- Bailer;
- Non-absorbent cord (polypropylene);
- Graduated cylinder; and
- Container for recovered oil.

III. Procedures

1. Notify GE representatives that manual NAPL recovery activities will be initiated so GE can initiate manifesting procedures, if necessary.

2. Perform water level/NAPL thickness measurement procedures in accordance with Appendix Q.

3. If LNAPL is present in the well and exceeds action level quantity (see Section V below), or is observed for the first time in an area (see Section VI below), it must be recovered for disposal by GE.

4. Remove LNAPL utilizing a bailer or peristaltic pump, transfer material into a container supplied by GE, and record volume.

5. If DNAPL is present in the well and exceeds action level quantity (see Section V below), or is observed for the first time in an area (see Section VI below), it must be removed for disposal by GE.

6. Remove DNAPL utilizing a peristaltic pump. Cut a section of tubing long enough to reach the bottom of the well. Leave excess tubing to allow connection to pump. Lower the tubing to the bottom of the well. Pump DNAPL directly into container supplied by GE to avoid spillage of material. Estimate the volume of DNAPL removed utilizing the diameter of the well and the measured thickness of the DNAPL layer. Record the estimated quantity removed onto the NAPL monitoring/recovery field log.

7. Do not transport liquid in vehicles along public roadways. Notify GE that LNAPL/DNAPL has been collected and is ready to be picked up for disposal.

8. Collect all personal protective equipment (PPE) and other wastes generated for disposal.

IV. Disposal Methods

Materials generated during the passive oil recovery procedures, including disposable equipment, will be disposed of in appropriate containers. Containerized waste will be disposed of by GE consistent with its on-going disposal practices.

V. Manual NAPL Removal Criteria

During routine NAPL monitoring/removal activities at select GE monitoring wells, LNAPL accumulations observed in excess of 0.25 feet are manually removed at the time of monitoring. For DNAPL, accumulations in excess of 0.5 feet are manually removed. Exceptions to these criteria are in place for certain wells that are located either upgradient of sensitive receptors (i.e., any measurable quantities of NAPL are manually removed) or within the capture zone of automated recovery systems (i.e., no NAPL is manually removed).

These manual removal criteria apply only during routine NAPL monitoring program events (i.e., weekly, monthly, and quarterly). No NAPL removal is required at wells monitored for other reasons between routine monitoring events (e.g., during NAPL recovery testing, well inventory inspections, or other data-gathering activities) or in connection with GE's semi-annual NAPL monitoring round during the spring and fall quarterly monitoring events (due to the performance of a bailing round, as discussed below).

Approximately 1 to 2 weeks prior to the spring and fall semi-annual monitoring events, all wells where the presence of NAPL was observed since the prior semi-annual event are monitored and measurable thicknesses of NAPL are manually removed (i.e., the bailing round). After allowing time for NAPL to return, if present, those wells are monitored again as part of the semi-annual monitoring event and the data obtained are utilized to estimate the current thickness of LNAPL in the area. Due to the large number of wells included in the semi-annual monitoring program, and the desire to collect the groundwater elevation data as quickly as possible to provide a more accurate account of flow conditions, no manual removal of NAPL from monitoring wells is required during the actual semi-annual data collection event (i.e., the monitoring round). The purpose for performing the bailing and monitoring rounds is to confirm that the NAPL present in a well is representative of the surrounding formation and does not reflect remnant oil that may have accumulated in the well since the last manual removal. This uniform removal procedure also provides a consistent basis for comparison of data with future NAPL monitoring data.

If a measurable thickness of NAPL is observed during the spring or fall semi-annual monitoring event in a well that was not addressed during the bailing round, the NAPL should be manually removed and the well should be monitored during the following week to gauge the NAPL thickness. The information obtained during that supplemental monitoring round is utilized in GE's assessment of the seasonal extent of NAPL.

VI. Assessment of New NAPL Observations

This section describes the process to investigate new or anomalous NAPL observations. Such observations may include either instrument detection of NAPL at a new location or detection of a type of NAPL not typically associated with a particular well (e.g., if DNAPL was observed in a monitoring well where LNAPL is typically observed). This process generally includes the following steps:

1. Confirm that NAPL is actually present at the well by bailing or pumping the well to verify that an instrument error did not occur. Additionally, the NAPL will be physically observed in a jar to visually assess its relative density compared to water.

2. Immediately notify the GE Project Manager of the new NAPL occurrence. The GE Project Manager will then make any required federal or state Agency notifications, as appropriate.

3. Initially, the monitoring frequency at the well will be at least once per week for a period of at least one month, and any observed NAPL will be removed. If additional wells are located in the vicinity and screened at the appropriate interval, they will also be monitored for NAPL presence.

4. Based on the results of Steps 1 and 2 above, GE may recommend that: a) the well be further evaluated for the potential installation of an automated recovery system based on the criteria in Section VII below; b) additional soil borings/monitoring wells be installed in the vicinity; or c) enhanced NAPL monitoring/ recovery activities be implemented.

After completion of these initial assessment activities, monitoring and manual NAPL recovery (if NAPL thicknesses exceed the standard manual removal criteria) activities will revert to their normal intervals, pending Agency approval of any recommendation made by GE.

VII. Criteria for Installation of Automated Recovery Systems

To aid in the assessment of whether additional automated recovery systems are necessary and feasible at a given location where NAPL is present, several key factors should be considered, specifically:

- The presence of other nearby active NAPL recovery systems;
- Quantity of NAPL available (on a continuing basis) to be recovered;
- Migration potential of the NAPL (considering historical monitoring data and capture areas of existing recovery systems); and
- Technical feasibility of installing an automated recovery system.

Each of these factors is discussed in more detail below.

If there are already active NAPL recovery systems operating nearby, an assessment must be made as to whether the NAPL area in question will be addressed by the existing system. Additional automated recovery systems are not required for NAPL areas that are within the capture zone of an operating active recovery system or positioned upgradient of it, such that the NAPL will ultimately be addressed by the existing recovery system.

Next, it must be confirmed whether sufficient quantities of NAPL are moving into a well to justify the potential installation of a recovery system. This determination is made through the performance of a NAPL recovery test conducted over a 2- to 3-day period. NAPL will be manually removed from the well, initially on an hourly basis, and the amount of NAPL returning to the well at each removal interval will be measured and recorded. Depending on the recovery rate, the time intervals of manual removal may be increased or decreased from the initial hourly interval. If the average NAPL quantity that returns to the well over the duration of the test is significant (e.g., greater than 0.5 liter per hour, or greater than 6 to 12 inches per hour in a 2-inch well), the location may be deemed a potential candidate for an automated recovery system based on NAPL quantity. NAPL samples may also be collected during this test and analyzed for chemical and/or physical parameters if such data do not already exist for the NAPL area in question. Physical testing will include specific gravity and viscosity. If warranted, interfacial tension may also be measured.

Following a determination that sufficient NAPL is potentially present, a more detailed analysis is necessary to confirm whether operation of an automated recovery system is appropriate to address the NAPL occurrence and to obtain sufficient information to design such a system. This phase of the evaluation process will vary based on area-specific considerations, but will generally include:

- Assessment of the NAPL physical and chemical properties to assess the migration potential of the NAPL and to aid in selection of pumping equipment and disposal options.

- Assessment of factors that might limit NAPL migration, such as viscosity of the NAPL, soil types, hydraulic factors, and/or presence of existing physical containment barriers. NAPLs with limited potential to migrate offsite or toward surface water bodies may be more appropriately addressed through other measures, such as an enhanced manual removal program.

- Evaluation of potential migration pathways of the NAPL. This evaluation may include the installation and monitoring of sentinel wells (if none already exist) downgradient of the NAPL area. In some cases, installation of an automated recovery system may be deferred until downgradient migration of NAPL can be further assessed by routine monitoring of sentinel wells.

Finally, if after completion of the above evaluations it is determined that additional responses to the presence of NAPL are necessary, the physical characteristics of the area where the system would be located must be taken into consideration, as installation of a recovery system may not be practical in some areas. A generalized automated recovery system will involve a recovery well equipped with NAPL and/or groundwater removal pumps, a holding tank or vessel for the NAPL that is removed, and either piping to route purged groundwater to GE's treatment facility or a large holding tank to store groundwater for disposal (which would need to be accessible to a tanker truck). Some locations may not allow for the placement of these items due to physical or property ownership constraints. In those cases, it may be necessary to implement alternative response actions, such as increased manual monitoring/removal.

Appendix S

Monitoring Well Installation and Development Procedures

Monitoring Well Installation and Development Procedures

- Appendix S-1: This is a standard operating procedure (SOP) that describes the standard procedures for installing and developing overburden groundwater monitoring wells. This SOP is unchanged in substance from the SOP included in the last version of the *Field Sampling Plan/Quality Assurance Project Plan* (FSP/QAPP), dated March 2007 and approved by the U.S. Environmental Protection Agency.

- Appendix S-2: This is a new SOP presenting the procedures for well development and sediment removal from wells using the air lift method

Appendix S-1

Standard Monitoring Well
Installation and Development
Procedures

Standard Monitoring Well Installation and Development Procedures

I. Introduction

Standard procedures for installing and developing overburden groundwater monitoring wells are presented in this Appendix. The monitoring well installation protocol has been developed in accordance with the Massachusetts Department of Environmental Protection (MDEP) Standard Reference for Monitoring Wells (MDEP, 1991; MDEP Publication No. WSC-310-91).

Soil borings and monitoring wells typically will be completed using the hollow-stem auger drilling method. However, direct-push techniques (e.g., Geoprobe® or cone penetrometer) may be used in some cases. No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools, etc.). Prior to beginning work, all underground utilities will be delineated by the drilling contractor or an independent underground utility locator service.

II. Materials

The following materials shall be available during soil boring and monitoring well installation activities, as required:

- Site Plan with proposed soil boring/well locations;
- project Work Plan and *Health and Safety Plan* (HASP);
- personal protective equipment (PPE), as required by the HASP;
- drilling equipment required by ASTM D-1586;
- appropriate sampling equipment (e.g., spatulas);
- equipment cleaning materials (specified in Appendix W);
- appropriate sample containers, labels, and chain-of-custody (COC) forms;
- insulated coolers with ice;
- photoionization detector (PID);
- well construction materials; and
- field notebook.

III. Procedures for Hollow-Stem Auger Monitoring Well Installation

The procedures for the installation of groundwater monitoring wells in soil using the hollow-stem auger drilling method are presented below:

Step 1 - Locate boring/well location, establish work zone, and set up sampling equipment cleaning area.

Step 2 - Advance soil boring to depth specified in work plan. Collect representative soil samples at intervals specified in work plan. The sampling method employed will be the American Society of Testing and Materials (ASTM) D-1586 - Standard Method for Penetration Test and Split-Barrel Sampling of Soils. Samples for laboratory analysis will be collected and handled using procedures outlined in Appendices A, B, C, and N of this document. Fully describe each soil sample, including 1) soil type; 2) color; 3) percent recovery; 4) relative moisture content; 5) soil texture; 6) grain size and shape; 7) consistency; and 8) any other pertinent observations. Record descriptions in field notebook. During soil boring advancement, document all drilling events, including blow counts (number of blows required to advance split-spoon sampler in 6-inch increments) and work stoppages, in field notebook. Blow counts may not be available if direct push methods are utilized.

Step 3 - Upon completion of the borehole to the desired depth, the monitoring well will be installed by lowering the screen and casing assembly with sump through the hollow axis of the auger column. Monitoring wells typically will be constructed of 2-inch diameter, flush-threaded PVC slotted well screen and blank riser casing. Smaller diameters may be utilized if wells are installed using direct-push methodology. The screen length typically will be 10 feet, but will be dictated by field conditions and objectives. The slot size typically will be either 0.010 or 0.020 inches. A 1-foot sump may be attached below the well screen if the well is being installed for DNAPL recovery/monitoring purposes. A blank riser will extend from the top of the screen to approximately 2.5 feet above grade or, if necessary, just below grade where conditions warrant a flush-mounted monitoring well.

Step 4 - When the monitoring well assembly has been set in place, a washed silica sand pack will be placed in the annular space from the bottom of the boring to a height of 1 to 2 feet above the top of the well screen. The graded filter sand pack will be consistent with the screen slot size and the soil particle size in the screened interval. A hydrated bentonite seal, a minimum of 2 feet thick, will then be placed in the annular space above the sand pack. If non-hydrated bentonite is used, the bentonite should be permitted to hydrate in place a minimum of 30 minutes before proceeding. Potable water may be added to hydrate the bentonite if the seal is above the water table. Monitor the placement of the sand pack and bentonite with a weighted tape measure. During the extraction of the augers, a cement/bentonite grout will be pumped through a tremie pipe to fill the annular space from the bentonite seal to a depth approximately 2 feet below the ground surface.

Step 5 - A vented protective steel casing (extended at least 1.5 feet below grade and 2 feet above grade) shall be placed over the riser casing and secured by a neat Portland Cement seal. The cement seal shall extend approximately 1.5 to 2 feet below grade and laterally at least 1 foot in all directions from the protective casing, and shall slope gently away to promote drainage away from the well. A vented slip-on steel cap will be fitted on and around the protective casing. Monitoring wells will be labeled with the appropriate designation both on the inner and outer well casings. A typical above-ground well completion is illustrated in Attachment S-1a.

At those locations where a flush-mounted installation is desired, the steel protective casing will be replaced with a 10-inch curb box or equivalent. When a flush-mounted installation is used, the PVC riser shall be sealed using an unvented expandable locking plug. A typical flush-mount completion is illustrated in Attachment S-1b.

Step 6 - During well installation record exact construction details and actual measurements relayed by the drilling contractor, and tabulate all materials used (e.g., screen and riser footages, bags of bentonite, cement, and sand) in the field notebook.

Step 7 - Following the completion of the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section IX below.

IV. Procedures for Direct-Push Monitoring Well Installation

The direct-push drilling method also may be used to complete soil borings and monitoring wells. Examples of this technique include the Diedrich ESP vibratory probe system or AMS Power Probe® dual-tube system. Environmental probe systems typically use a hydraulically-operated percussion hammer. Depending on the equipment used, the hammer delivers 140 to 350 foot pounds of energy with each blow. The hammer, operated at 1,200 blows per minute, provides the force needed to penetrate very stiff/medium dense soil formations. The hammer simultaneously advances an outer steel casing which contains a dual tube liner for sampling soil. Depending on the system utilized, the outside diameter (OD) of the outer casing ranges from 1.75 to 2.4 inches and the OD of the inner sampling tube ranges from 1.1 to 1.8 inches. The outer casing isolates shallow layers and permits the unit to continue to probe at depth. The double-rod system provides a borehole that may be tremie-grouted from the bottom up. Alternatively, the inside diameter (ID) of the steel casing provides clearance for the installation of small diameter (e.g., 0.75- to 1-inch ID) microwells. The procedures for installing monitoring wells in soil using direct-push methods are described below.

Step 1 - Locate boring/well location, establish work zone, and set up sample equipment cleaning area.

Step 2 - Advance soil boring to designated depth, collecting samples at intervals specified in the work plan. Samples will be collected using dedicated, disposable plastic liners. Describe samples in accordance with the procedures outlined in Step 2 of Section III above. Samples for laboratory analysis will be collected and handled in accordance with procedures described in Appendices A, B, C, and N of this document.

Step 3 - Upon completion of the borehole to the desired depth, install the microwell through the inner drill casing. The microwells will consist of approximately 1-inch ID PVC slotted screen and blank riser. The sand pack, bentonite seal, and cement/bentonite grout will be installed as described, where applicable, in Section III (Steps 3 and 4) above.

Step 4 - Install protective steel casing or flush-mount, as appropriate, as described in Section III (Step 5). During well installation, record exact construction details and tabulate all materials used.

Step 5 - Following the completion of the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section IX below.

V. Procedures for Well Point Installation

Well points will be installed either by hollow-stem auger or direct-push methods (as described above), or hand-driven where possible. The well point construction materials will consist of a 1- to 2-inch diameter threaded steel casing with either 0.010 or 0.020 slotted stainless steel screen. The screen length will vary depending on the hydrogeologic conditions of the site. The casings will be joined together with threaded couplings and the terminal end will consist of a steel well point. Sand pack will not be installed around the screen since the well points are intended for water level/NAPL monitoring and not groundwater sampling.

VI. Equipment Cleaning

All drilling equipment and associated tools, including augers, drill rods, sampling equipment, wrenches, and any other equipment or tools that may have come in contact with soil, shall be cleaned in accordance with the procedures outlined in Appendix W. Well materials and well development equipment will also be cleaned in accordance with the procedures outlined in Appendix W.

VII. Survey

A field survey control program will be conducted using standard instrument survey techniques to document well or piezometer location, ground, and inner and outer casing elevations. Generally, a local control baseline will be set up. If specified in the project-specific work plan, this local baseline control can then be tied into the appropriate vertical and horizontal datum such as the National Geodetic Vertical Datum of 1929 and the State Plane Coordinate System. At a minimum, the elevation of the top of the inner casing which is used for water level measurements should be measured to the nearest 0.01 foot. Elevations will be established in relation to the National Geodetic Vertical Datum of 1929. A permanent mark will be placed on the top of the inner casing to mark the point for water level measurements.

VIII. Procedures for Monitoring Well Development

A. Introduction

All monitoring wells or piezometers which yield water will be developed (i.e., cleared of fine-grained materials and sediments and any drilling fluids) to ensure the screen is transmitting groundwater representative of the surrounding formation groundwater. Development will be accomplished by surging (using a surge block, where possible) and evacuating well water by either pumping or bailing. Acceptable pumping methods include the use of the following:

- electric submersible pump;
- surface inertial pump (Waterra™ pump);

- centrifugal pump; and
- Moyno shaft drilling pump.

When developing a well using the pumping method, dedicated polyethylene tubing from the pump is lowered to the screened portion of the well; the tubing will be moved up and down the screened interval until the well yields relatively clear water. A procedure that may be used for well development includes moving groundwater through the well screen using a centrifugal pump and/or a submersible pump. The centrifugal pump uses atmospheric pressure to lift water from the well and therefore can only be used where the depth to water is less than 25 feet. The submersible pump is attached to the end of the tubing that goes into the well, and therefore pushes the water to the surface; this method is effective for all wells.

B. Materials

Materials for monitoring well development using a pump include:

- health and safety equipment, as required by the HASP;
- cleaning equipment, as required in Appendix W;
- PID to measure headspace vapors;
- polyethylene tubing (discarded between well locations);
- plastic sheeting;
- power source (generator or battery);
- field notebook;
- graduated pails;
- pump;
- appropriate containers; and
- monitoring well keys.

Materials for monitoring well development using a bailer include:

- PPE, as required by the HASP);
- cleaning equipment, as required in Appendix W;
- PID to measure headspace vapors;
- bottom-loading bailer, sand bailer;
- polypropylene rope;
- plastic sheeting;

- graduated pails;
- appropriate containers; and
- monitoring well keys.

C. Development Procedures

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

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

Step 2 - Place plastic sheeting around the well.

Step 3 - All equipment entering each monitoring well will be cleaned as specified in Appendix W.

Step 4 - Open the well cover while standing upwind of the well. Remove well cap. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in the field log. If the well headspace reading is less than 5 PID units, proceed; if the headspace reading is greater than 5 PID units, screen the air within the breathing zone. If the PID reading in the breathing zone is below 5 PID units, proceed. If the PID reading is above 5 PID units, move upwind from well for five minutes to allow the volatiles to dissipate. Repeat the breathing zone test. If the reading is still above 5 PID units, don appropriate respiratory protection in accordance with the requirements of the HASP. Record all PID readings. For wells which are part of the regular weekly monitoring program and prior PID measurements have not resulted in a breathing zone reading above 5 PID units, PID measurements will be taken monthly.

Step 5 - A surge block will be lowered into the screened portion of the well on a rigid pipe or high density tubing and cycled up and down to force water in and out of the screen slots and formation. After surging the well, formation water will be removed by pumping or bailing. Surging and bailing will be performed for a period of 30 to 60 minutes.

Step 6 - If well runs dry, shut off pump and allow well to recover.

Step 7 - Contain all water in appropriate containers.

Step 8 - Continue to remove groundwater until the well is relatively sediment free and turbidity has been reduced to below 50 NTU. If turbidity does not decrease with additional pumping, measure temperature, pH, specific conductivity, and turbidity at approximate 5-10 minute intervals. Other field parameters may also be measured, as appropriate. Record all information on a Well Development Field Log (Attachment S-1c). Development may be terminated once the specific conductance and temperature values remain within 3%, and pH remains within 0.1 units for three consecutive readings collected at approximate five minute intervals.

Step 9 - When complete, secure the lid back on the well.

Step 10- Place plastic sheeting and tubing in plastic bags for appropriate disposal and clean pump as specified in Appendix W.

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

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

Step 2 - Place plastic sheeting around the well.

Step 3 - Bailers and new rope will be cleaned as specified in Appendix W.

Step 4 - Open the well cover while standing upwind of the well. Remove well cap and place on the plastic sheeting. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in the field log. If the well headspace reading is less than 5 PID units, proceed; if the headspace reading is greater than 5 PID units, screen the air within the breathing zone. If the breathing zone reading is less than 5 PID units, proceed. If the PID reading in the breathing zone is above 5 PID units, move upwind from well for 5 minutes to allow the volatiles to dissipate. Repeat the breathing zone test. If the reading is still above 5 PID units, don appropriate respiratory protection in accordance with the requirements of the health and safety plan. Record all PID readings. For wells which are part of the regular weekly monitoring program and prior PID measurements have not resulted in a breathing zone reading above 5 PID units, PID measurements will be taken monthly.

Step 5 - Determine depth of well through examination of drilling log data and measure a length of rope at least 10 feet greater than the total depth of the well.

Step 6 - Secure one end of the rope to the well casing, secure the other end of the rope to the bailer. Test the knots and make sure the rope will not loosen. Check bailers to be sure all parts are intact and will not be lost in the well.

Step 7 - Lower bailer into well until bailer reaches the bottom of the well.

Step 8 - Surge/purge by raising and lowering the bailer at 2-foot intervals at least 10 times.

Step 9 - Contain all water in appropriate containers.

Step 10- Lower bailer back into the well and repeat surging/purging at an interval 2 feet above the previous interval.

Step 11- Repeat Step 8 and Step 9 until entire screen has been surged/purged and the purge water is relatively clear of silt and turbidity has been reduced to below 50 NTU. If turbidity does not decrease with additional bailing, measure temperature, pH, specific conductivity, and turbidity after removal of each quantity of groundwater equivalent to one well volume. Other field parameters may also be measured, as appropriate. Record all information on a Well Development Field Log (Attachment S-1c). Development may be terminated once the specific conductance and temperature values remain within 3%, and pH remains within 0.1 units for three consecutive readings collected at the interval specified above.

Step 12 - Upon completion of surging of the well, remove bailer and remove the rope from the bailer and the well.

Step 13 - Secure lid on well.

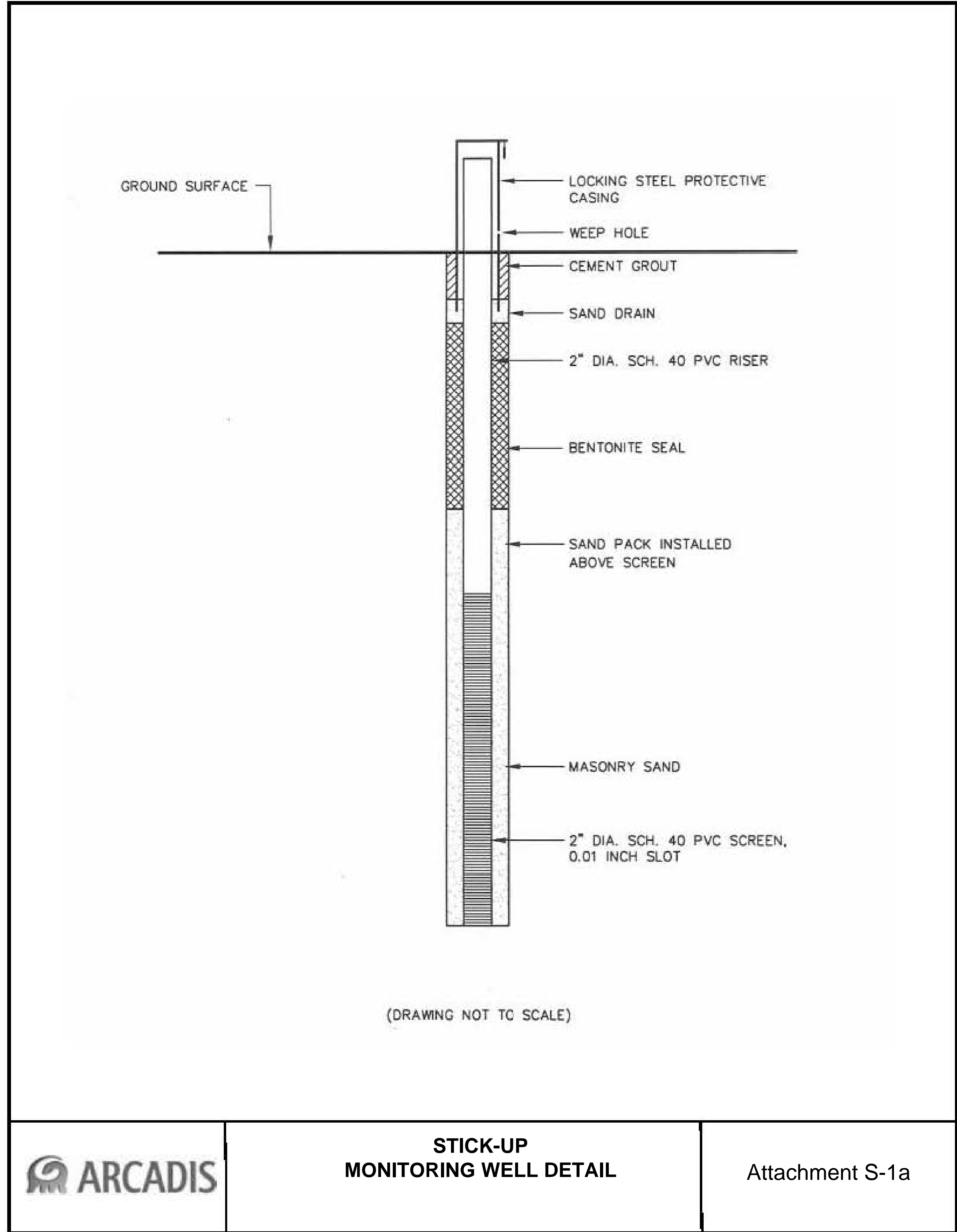
Step 14 - Place plastic sheeting and polypropylene rope in plastic bags for appropriate disposal and clean bailer as specified in Appendix W.

IX. Disposal Methods

Materials generated during monitoring well installation and development will be placed in appropriate containers.

Attachment S-1a

Stick-U Monitoring Well Detail



GROUND SURFACE
LOCKING STEEL PROTECTIVE CASING
WEEP HOLE
CEMENT GROUT
SAND DRAIN
2" DIA. SCH. 40 PVC RISER
BENTONITE SEAL
SAND PACK INSTALLED ABOVE SCREEN
MASONRY SAND
2" DIA. SCH. 40 PVC SCREEN, 0.01 INCH SLOT
(DRAWING NOT TO SCALE)
ARCADIS
STICK-UP
MONITORING WELL DETAIL
Attachment S-1a

Attachment S-1b

Flush Mount Monitoring Well Detail

GROUND SURFACE

WATER TIGHT, BOLTED COVER,
FLUSH-MOUNT PROTECTOR

2" DIA. LOCKABLE GRIPPER PLUG

SAND DRAIN

2" DIA. SCH. 40 PVC RISER

BENTONITE SEAL

SAND PACK INSTALLED
ABOVE SCREEN

MASONRY SAND

2" DIA. SCH. 40 PVC SCREEN,
0.01 INCH SLOT

(DRAWING NOT TO SCALE)

FLUSH MOUNT
MONITORING WELL DETAIL

Attachment S-1b

Attachment S-1c

Well Development Field Log

Attachment S-1c

General Electric Company – Pittsfield, Massachusetts

Site *GMA*

WELL DEVELOPMENT FIELD LOG

BBL Personnel:	**Well ID:**
Oversight Personnel:	**Date:**
Client / Job Number:	**PID (ppm)**
Weather:	**Time In:** **Time Out:**

Well Information

Height of Reference Point (TIC):	(feet AGS)	
Listed Total Depth of Well:	(feet BGS)	
Listed Screen Interval:	(feet BGS)	
Depth to Water (initial): (TIC)	(feet)	Time:
Depth to Water (with equipment): (TIC)	(feet)	Time:
Depth to Water (final): (TIC)	(feet)	Time:
Total Depth (initial): (TIC)	(feet)	
Total Depth (final): (TIC)	(feet)	
Depth to NAPL: (TIC) (DNAPL or LNAPL)	(feet)	
Length of Water Column:	(feet)	
Volume of Water in Well:	(gal)	

Equipment

Probe Type:	Water Level	Interface
Purging Method:	Waterra Pump ❐ Other_______ ❐	
Tubing Type:	Polyethylene ❐	Teflon lined ❐
Well Diameter:	1" ❐ 2" ❐	Other:

Conversion Factors				
gal / ft. of water	1" ID	2" ID	4" ID	6" ID
	0.041	0.163	0.653	1.469
1 gal = 3.785 L =3875 ml = 0.1337 cubic feet				

Purging Information

Pump Start time:		Pump End time:	
Duration of Pumping:	(min)		
Average Pumping Rate:	(ml/min)	Water-Quality Meter Type:	
Total Volume Removed:	(gal)	Did well go dry:	Yes ❐ No ❐

Time Elapsed	1	2	3	4	5	6	7	8	9
Volume Purged (liters)									
Rate (L/min)									
Depth to Water (ft.)									
Color									
Turbidity (NTU)									
Temp ©									
pH									
Conductivity (mS/cm)									
ORP (mV)									
DO (%)									

Note: Temperature, pH, and conductivity data will be collected if well development does not result in turbidities below 50 NTU. Optional parameters (i.e., ORP and DO) may also be collected.

Observations/Method Deviations

Appendix S-2

Well Development and Sediment Removal via Air Lift Method

Well Development and Sediment Removal via Air Lift Method

I. Scope and Application

This standard operating procedure (SOP) describes the use of air lift pumps as a method of well development and sediment removal from wells. While the goal of groundwater sampling is to obtain water samples that are representative of natural, undisturbed hydrogeologic conditions, all drilling methods disturb geologic materials around the well bore to some extent. Development of remediation wells (monitoring wells, piezometers, injection wells, extraction wells) is needed to repair (to the extent practicable) damage to the formation caused by drilling, and to remove fine-grained sediments and drilling fluids introduced during the drilling process. Well development enhances the hydraulic connection between the well and the surrounding formation, ensuring that the screen transmits groundwater that is representative of the surrounding formation. Periodic redevelopment may also be necessary to improve the operation of extraction or injection wells.

The ultimate goal of any development technique is to create a filter pack that is coarsest near the well screen and becomes progressively finer until it blends with the native formation. The ideal development would merge the filter pack seamlessly into the formation, without a noticeable change in grain size.

Development through an air lift pulls formation water into the well screen while simultaneously evacuating water from the well (ideally maintaining an in-well water level that is equal to or below the static water level, but always less than 20 percent of the available head space in the well).

Note: For the most effective development of the filter pack, the screened interval would need to be gently surged using a surge block. This can be done during the air lift process by attaching a surge block to the point or by using an inertia pump (Waterra or similar) to remove fines freed from filter pack.

Selection of the appropriate air lift point and delivery pressure will be based on well details, tubing, and pump specifications. The disposal of investigation-derived waste (IDW) generated during the lifting process must also be taken into consideration.

In general, air lifting involves lowering an air lift assembly consisting of two tubes (see Figures 1 and 2 in Attachment S-2a), an air inflow tube and a water outflow tube (commonly referred to an educator pipe), into the well screen and forcing air into the nozzle causing a vacuum action which will lift the sediment and water out of the well.

Air lift point sizes are selected based on two factors, well diameter and intended purge rate/volume. Air lift points (specifically Buffalo Air Lift type pumps [see manufacturer's specification sheet in Attachment S-2a]) will produce purge volumes between 0.5 and 6 gpm. The extracted flow from the well and the approximate pressure delivered to the air lift to achieve the target flow rates can be determined by considering the following:

- screen material and opening configuration;
- air lift point size;
- tubing pressure limitations; and
- water column above air lift point.

II. Personnel Qualifications

Well development activities will be performed by persons who have been trained in proper field procedures. Well development activities will be performed under the guidance of an experienced field geologist, engineer, or technician.

III. Equipment List

General materials for well development include:

- Personal protective equipment (PPE) – specifically eye protection, nitrile gloves, hearing protection, and steel toe boots; as well as any other safety equipment required by the site-specific Health and Safety Plan (HASP);
- Cleaning equipment;
- Water level meter and/or oil/water interface probe;
- Water quality meter that is capable of recording pH, temperature, conductivity, and turbidity;
- Photoionization detector (PID) to measure headspace vapors (recommended; may be required by the site-specific HASP);
- Plastic sheeting;
- Polypropylene graduated beaker with handle, 5000 mL;
- Stop watch or chronograph wrist watch;
- Two (2) five-gallon graduated pails with lids;
- Drum(s) or tank(s) to contain purge water, and equipment to move the container(s);
- Field notebook;
- Well construction logs (or summary table) indicating completed well depths and screened intervals; and
- Monitoring well keys.

Materials needed specifically for the air lift method include:

- Down-hole air lift assembly consisting of:
  - Air Lift Point (see Figure 2 in Attachment S-2a):
    1) For wells with a diameter of 0.75" - 2": 6.5" long x 11/16" diameter stainless steel point with 3/8" barb for water line effluent line and 1/4" barb for air line influent; or
    2) For wells with a diameter of 2"- 4": 12" long x 1.3" diameter stainless steel point with 1" barb for water effluent line and 1/4" barb for air influent;
  - 1/2" OD flexible tubing (for 11/16" air lift point);
  - 1" OD flexible or 1" PVC piping (for 1.3" air lift point);
  - 1/4" OD flexible tubing for air supply line;
  - Surge block designed for well diameter; and
  - Associated tubing bundled together (e.g., with zip ties or silicone tape).

  Note: The air lift assembly must be sufficiently rigid and bundled to minimize friction between the well casing and the tubing and to allow vertical movement. Ease of the air lift hose/ movement within the well can become a concern in small (i.e., 2-inch and smaller) diameter wells if the correct air lift point is not correctly identified.

- Above-grade jetting assembly consisting of:
  - A pressure-controllable air compressor;
  - Air hose with quick connect fitting to accommodate ¼"OD tubing;
  - Whip guards for air compressor tubing;
  - Inverter with jumper cables (for locations where a generator is not practical);
  - 1000W or greater generator; and
  - Extension cord with GFCI.

IV. Cautions

Delivery pressures greater than 50 pounds per square inch (psi) are often required to achieve effective air lifting. All tubing/piping, connections, and pumps should be rated for the anticipated delivery pressures, and should be inspected for damage prior to and periodically during use.

Care should be taken when testing the air lift point above ground. High pressure air exiting the point may pose a projectile risk if it comes into contact with dirt and sands.

V. Health and Safety Considerations

Field activities associated with well development by air lift will be performed in accordance with the site-specific HASP, a copy of which will be present on-site during such activities. Note that additional precautions may be required to account for the use of pressurized equipment or handling large storage vessels.

VI. Procedure

The procedures for well development and sediment removal using the air lift method are outlined below. As noted above, when developing the screen zone, a surge block needs to be used in addition to the air lift point. These procedures are applicable to wells that are screened primarily in sand and silt formations.

1. Don appropriate PPE (as required by the HASP).

2. Using a non-phosphate cleaner (e.g., Alconox) and potable water, clean and double rinse all non-dedicated equipment (air lift point) that will enter the well (refer to separate equipment cleaning procedures where applicable).

3. Breathing zone testing is recommended (to be determined by the project team). If required:

   a. Open the well cover while standing upwind of the well; remove the well cap; insert the PID probe approximately 4 to 6 inches into the casing or the well headspace; cover with gloved hand.

   b. Record the PID reading in the field notebook.

   c. If the well headspace reading is less than 5 ppm, proceed; if the headspace reading is greater than 5 ppm, screen the air within the breathing zone. If the PID reading in the breathing zone is below 5 ppm, proceed. If the PID reading is above 5 ppm, move upwind from the well for 5 minutes to allow the volatiles to dissipate, then repeat the breathing zone test. If the reading is still above 5 ppm, don the appropriate respiratory protection in accordance with the requirements of the HASP. Record all PID readings.

4. Measure the depth to water and total well depth. Check for the presence of non-aqueous-phase liquid (NAPL). *If NAPL is present, do not continue development until consulting with the task manager (or technical lead).* Compare the well depth to the as-built construction details. Calculate the volume of water in the well casing. (See attached volume calculation table in Attachment S-2b.)

5. Push on all tubing connections to the applicable barbs on the air lift point using an adjustable tightening clamp.

   - Check to confirm tubing is tightly secured to point by slightly pulling hoses.

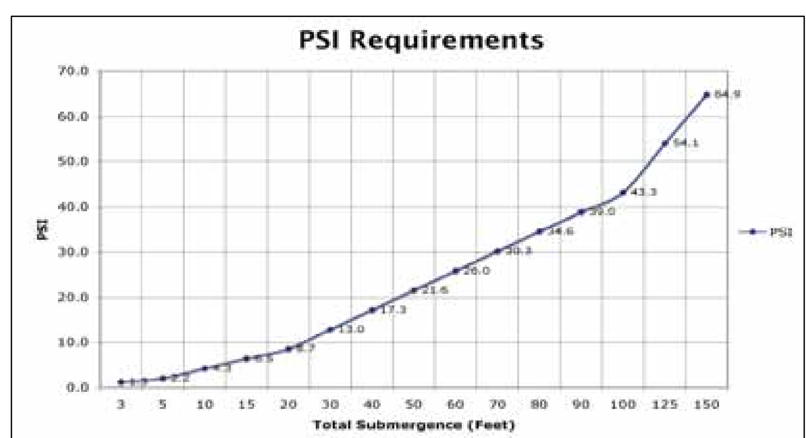
6. Determine purging parameters and set-up:

   a. Calculate the operational air lift setting:

FORMULAS

$$\frac{\text{Total Depth of Well} - \text{Depth To Water}}{2.31} = \text{PSI}$$

$$\left(1 - \frac{\text{Depth To Water}}{\text{Total Depth of Well}}\right) \times 100 = \text{\% SUBMERGENCE}$$



   b. If a surge block is to be used, attach it to or above the air lift point.

   c. Remove interface probe from the well, and then lower the air lift point into the well. Check that the air lift point and tubing is loose enough to permit the point to move up and down inside the well casing without significant effort (see Figure 1 in Attachment S-2a). Mark on the hose the total depth from the top of the inner casing. Ensure that adequate tubing is attached to allow the pump to reach the bottom of the well if sediment is removed.

   d. Connect the ¼'' OD air line to the quick connect on the air hose. Close the air valve. Always raise the pump off the bottom of well prior to turning the air supply on and off.

   e. Place the end of the water discharge line in a bucket with a hole or slits cut in the lid to prevent splashing.

   f. Turn on air compressor. Rotate pressure valve to the value calculated PSI identified in step "a". Slowly open the air valve.

   g. After starting air flow, raise pump slowly 6 inches from the bottom and lower back. It is best to have good water flow before attempting sediment removal at the bottom of the well. If pump does not start, slowly raise pressure to a level that produces water. (Note: if the volume of water

in the well is less than the volume of the discharge tubing, the pump may not lift any water until the well has recharged. Do not shut off the pump in this case as the water will drain into the well, eliminating recharge.)

g. Place a water level meter into the well to monitor the water level during operation.

7. For well screen development, surge and purge the saturated portion of the well screen in 2-foot increments, as follows: (For sediment removal from base of well, see Step 8.)

   a. Start sediment and water removal in the bottom 2-foot interval of the well screen. While purging:

      i. Turn on the power supply and air compressor. Set pressure gauge to appropriate value as calculated in Step 6. Open valve and collect initial flow rate using a stop watch and graduated beaker.

      ii. Gently surge the well while pumping by slowly moving the air lift point/surge block up and down the well screen at no greater than 0.5 ft/sec. Vigorous surging is not appropriate. Do not reverse the up/down stroke suddenly.

      ii. Hold the water line away from the body. If flow/surging rates are imbalanced or a sediment blockage prevents flow, the tool may push upward or downward. Do not force the tool to remain stationary; adjust point placement and surging rates as needed.

      iv. Do not let the tool remain in one position for longer than a few seconds.

   b. Continue purging in the 2-foot interval for 10 minutes.

   c. Repeat steps 7a and 7b in the next 2-foot interval of screen until the entire length of the saturated screen interval has been developed.

   Sediment loading and turbidity of the extracted water should improve throughout the removal process. Visual observations of the sediment and turbidity should be recorded in the field notes or the well development log (Attachment S-2c). The project team may opt to record water quality parameters (temperature, conductivity, pH, turbidity) during development. At a minimum, the initial purge water and each well volume removed should have water quality measurements recorded.

8. For sediment removal from the base of the well, position the pump at or just above the measured bottom of the well. Occasionally raise and then lower the pump several feet if flow slows down, as this may help restart the flow if the pump begins to fill with silt. Be careful not to allow the pump to suction to the bottom of the well.

9. If possible, monitor and record water level throughout the process, beginning at 5-minute intervals and then at longer intervals if there is little change. Decreasing the extraction rate may be necessary to prevent the water level in the well from reaching the bottom of the point.

10. After air lifting, measure the depth to water and the total well depth, and check for the presence of NAPL. Confirm that the total depth of the well matches the as-built well depth within a reasonable tolerance. If a discrepancy exists, note it, and evaluate it to the degree feasible. Continue if necessary.

11. When complete, re-secure the well cover.

12. Using a non-phosphate cleaner (e.g., Alconox) and potable water, clean and double rinse all non-dedicated equipment that entered the well (refer to separate equipment cleaning procedures where applicable). Place disposable materials in plastic bags for appropriate disposal, and decontaminate reusable, downhole pump components.

VII. Waste Management

IDW generated during well development may include disposable equipment and PPE, purged groundwater, and water associated with equipment cleaning. All disposable and liquid waste should be handled and disposed of in accordance with project plans and applicable regulations.

VIII. Data Recording and Management

Well development activities will be documented in a proper field notebook and/or Personal Digital Assistant (PDA). Pertinent information will include:

- General Field Notes:
  - personnel present on-site;
  - times of arrival and departure;
  - significant weather conditions; and
  - timing of well development activities.
- Air Lift Field Notes:
  - observations of NAPL;
  - air compressor pressure;
  - water levels before and during testing;
  - observations of purge water color, turbidity, odor, and sheen over time;
  - purge rate;

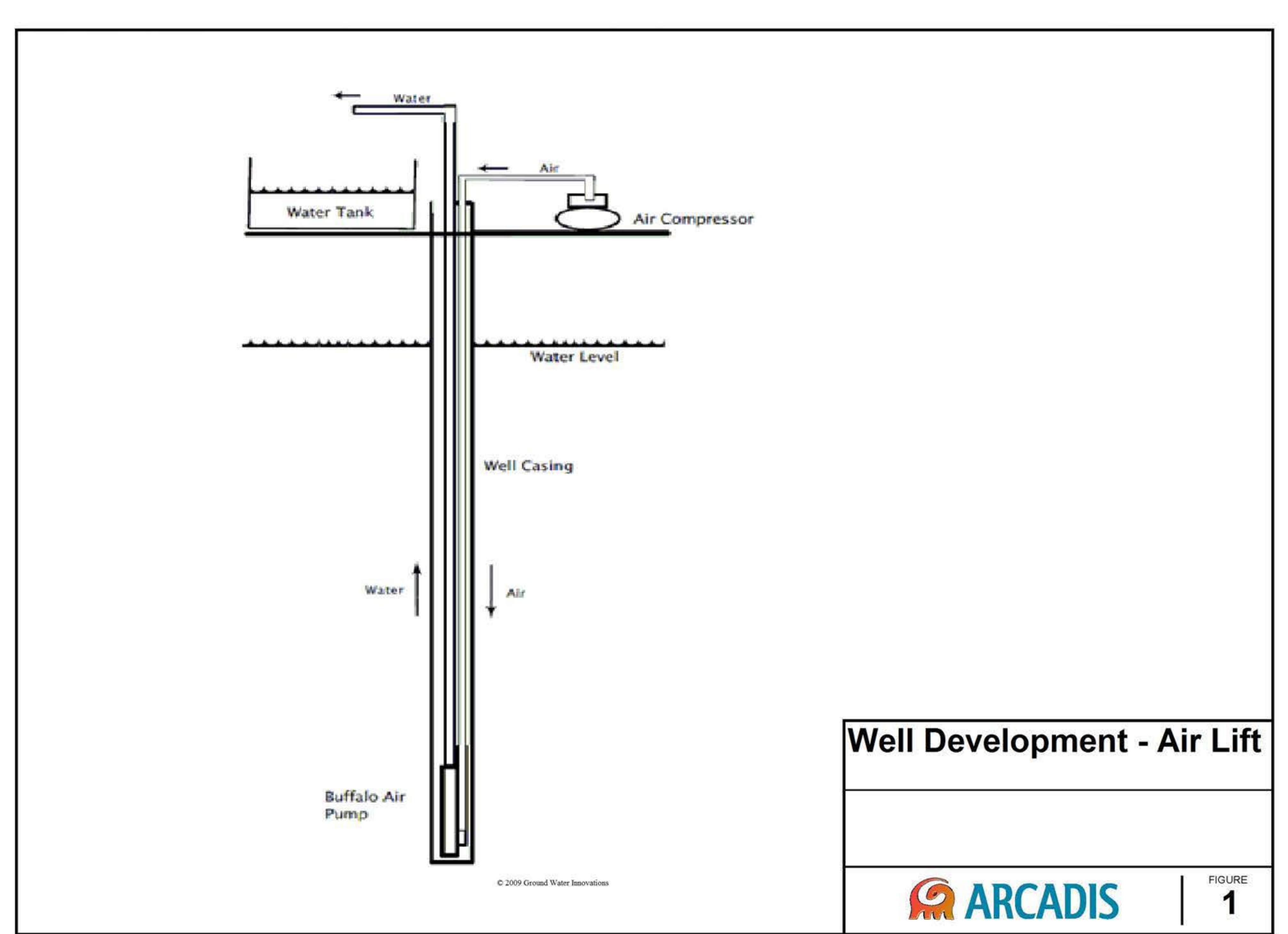
- initial and final total depth of well; and
- water quality parameters (specified by project technical lead).

IX. Attachment

Well Development Log (Attachment S-2c)

Attachment S-2a

Figures and Manufacturer's Specifications


Water
Air
Water Tank
Air Compressor
Water Level
Well Casing
Water
Air
Buffalo Air
Pump
© 2009 Ground Water Innovations
Well Development - Air Lift
ARCADIS
FIGURE
1

2" Air Lift Point (Top View)

Well Development - Air Lift

ARCADIS

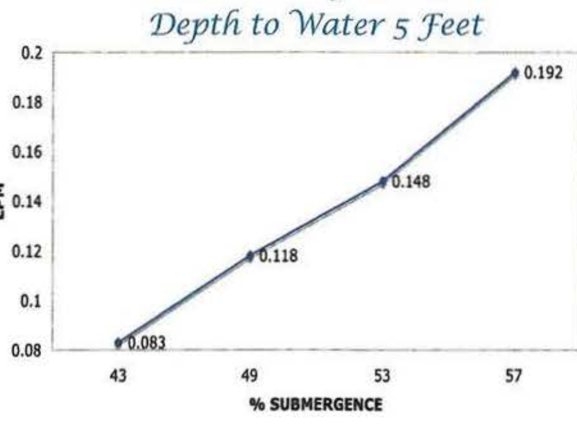
FIGURE 2

BUFFALO AIR PUMPS

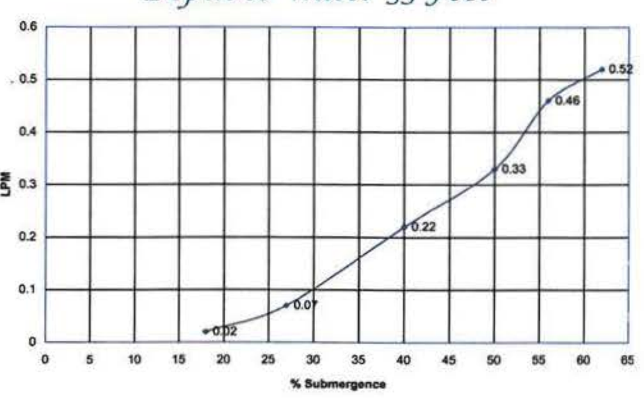
WWW.GROUNDWATERINNOVATIONS.COM

3/4 Inch Pump

Total Well Depth 12 Feet
Depth to Water 5 Feet

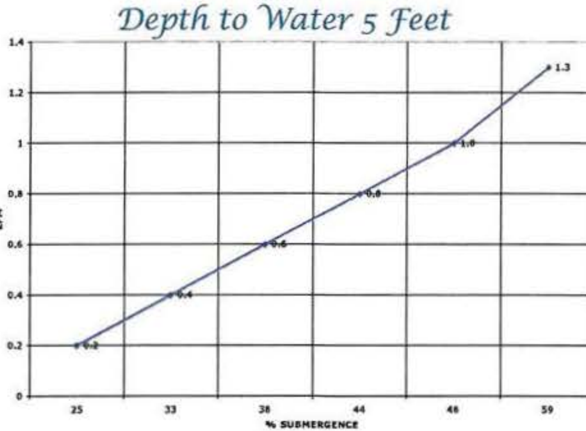


Total Well Depth 85 Feet
Depth to Water 33 Feet

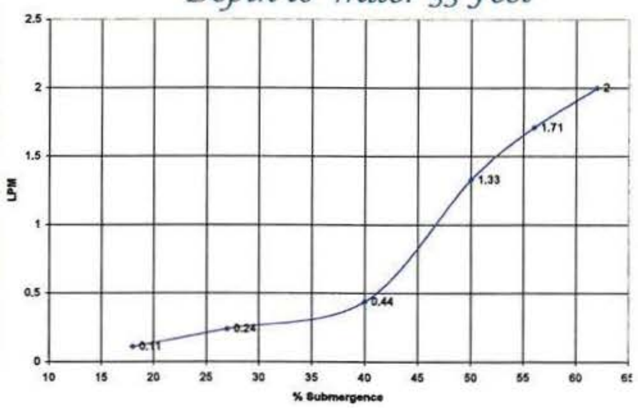


1 Inch Pump

Total Well Depth 12 Feet
Depth to Water 5 Feet

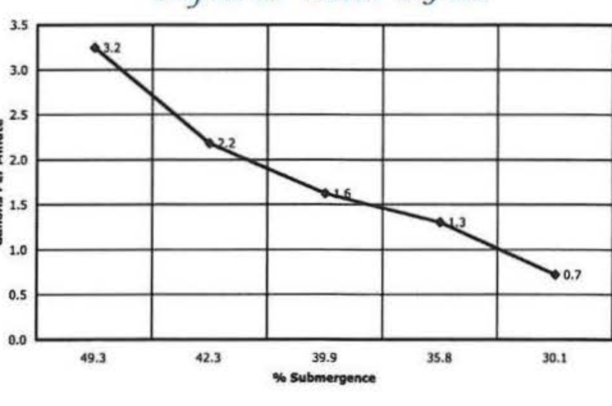


Total Well Depth 85 Feet
Depth to Water 33 Feet

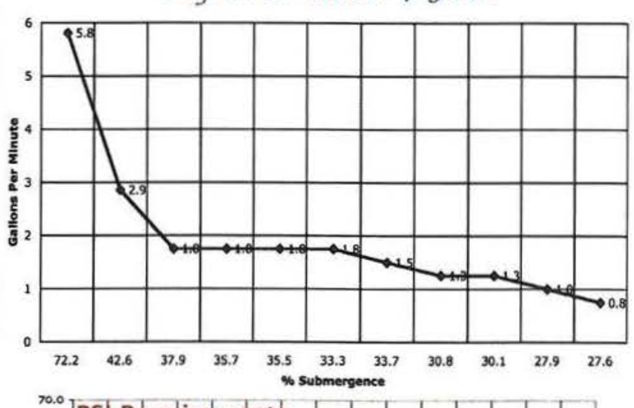


2 Inch Pump

Total Well Depth 21 Feet
Depth to Water 6 Feet



Total Well Depth 108 Feet
Depth to Water 7 Feet



PUMP SPECIFICATIONS

ITEM NAME	ITEM DESCRIPTION	WEIGHT (LBS)	MATERIAL	LENGTH	DIAMETER
Buffalo Air Pump	Air Lift Pump for 2 Inch + Wells	2	304 Stainless Steel	12	21/16 ths
Buffalo Air Pump	Air Lift Pump for 1 Inch Micro Wells	0.94	304 Stainless Steel	8.5	15/16 ths
Buffalo Air Pump	Air Lift Pump for 3/4 Inch Micro Wells	0.13	304 Stainless Steel	6.5	11/16 ths

PSI Requirements

PSI

Total Submergence (Feet)

841 East Fort Avenue * Suite 262 * Baltimore * MD 21230 * 800.476.5170 * Robert@groundwaterinnovations.com

Attachment S-2b

Well Volume Calculations Table

Attachment S-2b

WELL VOLUME CALCULATIONS

DIAMETER (inches)	DIAMETER (feet)	RADIUS (feet)	R-SQ (sq. feet)	VOLUME (gal/foot)	VOLUME (liter/foot)	VOLUME (liter/0.5foot)	VOLUME (liter/0.25foot)	VOLUME (ml/0.1foot)	VOLUME (ml/0.01foot)
0.25	0.021	0.010	0.000	0.003	0.010	0.005	0.002	0.965	0.097
0.38	0.031	0.016	0.000	0.006	0.022	0.011	0.005	2.172	0.217
0.50	0.042	0.021	0.000	0.010	0.039	0.019	0.010	3.861	0.386
0.75	0.063	0.031	0.001	0.023	0.087	0.043	0.022	8.687	0.869
1.00	0.083	0.042	0.002	0.041	0.154	0.077	0.039	15.444	1.544
1.25	0.104	0.052	0.003	0.064	0.241	0.121	0.060	24.131	2.413
1.50	0.125	0.063	0.004	0.092	0.347	0.174	0.087	34.748	3.475
1.75	0.146	0.073	0.005	0.125	0.473	0.236	0.118	47.297	4.730
2	0.167	0.083	0.007	0.163	0.618	0.309	0.154	61.775	6.178
3	0.250	0.125	0.016	0.367	1.390	0.695	0.347	138.994	13.899
4	0.333	0.167	0.028	0.653	2.471	1.236	0.618	247.100	24.710
6	0.500	0.250	0.063	1.469	5.560	2.780	1.390	555.975	55.598
8	0.667	0.333	0.111	2.611	9.884	4.942	2.471	988.400	98.840
10	0.833	0.417	0.174	4.080	15.444	7.722	3.861	1,544.376	154.438
12	1.000	0.500	0.250	5.876	22.239	11.120	5.560	2,223.901	222.390
16	1.333	0.667	0.444	10.445	39.536	19.768	9.884	3,953.602	395.360
24	2.000	1.000	1.000	23.502	88.956	44.478	22.239	8,895.603	889.560
36	3.000	1.500	2.250	52.880	200.151	100.076	50.038	20,015.108	2,001.511

VOLUME IN GALLONS = [PI * (RADIUS IN FEET) SQUARED * (7.481 CU. FT. PER GALLON)] * (LENGTH OF WATER COLUMN IN FEET)
THESE CONVERSIONS CAN BE USED TO OBTAIN WELL VOLUMES FOR PURGING PURPOSES, BOREHOLE VOLUMES FOR GROUTING REQUIREMENT CALCULATIONS, OR VOLUMES WITHIN SAMPLE TUBING INSERTED INTO A WELL.

Attachment S-2c

Well Development Field Log

Attachment S-2c

General Electric Company – Pittsfield, Massachusetts

Site *GMA*

WELL DEVELOPMENT FIELD LOG

BBL Personnel:		**Well ID:**	
Oversight Personnel:		**Date:**	
Client / Job Number:		**PID (ppm)**	
Weather:		**Time In:**	**Time Out:**

Well Information

Height of Reference Point (TIC):	(feet AGS)	
Listed Total Depth of Well:	(feet BGS)	
Listed Screen Interval:	(feet BGS)	
Depth to Water (initial): (TIC)	(feet)	Time:
Depth to Water (with equipment): (TIC)	(feet)	Time:
Depth to Water (final): (TIC)	(feet)	Time:
Total Depth (initial): (TIC)	(feet)	
Total Depth (final): (TIC)	(feet)	
Depth to NAPL: (TIC) (DNAPL or LNAPL)	(feet)	
Length of Water Column:	(feet)	
Volume of Water in Well:	(gal)	

Equipment

Probe Type:	Water Level	Interface
Purging Method:	Waterra Pump ☐ Other_______ ☐	
Tubing Type:	Polyethylene ☐	Teflon lined ☐
Well Diameter:	1" ☐ 2" ☐	Other:

Conversion Factors				
gal / ft. of water	1" ID	2" ID	4" ID	6" ID
	0.041	0.163	0.653	1.469
1 gal = 3.785 L =3875 ml = 0.1337 cubic feet				

Purging Information

Pump Start time:		Pump End time:	
Duration of Pumping:	(min)		
Average Pumping Rate:	(ml/min)	Water-Quality Meter Type:	
Total Volume Removed:	(gal)	Did well go dry:	Yes ☐ No ☐

Time Elapsed	1	2	3	4	5	6	7	8	9
Volume Purged (liters)									
Rate (L/min)									
Depth to Water (ft.)									
Color									
Turbidity (NTU)									
Temp ©									
pH									
Conductivity (mS/cm)									
ORP (mV)									
DO (%)									

Note: Temperature, pH, and conductivity data will be collected if well development does not result in turbidities below 50 NTU. Optional parameters (i.e., ORP and DO) may also be collected.

Observations/Method Deviations

Appendix T

Magnetometer Survey Procedures

Magnetometer Survey Procedures

I. Introduction

A magnetometer survey is typically performed to detect the presence of buried ferrous metal objects. The magnetometer operates on the principle of measuring the earth's magnetic field and deviations in this field caused by the presence of ferrous metal objects. The intensity and variation caused by such objects are related to the depth and mass of the buried object and, to a lesser degree, the orientation of the object. Magnetometer surveys can be performed using either a single sensor or dual sensors (gradiometer) depending on the objectives of the survey and the size of the subsurface features to be delineated.

The following methodologies will be utilized to perform magnetometer surveys.

II. Materials

The following equipment and materials will be available, as required, during magnetometer surveys.

- Health and safety equipment (as required in the Health and Safety Plan);
- Appropriate forms/field notebook; and
- Geometrics G-858 portable cesium magnetometer or gradiometer, or the equivalent
- Trimble AG-132 Global Positioning System (GPS) or equivalent, (optional equipment for the magnetometer);
- Measuring tapes (100- to 300-foot lengths, as needed);and
- Survey stakes, marking paint, traffic cones, or other visual marker aids.

III. Procedures

Step 1 - Identify the traverse location on the appropriate form (Attachment T-1) and on the field notebook along with other appropriate information.

Step 2 - Don personal protective equipment (as required by the Health and Safety Plan).

Step 3 - Establish grid system by standard surveying techniques to document the location of each grid point. The grid spacing will be sufficient to detail the site(s) location, boundaries, and survey targets.

Step 4 - Establish a base-station location in an area free of interferences to collect background data to determine the daily fluctuations (diurnal) in the earth's magnetic field during the field survey. Base station measurements can be collected every 30 minutes (on average) using the same Geonics G-856 magnetometer, or using a separated magnetometer set up to collect data at selected intervals.

Step 5 - Calibrate (adjust) the magnetometer, if necessary, before beginning survey activities to account for the earth's magnetic field in the project area.

Step 6 - Utilizing a Geometrics G-858 magnetometer or the equivalent, conduct the survey. Operate the magnetometer in accordance with the operating manual.

Step 7 - At each point of the grid system, record the time, station location, and magnetometer readings on a standard form (Attachment T-1), or in the magnetometer's digital memory. Note locations containing metal debris, equipment, above ground structures, and utilities in the field book.

Step 8 - The data from the magnetometer surveys will be corrected for diurnal variation using the base station measurements before contouring and evaluating the data. Download and correct the magnetometer data using the software supplied with the instrument, Geometrics MagMap 2000, or equivalent.

Step 9 - Using the corrected magnetometer data, plot the data set utilizing appropriate contouring software (e.g., Golden Software, Inc. – Surfer, or equivalent).

Attachment T-1

Magnetometer Survey

MAGNETOMETER SURVEY

CREW____________________ WEATHER_______________ NOTES______________

____________________ ______________________

____

DATE/TIME	STATION	READING

Appendix U

Seismic Refraction Survey Procedures

Seismic Refraction Survey Procedures

I. Introduction

The following methodologies will be utilized to perform seismic refraction surveys.

II. Materials

The following equipment and materials will be available, as required, during seismic refraction surveys.

- Health and safety equipment (as required in the Health and Safety Plan);
- Appropriate forms/field notebook;
- Geometrics Nimbus Model ES-1210F multi-channel signal enhancement seismograph, or equivalent;
- Geophones and connecting cables; and
- Seismic source (sledge hammer, explosives, or other device).

III. Procedures

Step 1 - Identify the traverse location in the field notebook along with other appropriate information.

Step 2 - Don personal protective equipment (as required by the Health and Safety Plan).

Step 3 - Lay out seismic refraction lines along the ground surface between previously-surveyed grid points, between test borings and/or monitoring wells, or in areas determined by the geologist.

Step 4 - During cable layout, flag the shot points and label with location, line number, and forward/reverse. These flagged seismic lines will later be surveyed by standard techniques to document the location of the seismic line and the elevations of the shot points. If the terrain along the line has substantial relief, additional elevations will be acquired along the seismic line at selected geophone positions.

Step 5 - The exact spacing of the geophones will be determined by the geologist on-site after trial lines are completed. Select the spacing to define the best configuration to provide the most data on overburden and bedrock velocities.

Step 6 - Utilizing an EG&G Geometrics Nimbus ES-1210F multi-channel signal enhancement seismograph, or equivalent, perform the survey. Operate the instrument in accordance with guidelines outlined in the operating manual.

Step 7 - At a minimum, perform forward and reverse shots at each end of each seismic line and at least one midpoint shot. These shots will be offset between the geophones at the end of the cable. Supplementary shots along the length of the line, or outside the line endpoints may be made if considered necessary by the geologist.

Step 8 - Given the depth to bedrock, explosives may be utilized for the shot. In this case, a licensed expert must be used. Energy can also be generated on the surface with a weight dropped on a steel plate attached to a sensor. Drop the weight until a sufficient record is achieved by signal enhancement. The geologist will determine which method is suitable.

Step 9 - Perform data analysis using commercially available software, or manually using the crossover-distance method.

Step 10 - Interpret distance and arrival times from each record and plot graphically for each seismic line. Interpret the data by manual manipulation of the data or a computer model using both intercept time and critical distance techniques.

Appendix V

Ground Penetrating Radar (GPR) Procedures

Ground Penetrating Radar (GRP) Procedures

I. Introduction

GPR equipment transmits high frequency electromagnetic waves into the ground and detects energy reflected back to the surface. Energy is reflected along subsurface interfaces that possess different electrical properties. Reflections typically occur at lithologic contacts or when the electromagnetic waves encounter subsurface materials having high electrical contrasts, including metal objects such as underground storage tanks (USTs), drums, and utility pipes. These reflections are detected by the antenna and processed into an electrical signal, which can then be used to image the subsurface feature.

The following methodologies will be used to perform GPR surveys.

II. Materials

The following equipment will be available, as required, during GPR surveys.

- Health and safety equipment (as required in the Health and Safety Plan);
- Appropriate forms or field notebook;
- Geophysical Survey System, Inc. (GSSI) Subsurface Interfacing Radar (SIR) System-2000 radar or equivalent;
- One antenna of an appropriate frequency (typically 100 to 500 megahertz) to achieve the survey depth needed, and delineate the subsurface features of interest;
- Trimble AG-132 GPS, or equivalent (optional with GPR system)
- Non-conductive measuring tape; and
- Connecting cables, survey wheel, and 12-volt power source.

III. Procedures

Step 1 - Identify the traverse location(s) in the field notebook or on a site plan map.

Step 2 - Don personal protective equipment (as required by the Health and Safety Plan).

Step 3 - Establish a temporary control grid over the designated survey area(s) using conventional surveying methods and/or referenced to the site plan using a baseline established from site features. Layout the measuring tape along the desired traverse, or mark a reference grid on the ground using the measuring tape.

Step 4 - Initial calibration of the GPR system and antenna will be performed using subsurface soil boring information, if available, and observed response of the GPR's analog signal. Calibration of the system will be completed using the GSSI system setting, and adjusting the range and dielectric constant parameters to the approximate subsurface conditions at the site. If available, calibrate the depth (using the dielectric constant) of the GPR over a buried pipe (or other object) of known depth. Re-calibrate the equipment if the antenna or system settings are changed.

Step 5 - Connect the GPR control unit and antenna with appropriate cables, and adjust the instrument gains, if needed, to obtain a satisfactory record throughout the desired survey depth range.

Step 6 - Use the survey control grid to determine the GPR survey line location and sequence for collecting the GPR data. Optionally, use a differential GPS system connected to the GPR system to locate the data collected along each survey line.

Step 7 - Record GPR data while slowly pulling the antenna(s) along the survey traverse(s). Annotate the record using the antenna's marker switch, at even distance increments (10 feet, or as needed).

Step 8 - Make note of any variable surface condition (e.g., terrain changes, surface cover materials, standing water) that could affect data interpretation. Also note any surface expressions of potential buried utilities or structures.

Step 9 - Conduct data analysis in accordance with the manufacturer's recommendations using RADAN for Windows software, or equivalent and industry practice.

Appendix W

Equipment Cleaning Procedures

Equipment Cleaning

I. Introduction

The equipment cleaning procedures described herein apply to: (a) the cleaning of relatively small soil and water sampling equipment in the field or potentially at a temporary Equipment Decontamination Area (EDA) set up outside GE's Building 78; and (b) the cleaning of heavy equipment and subsurface soil sampling equipment at an EDA outside Building 78 or an alternate location at the job site. Non-disposable equipment will be cleaned after completing each sampling event, between sampling events, and prior to leaving the site. Cleaning procedures of sampling equipment will be monitored through collection of field blank samples or wipe samples as specified in the applicable work plan.

II. Cleaning of Relatively Small Sampling Equipment

This procedure applies to the cleaning of relatively small sampling equipment used for the collection of surface soil, groundwater, sediment, or surface water samples or for water testing or geophysical investigations. Such equipment may include soil core samplers, bailers, well pumps, spatulas, etc. Such equipment will be cleaned in the field prior to and between sample collections (or other use) at a designated equipment cleaning area established within or adjacent to the specific work area as approved by GE and the site supervisor, or potentially at a temporary EDA next to Building 78.

A. Cleaning Materials

The following materials, as required, will be available during cleaning procedures:

- health and safety equipment, as required in GE's Health and Safety Plan (HASP);
- distilled/deionized water;
- non-phosphate soap (e.g., Alconox);
- tap water;
- appropriate cleaning solvent (e.g., hexane, acetone);
- nitric acid (10% solution or greater);
- rinse collection plastic containers;
- plastic overpack drum
- brushes;
- plastic sheeting;
- large heavy-duty garbage bags;

- spray bottles;
- resealable-type bags;
- handiwipes; and
- field notebook.

B. Cleaning of Small Sampling Equipment when Analyzing for Organic Constituents

Step 1 - On clean 6-mil or thicker plastic sheeting, in a clean 5-gal bucket, use a non-phosphate detergent and water wash to removal all visible particulate matter and any residual oils or grease.

Step 2 - Within the 5-gal bucket, on top of the protective sheeting, use a tap water rinse to remove the detergent solution.

Step 3 - Within the 5-gal bucket, use a solvent rinse with hexane (unless volatiles are being sampled, in which case methanol should be used).

Step 4 - Within the 5-gal bucket, use a distilled/deionized water rinse.

Step 5 - Repeat solvent and water rinse two more times (i.e., triple rinse) and allow to air dry.

C. Cleaning of Small Sampling Equipment when Analyzing for Inorganic Constituents

Step 1 - On clean 6-mil or thicker plastic sheeting, in a clean 5-gal bucket, use a non-phosphate detergent and water wash to removal all visible particulate matter and any residual oils or grease.

Step 2 - Within the 5-gal bucket, on top of the protective sheeting, use a tap water rinse to remove the detergent solution.

Step 3 - Within the 5-gal bucket, use a nitric acid rinse.

Step 4 - Within the 5-gal bucket, use a distilled/deionized water rinse.

D. Decontamination of Submersible Pumps

Submersible pumps may be used to evacuate stagnant groundwater in the well casing. The pumps will be cleaned and flushed between uses. This cleaning process will consist of an external detergent wash (e.g., Alconox or similar) and tap water rinse, followed by a flush of potable water through the pump in a clean 5-gallon bucket. Then a final flushing will be accomplished by the use of an appropriate container filled with distilled water. The pump will run long enough to effectively flush the pump housing and hose.

Caution should be exercised to avoid contact with the pump casing and water in the container while the pump is running (do not use metal drums or garbage cans) to avoid electric shock. The pump should be disconnected from a power source before handling. The pump and hose should be placed on clean polyethylene sheeting to avoid contact with the ground surface.

E. Disposal Methods

Rinse water, personal protective equipment (PPE), and other residuals generated during the equipment cleaning procedures will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its ongoing disposal practices.

III. Decontamination of Heavy Equipment and Subsurface Soil Sampling Equipment

This procedure applies to the decontamination of heavy equipment, such as drill rigs, well casings, auger flights, split spoons, etc. Such equipment will be cleaned within a temporary EDA located next to GE's Building 78 or at an appropriate alternate location on the job site approved by GE and the site supervisor. The temporary EDA must be constructed in a way to provide adequate splash protection for the areas outside the EDA and surround the intended items to be cleaned. Heavy equipment may potentially retain contaminants from other sources, such as roadways or storage areas, or have soil material from previous job sites that have not been removed.

If heavy equipment brought on site is suspected to contain contaminants from a prior job, it will be thoroughly cleaned according to the procedures described below. It will also be cleaned between drilling locations. Portions of the equipment that are in close proximity to materials being sampled, such as auger flights, drill rods, and drill bits, will be targeted for cleaning.

A. Safety Precautions

Before a piece of equipment can be cleaned, it must be disconnected and disabled in accordance with standard Energy Control and Power Lock-Out Procedures as presented in GE's HASP (Section 4.16). All energy sources including stored energy must be removed prior to cleaning.

Do not attempt to clean equipment that is in service or still connected to power.

Protective clothing, in addition to that specified by general plant safety procedures (i.e., safety glasses, safety-toe shoes), is required during cleaning. The cleaning contractor shall have a written HASP appropriate for the expected operations including measurements for determining the need for more stringent levels of protection. The minimum allowable protective clothing shall include:

- plastic face shields;
- disposable Tyvek coveralls (Dupont/Saranex 23-P or equal);

- impervious rubber boots (neoprene, viton, or equal); and
- impervious gloves (neoprene, viton, or equal).

Additional protective equipment may be required for some tasks. These contingencies should be included in the HASP.

B. Required Equipment

The following equipment will be required for use during cleaning procedures:

- utility pump;
- lint-free absorbent towels;
- 6-mil polyethylene sheeting;
- assorted scrub brushes;
- waste disposal drums;
- cleaning fluids such as Knights Super Kleen, Simple Green, Aquanex MC, Zep Formula 50, Zep Big Orange, or equal;
- aluminum duct tape; and
- oil/water absorbent Speedi-Dry compounds.
- steam pressure washer (e.g., MI-T-M Steam Pressure Washer or similar)

C. Set Up

Step 1 - Put on protective clothing: face shield, impervious boot covers and Tyvek.

Step 2 - Provide proper signs and barricades for the cleaning area to control access.

Step 3 - Set up the temporary EDA next to Building 78 or at the designated GE-approved site location. This area should be larger than the intended items to be cleaned and should provide splash walls to contain all solids and liquids generated.

Step 4 - Construct the EDA out of 2x8 inch wood planks or similar (e.g., hay bales) to create a four-sided berm. Placed on top of the berm should be two layers of minimum 6 mil polyethylene sheeting. All solids and liquids produced during the procedure should pool in this area until disposed of appropriately.

Step 5 - Place the item(s) to be cleaned on a surface (e.g., plastic/wood pallet) inside the temporary EDA.

D. Cleaning Procedures

Step 1 - Pre-clean the entire piece of equipment to remove all loose dust, dirt, scale, etc. using a metal scraper and/or steel brush, by scraping, chipping, brushing and spot cleaning with solvent or detergent to remove encrusted materials.

Step 2 - Apply the cleaning solution to each surface of the item via a mist, aerosol spray, or plastic brush soaked in the cleaning solution. Make sure that all surfaces are wetted. Use scrubbing brushes, if necessary, to loosen any visible dirt, stains, grease, etc. and then wipe down all surfaces with clean absorbent towels to remove any remaining particles. For larger items, it may be appropriate to clean the equipment in sections.

Step 3 - Rinse the equipment with water supplied from Building 78 or a potable water source on the temporary EDA with a steam/hot water high pressure washer (1,500 psi or greater).

Step 4 - Repeat Steps 2 and 3. The item should be clean and dry. The equipment is ready to be re-used on site. However, if the equipment is leaving the site to be used elsewhere, it must be wipe tested to demonstrate that it meets the applicable conditions for off-site re-use.

Step 5 - Completely cover the equipment with polyethylene sheeting and secure the sheeting if equipment will be left near Building 78.

Step 6 - Use the designated utility pump to pump all liquids from the plastic sheeting in the EDA to a 55-gallon drum, which will be staged at Building 78 prior to disposal.

Step 7- Wrap up the plastic sheeting and dispose of it in a 55-gallon drum with PPE and other disposable items, to be staged at Building 78.

Step 8 - Before leaving the area where a piece of equipment has been cleaned, conduct a final check to make sure all discarded materials, including paper towels, plastic sheeting, disposable gloves, etc., have been picked up and placed in a properly labeled drum.

Step 9 - As employees leave the cleaning area, cover boots and disposable gloves must be left behind. At the end of the day, all reusable PPE must be cleaned and stored on-site. No contaminated PPE or equipment will be permitted to leave the site.

E. Handling and Disposal of Waste Materials

All liquid and solid materials, including spent detergents, rinse waters, disposable clothing, residues from scraping and vacuuming, paper towels, plastic and any other wastes generated during cleaning procedures, shall be collected and stored in DOT-approved drums. All drums shall be properly marked, labeled, stored, and disposed of in accordance with existing site waste management procedures.

Appendix X

Building Material Sampling Procedures

Building Material Sampling Procedures

- Appendix X-1 – Wood Floor Sampling Procedures
- Appendix X-2 – Sheetrock Sampling Procedures
- Appendix X-3 – PCB Wipe Sampling Procedures
- Appendix X-4 – Oil Reservoir Sampling Procedures
- Appendix X-5 – Concrete Floor Sampling Procedures
- Appendix X-6 – Wood Column Sampling Procedures
- Appendix X-7 – Brick Wall Sampling Procedures
- Appendix X-8 – Wood Beam & Joint Sampling Procedures
- Appendix X-9 – Roof Deck Sampling Procedures

Appendix X-1

Wood Floor Sampling Procedures

Wood Floor Sampling Procedures

Proper personal safety equipment shall be worn in accordance with the Site Health and Safety Plan.

Before locating the exact sample location for drilling, check to make sure there are no electrical, plumbing, or any other obstructions beneath the desired sample location.

1. Documentation shall be in accordance with Appendix L.

2. Decontaminate drill bit in accordance with the procedures presented in Appendix W.

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

4. Position paper collection plate with center hole cut out over determined sample location on the floor.

5. Drill 1-inch diameter hole in center of template through floor to the required depth (i.e., single or multiple floor layers).

6. Tap drill bit with wood tool to remove remaining wood debris and collect on collection plate.

7. Collect the wood sample debris in sample container by allowing debris to drop through the center hole into the sample container.

8. Close the sample container.

9. Repeat procedure at nearby locations until sufficient volume has been obtained to complete the sample requirement.

10. Cap and seal the sample container.

11. Remove and dispose of protective gloves in appropriate container.

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

Appendix X-2

Sheetrock Sampling Procedures

Sheetrock Sampling Procedures

Proper personal safety equipment shall be worn in accordance with the *Site Health and Safety Plan* (HASP).

Before locating the exact sample location for drilling, check to make sure there are no electrical, plumbing, or any other obstructions beneath the desired sample location.

1. Documentation shall be in accordance with Appendix L.
2. Decontaminate drill bit in accordance with the procedures presented in Appendix W.
3. Put on clean latex/nitrile protective gloves for each new sample collection.
4. Position paper collection plate with center hole cut out over determined sample location on the sheetrock.
5. Drill 1-inch diameter hole in center of template through sheetrock to the required depth (i.e., single or multiple floor layers).
6. Tap drill bit with sheetrock tool to remove remaining sheetrock debris and collect on collection plate.
7. Collect the sheetrock sample debris in sample container by allowing debris to drop through the center hole into the sample container.
8. Close the sample container.
9. Repeat procedure at nearby locations until sufficient volume has been obtained to complete the sample requirement.
10. Cap and seal the sample container.
11. Remove and dispose of protective gloves in appropriate container.
12. Complete chain-of-custody forms, prepare shipping containers, and send to laboratory for analysis.

Appendix X-3

PCB Wipe Sampling Procedures

PCB Wipe Sampling Procedures

Lockout/Tagout: Before a piece of equipment can be sampled, it must be disconnected and disabled in accordance with standard Energy Control and Power Lock Out Procedures. All energy sources, including electrical, mechanical, and potential, must be de-energized prior to sampling activities.

Proper personal safety equipment shall be worn in accordance with the Site Health and Safety Plan.

1. Documentation shall be in accordance with Appendix L.

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

3. Place paper template with 10-cm x 10-cm cutout on horizontal surface of equipment.

4. Remove hexane-soaked gauze pad from sample vial. Using gauze pad, wipe entire cutout area from side to opposite side. Refold gauze pad and wipe in perpendicular direction of first wipe, side to opposite side of entire cutout area.

5. Place sample gauze pad back into sample vial.

6. Remove protective gloves while holding template and dispose of used gloves and template in appropriate container.

7. Repeat procedure for total number of samples required.

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

Appendix X-4

Oil Reservoir Sampling Procedures

Oil Reservoir Sampling Procedures

Lockout/Tagout: Before a piece of equipment can be sampled, it must be disconnected and disabled in accordance with standard Energy Control and Power Lock Out Procedures. All energy sources, including electrical, mechanical, and potential, must be de-energized prior to sampling activities.

Proper personal safety equipment shall be worn in accordance with the Site Health and Safety Plan.

1. Documentation shall be in accordance with Appendix L.

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

3. Obtain an oil sample from one of the following procedures:

    a. Place a catch pan below work area to collect possible oil drippings. Loosen the drain plug on the oil reservoir. Do not fully remove. Apply pressure on the plug to prevent the plug from falling out. Place the sample vial under the plug to catch oil. Reduce pressure on the plug to allow oil to flow out. If sediment/sludge is noticed, drain until oil clearly flows without the sediment/sludge. Fill the sample vial to top. Reconnect and tighten the drain plug. Cap and seal the sample vial. Wipe and clean any excess oil from the sample vial and equipment.

    b. Place a disposable pipette in the oil fill neck hole to the bottom of the reservoir. Place a finger on top of the pipette to make an airtight seal. Make sure there is no sediment/sludge in the bottom of the pipette. Remove the pipette from the reservoir, place in a sample vial, and release the finger to allow oil to fill the sample vial. Repeat procedure to obtain a full vial oil sample. Cap and seal the sample vial. Wipe clean any excess oil from the sample vial and equipment.

4. Dispose of the pipette, gloves, excess oil, and catch pan in appropriate containers.

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

Appendix X-5

Concrete Floor Sampling Procedures

Concrete Floor Sampling Procedures

Pre-Sampling Procedures:

- Prior to the initiation of sample collection, don proper personal protective equipment (PPE) in accordance with the Site Health and Safety Plan, and wear such equipment throughout sample collection activities.

- Make sure the concrete floor and surrounding area are sound and in good condition before executing any sampling activities.

- Prior to penetrating any surface, verify the locations of utilities. Location of utilities should be verified visually, and include identification of electrical, water, and/or process lines that would interfere with sampling. If based on visual review the potential for underground lines exists, GPR or another form of utility investigation may be implemented.

Equipment List:

- Corded rotary hammer drill

- Masonry coring bit – recommend minimum of two (2) 1-inch bits

- Extension cords and ground fault circuit interrupter (GFCI)

- Paper plates or equivalent

- Dust masks

- Leather or cut resistant work gloves

- Latex/Nitrile gloves

- Sample collection jars – necessary sample volume and appropriate sample containers will be indicated by the laboratory.

Sample Collection Procedure:

1. Documentation shall be in accordance with Appendix L. All information should be properly documented on the sample collection log.

2. While wearing appropriate PPE, decontaminate drill bit in accordance with the protocols presented in Appendix W.

3. Over protective leather or cut resistant gloves, put on clean latex/nitrile protective gloves for each new sample collection.

4. Place a clean dedicated paper template with a 1-inch diameter center hole cut out over determined sample location.

5. Using a rotary hammer drill with a 1" masonry coring bit, drill the full depth of the concrete floor (or as directed by the scope of work). Concrete debris should be uplifted and deposited on paper template.

6. Remove and tap drill bit to remove remaining concrete debris and collect on plate. Repeat procedure at nearby locations until sufficient volume has been obtained to complete the sample requirement.

7. Using a dedicated sample collection plate, mix the collected sample ensuring a homogenous mixture. Deposit sample into sample container(s)

8. Fill, cap or seal sample container. Label appropriately for parameters to be analyzed.

9. Remove and dispose of protective gloves and sample collective plates in appropriate container.

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

Appendix X-6

Wood Column Sampling Procedures

Wood Column Sampling Procedures

Pre-Sampling Procedures:

- Prior to the initiation of sample collection, don proper personal protective equipment (PPE) in accordance with the Site Health and Safety Plan, and wear such equipment throughout sample collection activities.

- Make sure wood component is sound and in good condition before executing any sampling activities.

- Prior to penetrating any surface, verify the locations of utilities. Location of utilities should be verified visually, and include identification of electrical, water, and/or process lines that would interfere with sampling.

Equipment List:

- Corded or cordless drill – if cordless, recommended 18V or greater

- Extension cords and ground fault circuit interrupter (GFCI)

- Paper plates or equivalent

- Leather or cut resistant work gloves

- Latex/Nitrile gloves

- A minimum of two (2) 1-inch paddle drill bits

- Sample collection jars – necessary sample volume and appropriate sample containers will be indicated by the laboratory.

Sample Collection Procedure:

Note: Drill bit should be 1 inch in diameter to ensure proper sample collection and volume needed for analysis.

1. Documentation shall be in accordance with Appendix L. All information should be properly documented on the sample collection log.

2. While wearing the appropriate PPE, decontaminate drill bit and trowel/chisel in accordance with the protocols presented in Appendix W.

3. Over protective leather or cut resistant gloves, put on clean latex/nitrile protective gloves for each new sample collection.

4. Place a clean dedicated collection plate or attach a dedicated plastic sample bag below drilling location on both sides of column to catch wood debris sample.

5. Drill hole through entire width of column, cleaning out the hole frequently as wood chips tend to build up and can inhibit the drill bit.

6. Remove and tap drill bit with wood trowel/chisel to remove remaining wood debris and collect in the sample bag or on plate. Repeat procedure at nearby locations until sufficient volume has been obtained to complete the sample requirement.

7. Mix collected sample ensuring a homogeneous mixture and deposit mixed sample in sample container. Fill cap or seal sample container. Label appropriately for parameters to be analyzed.

8. Remove and dispose of protective gloves and sample collection plate or bag in appropriate container.

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

10. Make repairs to column by filling entire volume of hole with Abatron Wood Epoxy, Evclid Eucopoxy Vertigel, or equivalent.

Appendix X-7

Brick Wall Sampling Procedures

Brick Wall Sampling Procedures

Pre-Sampling Procedures:

- Prior to the initiation of sample collection, don proper personal protective equipment (PPE) in accordance with the Site Health and Safety Plan, and wear such equipment throughout sample collection activities.

- Make sure the brick wall and surrounding area are sound and in good condition before executing any sampling activities.

- Prior to penetrating any surface, verify the locations of utilities. Location of utilities should be verified visually, and include identification of electrical, water, and/or process lines that would interfere with sampling.

Equipment List:

- Corded rotary hammer drill

- Masonry coring bit – recommend minimum of two (2) 1-inch bits

- Extension cords and ground fault circuit interrupter (GFCI)

- Paper plates or equivalent

- Dust masks

- Leather or cut resistant work gloves

- Latex/Nitrile gloves

- Sample collection jars – necessary sample volume and appropriate sample containers will be indicated by the laboratory.

Sample Collection Procedure:

1. Documentation shall be in accordance with Appendix L. All information should be properly documented on the sample collection log.

2. While wearing the appropriate PPE, decontaminate drill bit in accordance with the protocols presented in Appendix W.

3. Over protective leather or cut resistant gloves, put on clean latex/nitrile protective gloves for each new sample collection.

4. Place a clean dedicated collection plate or attach a dedicated plastic sample bag below drilling location to catch brick debris sample.

5. Using a rotary hammer drill equipped with a masonry coring bit, drill into brick wall (total sample depth will be indicated in the scope of work). Brick debris should fall on collection plate or into the plastic sample collection bag. Continue drilling until adequate sample depth has been reached.

6. Remove and tap drill bit to remove remaining brick debris and collect on plate. Repeat procedure at nearby locations until sufficient volume has been obtained to complete the sample requirement.

7. Using a dedicated sample collection plate, mix the collected sample ensuring a homogeneous mixture.

8. Fill, cap or seal sample container. Label appropriately for parameters to be analyzed.

9. Remove and dispose of protective gloves and sample collection plate or bag in appropriate container.

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

Appendix X-8

Wood Beam & Joint Sampling Procedures

Wood Beam and Joist Sampling Procedures

Pre-Sampling Procedures:

- Prior to the initiation of sample collection, don proper personal protective equipment (PPE) in accordance with the Site Health and Safety Plan, and wear such equipment throughout sample collection activities.

- Make sure wood component is sound and in good condition before executing any sampling activities.

- Prior to penetrating any surface, verify the locations of utilities. Location of utilities should be verified visually, and include identification of electrical, water, and/or process lines that would interfere with sampling.

Equipment List:

- Corded or cordless drill – if cordless, recommended 18V or greater

- Extension cords and ground fault circuit interrupter (GFCI)

- Paper plates or equivalent

- Leather or cut resistant work gloves

- Latex/Nitrile gloves

- A minimum of two (2) 1-inch paddle drill bits

- Sample collection jars – necessary sample volume and appropriate sample containers will be indicated by the laboratory.

Sample Collection Procedure:

Note: Drill bit should be 1 inch in diameter to ensure proper sample collection and volume needed for analysis. Do not drill or cut in middle third of the total length or near the bottom of the beam or joist.

1. Documentation shall be in accordance with Appendix L. All information should be properly documented on the sample collection log.

2. While wearing the appropriate PPE, decontaminate drill bit and trowel/chisel in accordance with the protocols presented in Appendix W.

3. Over protective leather or cut resistant gloves, put on clean latex/nitrile protective gloves for each new sample collection.

4. Place a clean dedicated collection plate or attach a dedicated plastic sample bag below drilling location on both sides of beam or joist to catch wood debris sample.

5. Drill hole through entire width of beam or joist, cleaning out the hole frequently as wood chips tend to build up and can inhibit the drill bit.

6. Remove and tap drill bit with wood trowel/chisel to remove remaining wood debris and collect in the sample bag or on plate. Repeat procedure at nearby locations until sufficient volume has been obtained to complete the sample requirement.

7. Mix collected sample ensuring a homogeneous mixture and deposit mixed sample into sample container using the decontaminated trowel. Fill cap or seal sample container. Label appropriately for parameters to be analyzed.

8. Remove and dispose of protective gloves and sample collection plate or bag in appropriate container.

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

10. Make repairs to beam or joist by filling entire volume of hole with Abatron Wood Epoxy, Evclid Eucopoxy Vertigel, or equivalent.

Appendix X-9

Roof Deck Sampling Procedures

Roof Deck Sampling Procedures

This SOP is to be used for the collection of samples from the materials most commonly utilized in the composition of roof decking. Such materials include, but are not limited to: concrete, wood planking, corrugated metal, composite wood paneling, and polystyrene, as well as the applicable coatings such as tar, sealants and paints. Pre-Sampling Procedures:

- Prior to the initiation of sample collection, don proper personal protective equipment (PPE) in accordance with the Site Health and Safety Plan, and wear such equipment throughout sample collection activities.

- Make sure roof component is sound and in good condition before executing any sampling activities. It is recommended that, prior to sampling the roof deck, the structure be inspected by a structural engineer to ensure the safety of field personnel.

- Prior to penetrating any surface, verify the locations of utilities. Location of utilities should be verified visually, and include identification of electrical, water, and/or process lines that would interfere with sampling.

- Coordinate with GE project manager and/or subcontractor to repair roof following execution of sampling procedure (if necessary).

Equipment List:

- Hammer or mallet

- Stainless steel mixing bowl

- Sharpened chisels – minimum of two (2)

- Retractable utility knife with replacement blades

- Leather or cut resistant work gloves

- Latex/Nitrile gloves

- Sample collection jars – necessary sample volume and appropriate containers will be indicated by the laboratory.

Sample Collection Procedure:

1. Documentation shall be in accordance with Appendix L. All information should be properly documented on the sample collection log.

2. While wearing property PPE, decontaminate hammer, chisel, and utility knife blade in accordance with the protocols presented in Appendix W.

3. Over protective leather or cut resistant gloves, put on clean latex/nitrile protective gloves for each new sample collection.

4. Use new clean razor blade (on utility knife) to cut roof membrane and any built-up material (if installed) and expose 4-inch by 4-inch area of decking (usually wood). If roof membrane is not present, then proceed directly to the next procedure.

5. Using the chisel and hammer/mallet for wood and concrete or metal sheers for metal, cut or chip out a 4-inch by 4-inch area from roof decking, including all built-up material; if possible break up the material so it will fit into the applicable sample containers. Mix the removed material in the clean sampling bowl and place the removed materials into the sample container(s).

6. Close, cap, seal sample container, and label appropriately.

7. Remove and dispose of protective gloves in appropriate container.

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

Appendix Y

Selection of Drilling Methods

Selection of Drilling Methods

I. Introduction

This Appendix provides information to be utilized when selecting a drilling method to install soil borings, collect soil samples, collect geotechnical data, and/or install monitoring wells. These differing objectives, combined with the variety of subsurface conditions at different sampling locations, require that judgment be made regarding the drilling methodology to be employed. Drilling may be performed utilizing one or more of the following techniques: hollow-stem auger, direct-push/percussion, driven casing, spun casing, air and mud rotary, and rotosonic or sonic drilling. The appropriate sampling method will be identified prior to the onset of sampling, but may be modified in the field depending on the conditions encountered.

II. Selection of Drilling Method

The specific goal of the drilling program, known subsurface conditions, site accessibility/space restrictions, and type of terrain should be considered prior to selection of the drilling method to be utilized. In addition, cost, installation time, and the ability to recover undisturbed and reliable samples should also be considered. In certain situations, multiple drilling methods may be necessary. The following drilling methods may be utilized for a variety of situations:

- *Hollow-Stem Auger* - The hollow-stem auger method is frequently used to install monitoring wells in unconsolidated materials/soils. The augers rotate as they drill into the ground, evacuating soil along a continuous flight outside of the augers. The system (powered mechanically or hydraulically) uses a cutting head attached to the lead auger to penetrate soils. An auger plug or interior bit may be inserted into the lead auger during advancement to stop any cuttings from coming up into the stem. Samples are collected by driving a split-spoon or pushing a Shelby tube (for clay soils) in front of the auger advancement to obtain undisturbed samples.

  Advantages to this form of drilling include ease of mobilization, relatively fast operation, and monitoring wells (screen and riser) can be installed prior to the removal of the augers, ensuring a good sand pack and bentonite seal, and reducing the possibility of cave-in. In addition, hollow-stem augering does not require that drilling fluids or lubricants be introduced into the subsurface. Disadvantages include difficulty drilling in dense soils or cobbles and the generation of a high volume of waste cuttings. In addition, flowing or water-bearing sands may pose a problem to auger drilling, as these conditions have a tendency to push the saturated sands into the auger stem due to head pressure, which can inhibit soil sampling and may lock up the augers. Overall, the hollow-stem auger is the most commonly used form of drilling for environmental investigations, particularly for geotechnical purposes, due to the ability to obtain blow counts during split-spoon sampling.

- *Direct-Push* - Direct-push drilling involves the advancement of a hollow barrel containing a PVC tubular liner using hydraulics and a hammering mechanism (typically by Geoprobe® or Powerprobe® drill rigs, although sampling can also be performed manually or through the use of a jackhammer) for the collection of soil samples. Direct-push methods may vary slightly depending on the drill rig manufacturer. The hollow barrels or samples are typically 4 feet in length and 1 to 2 inches in diameter, and are advanced and retracted for sample analysis/observation. The disposable PVC liners are removed from the samplers and split to obtain the soil sample. The liners are attached to the inside of the lead barrel by a cutting shoe and are driven continually deeper into the ground using extension rods. Direct-push probing units can also be utilized to collect discrete groundwater samples using a stainless steel screen contained in the outer barrel. The sampler is advanced to the desired depth using an expendable drive point, upon which time the outer sheath is retracted, exposing the screen. Water entering the screen is then sampled using a peristaltic pump, positive displacement tubing with foot valve, or bailer. Shallow, small diameter piezometers/monitoring wells can also be installed by most direct-push drill rigs.

  The primary advantages of direct-push drilling include rapid sampling to shallow and minimal amounts of waste soil generation. Decreased possibility of cross-contamination due to disposable/one-time-use sampling equipment and reduced contamination time also make this form of drilling desirable. The disadvantages to direct-push drilling include the inability to penetrate dense or gravelly material, inability to obtain geotechnical information via blow counts, and the potential introduction of cave-in into the borehole while retracting the sampler. Due to the small diameter of the borehole, standard size monitoring wells cannot be installed in direct-push borings.

- *Spun Casing* - In this technique, a straight casing is advanced by rapid rotation and hydraulic down-force pressure. The lead casing is equipped with a spin shoe/cutter head, enabling it to cut/tear through the unconsolidated soils. Water is typically introduced into the casing stem during advancement to cool the bit and clean out the cuttings from the borehole. For sampling purposes, a split-spoon sampler or Shelby tube is advanced through and in front of the casing in order to obtain undisturbed samples. Similar to hollow-stem augers, spun casing allows the placement of a monitoring well prior to removal from the ground, ensuring the integrity of the borehole and allowing for a good sand pack and seal. The advantage of spinning casing as compared to augering is the ability to penetrate through more dense and cobbly soil without sacrificing borehole integrity. Wells installed through spun casing tend to develop more readily than those installed with augers since the borehole is installed with a straight casing, minimizing disturbance to the remaining soils and the majority of soil cuttings are flushed to the surface inside the casing rather than along the soil wall outside the augers. The main disadvantage of using casing over augers is the addition of water or mud to the boring for cooling and evacuation purposes. In addition to increased generation of waste materials, the use of water may inhibit identification of the water table within soil samples, as well as limit the use of the collected samples for chemical characterization.

- *Driven Casing (Drive and Wash)* - This method is very similar to spun casing except that the casing is advanced by driving the casing either mechanically (typically using a 300-pound hammer) or hydraulic hammering, as opposed to spinning. Instead of a cutting head, the lead casing is equipped with a sharper drive shoe. Because no rotary motion or drilling fluid is applied during casing advancement, soil enters the hollow-stem and is removed through the use of a roller bit and the injection of water into the casing until the soil is cleaned out. Undisturbed split-spoons or Shelby tube samples are collected through and in front of the casing string. This method of drilling enables the collection of blow count data for split-spoon sampling and also during advancement of the outer casing, for use in additional geotechnical applications.

  The advantage of driven casing, as compared to augering, is the ability to install and retrieve the casing through flowing sands. Wells installed through driven casing, like those in spun casing, tend to develop more readily than those installed with augers. Disadvantages include decreased production rates, particularly in cobbly material, as well as issues related to the introduction of water into the borehole and subsequent waste handling.

- *Mud Rotary* - In this method of drilling, boreholes are advanced by rotating a drill pipe by means of a hydraulic powered top head drive, with a bit attached to the bottom of the pipe. The bit cuts and breaks up the material as it penetrates the formations. Drilling fluid or mud is pumped through the rotating drill pipe and through holes in the bit. This fluid swirls in the bottom of the hole, picking up material broken by the bit, then flows upward in the space outside the drill pipe, carrying the cuttings to the surface and clearing the hole. The drill pipe and bit move downward deepening the hole as the operation proceeds. At the surface, drilling mud flows into a tank and the cuttings settle to the bottom. From the settling chamber of the tank, fluid overflows into another chamber from which it is picked up by the suction hose of the mud pump and recirculated through the drill pipe. In the rotary drilling method, the casing pipe is not introduced until after the drilling operations are completed. The walls of the hole are held in place by the pressure of the drilling mud against the sides of the borehole. Split-spoon soil samples may be collected for stratigraphic characterization and geotechnical purposes, but the presence of the drilling mud may preclude the acquisition of useful samples for chemical characterization.

  Advantages of this form of drilling include the ability to advance through dense unconsolidated or cobbly soils, running sands, and bedrock at great depths. A common use of mud rotary drilling is in the installation of cased borings or double-cased monitoring wells. Mud rotary drilling may be utilized to drill through difficult terrain and to set an upper casing. The casing is then cleaned out and drilling proceeds using a different drilling method, as necessary. Disadvantages primarily involve the employment of the drilling mud, which must be properly disposed of and may increase the possibility of cross-contamination between different soil layers, as well as have an impact on well development. Sufficient space is required to place a mud pit or recirculation tub to utilize this drilling method.

- *Air Rotary* - This method is basically the same as mud rotary except that the mud pump is replaced by an air compressor. The air line is connected to a swivel hose at the top of the head drive. Compressed air is forced down through the drilling pipe and out through the holes at the bottom of the rotary drill bit. A small stream of water is often introduced into the air system to help cool the drill bit and control dust. The air serves to cool the drill bit and force cuttings up out of the hole, where they are collected through a cyclone at the top of the hole. Advantages of air rotary over mud include the reduced chance of cross-contamination between soil layers and the reduced amount of waste water generated. However, unlike mud rotary drilling, once the air pressure is turned off, loose formations may cave in against the drill pipe. Therefore, this method is not as useful for installing casings in certain formations.

- *Sonic Drilling* - Sonic drilling (also referred to as vibratory or rotosonic drilling) uses a combination of mechanically-generated vibrations and rotation (typically slow) to penetrate the subsurface material. The drill head consists of two counter-rotating, out of balance rollers (oscillator) that cause the drill pipe to vibrate. Resonance occurs when the frequency of the vibrations equals to the natural frequency of the drill pipe. The resonance and weight of the drill pipe, along with the down force of the drill head, permit penetration of the formation without the additional of drilling mud or lubricating fluids. A dual string assembly allows advancement of an inner casing used to collect core samples while an outer casing maintains borehole integrity. Small amounts of air and water can be used to remove the material between the inner and outer casing. Advantages to this form of drilling include its extremely high drilling rates and low generation of waste cuttings. The possibility of sand bridging during well installations is also minimized due to the vibratory feature of the rig. Disadvantages to this form of drilling are its high cost, use of drilling fluid, and limited availability of this relatively new drilling technology.

- *Coring/Rotary Diamond Drilling* - This method employs industrial diamonds embedded into a spin shoe attached to a core barrel (typically 10 to 26 feet in length). The barrel is spun down through bedrock while water is being added to cool the cutting surface. The bit advances through rock with a solid core remaining inside the tube or core barrel. The bedrock cores are retrieved and inspected in approximate 5-foot lengths. This method is limited to sampling bedrock.

Appendix Z

Monitoring Well Inventory
Procedures

Monitoring Well Inventory Procedures

I. Introduction

This appendix specifies the procedures for performing inventories of existing monitoring wells. Monitoring well inventories are periodically conducted to assess the integrity of existing monitoring wells and to identify the need for repairs, replacement of parts, or replacement of wells that are determined to no longer be usable. A well inventory involves an inspection of the overall condition of the well, comparison of measurable quantities (e.g., riser stickup relative to grade and total depth), general verification of survey coordinates and elevation, and measurement of depth to water in the well.

II. Equipment and Materials

The following materials will be available, as required, during performance of a monitoring well inventory:

- health and safety equipment, as required by the site *Health and Safety Plan* (HASP);
- ruler or tape measure;
- water level indicator and/or interface probe;
- cleaning equipment (as required in Appendix W);
- well construction information; and
- field notebook.

If feasible, a supply of typical replacement parts (e.g., locks, bolts, and well caps) should be available to enable immediate usage as necessary.

III. Procedures

The typical procedure for assessing the integrity of a monitoring well is outlined below.

Step 1 - Prior to mobilizing in the field, obtain a list of monitoring wells to be inventoried and available information concerning their location and physical characteristics.

Step 2 - Identify site and well identification number on a Monitoring Well Integrity Assessment Form (Attachment Z-1). Record all observations on this form, supplemented by notes in the field notebook, if necessary.

Step 3 - Examine the well for the presence of an identification marker. If absent, label well with the appropriate number.

Step 4 - Examine the surface condition of the well. Record the type of well (i.e., flush-mount or above-grade stickup) and the condition of the well and surface seal. Confirm the protective casing is not bent, the PVC casing is not broken or chipped and there is no evidence of frost heaving. Measure the above-grade portion of the well stickup and compare to the known length of the stickup measured during well installation. If the difference between the observed stickup length and the known stickup length is greater than 0.1 foot, the monitoring well location and elevation should be resurveyed.

Step 5 - Unlock and open the well. Record the type (e.g., PVC or stainless steel), dimensions (i.e., casing diameter and stickup relative to grade), condition of the well casing, and type of well cap. If well cap is missing, replace with available parts or record the type of cap required.

Step 6 - Locate the marked measuring point along the top of the well casing. If no mark is visible, add a mark at the highest point of the casing.

Step 7 - Measure the depth to water and total depth of the well following the procedures specified in Appendix Q. For total depth measurements, account for any difference in calibration of the measuring tape on the probe (i.e., distance from part of probe which measures depth to water and the physical bottom of the probe which will measure total depth of the well). Record any obstructions encountered and a description of the feel of the well bottom (i.e., soft due to sediment or hard). Check well for the presence of non-aqueous phase liquid (NAPL) and record observations on the Monitoring Well Integrity Assessment Form (see Section IV below). If NAPL is observed, the appropriate Project Manager should be notified.

Step 8 - Compare all observations concerning the measured dimensions of the well with the listed values. Based on these results, as well as other observations concerning the condition of the well, record any appropriate recommendations on the Monitoring Well Integrity Assessment Form. Perform any recommended maintenance activities which can be accomplished with available equipment.

Step 9 - Remove all equipment from the well. If no additional maintenance activities are to be performed, close the well and collect all personal protective equipment (PPE) and other wastes generated for disposal (See Section V below).

IV. Follow-Up Activities

Depending on the results of the well inventory, several additional activities may be warranted prior to future usage of the well. Typical follow-up activities include replacement of missing parts, removal of sediment from the base of the well, re-surveying of the well, or complete replacement if the well is determined to be unusable. These activities are briefly discussed below.

As stated above, a supply of locks, bolts, and well caps should be available for immediate usage during performance of the well inventories. However, it is not feasible to maintain a supply of all potential replacement parts due to the variety of well types in use. Therefore, a list of required replacement parts will be compiled during the performance of a well inventory event. At the conclusion of the event, the necessary replacement parts for all wells should be obtained and installed.

Sediment accumulation occurs to some degree in all monitoring wells, particularly those which are not pumped on a routine basis. If a sufficient quantity of sediment which may adversely impact future groundwater sampling or NAPL monitoring activities is observed during a well inventory (i.e., a sediment accumulation of greater than 1 foot above the bottom of the well screen), activities should be taken to remove the sediment. These activities will involve the removal of sediment by either pumping or bailing the well, followed by re-measurement of the total depth of the well to confirm a total depth near the reported values. The removed sediment should be inspected for the presence of filter pack materials which may indicate that the well screen has been damaged. If initial efforts are unsuccessful in clearing the sediment accumulations, the well may need to be re-developed (see Appendix S) or replaced.

The measuring points marked on the well risers will be utilized as a base datum in the determination of groundwater elevations. The distance of these markers from the ground surface will be verified against listed values during well inventory activities. Minor variations between listed and measured values may be attributed to an uneven ground surface around the well or to enhancements to the ground surface, such as paving or grading activities that may have been performed since installation of the well. Therefore, such minor variations (i.e., less than 1 inch) will be discounted and existing survey information for the measuring point on the well will be assumed to be accurate. Greater discrepancies may be attributed to damage or modifications to the well, such as cutting or lengthening the well riser. In these situations, the well should be re-surveyed to establish a new datum for future groundwater elevation measurements.

Replacement or decommissioning of a well may be warranted if the well is broken, obstructed, or otherwise compromised. If the well cannot be adequately repaired and is required for future monitoring purposes, a replacement well should be installed if no suitable alternate wells are located in the vicinity.

V. Disposal Methods

Materials generated during well inventory activities, including disposable equipment, will be disposed of in appropriate containers. Containerized waste will be disposed of by GE consistent with its ongoing disposal practices.

Attachment Z-1

Monitoring Well Integrity Assessment Form

MONITORING WELL INTEGRITY ASSESSMENT

Site Name: ______________________________

Well I.D.: ______________________________

Date: ______________________________

(For each item, circle the appropriate response or fill in the blank)

Well I.D. Clearly Marked: YES NO

Well Completion: FLUSH MOUNT ABOVE-GRADE STANDPIPE

Lockable Cover: YES NO DAMAGED (*Describe below*)

Lock Present: YES NO ADDED Key Brand/Number: ______________

Measuring Point Marked: YES NO ADDED

Well Riser Diameter (inches): __________

Well Riser Type: PVC Stainless Steel Other (*Describe*) ________________________

Surface Condition

Cement Intact: YES NO (*Describe below*)

Curb Box/Well Cover Present: YES NO DAMAGED (*Describe below*)

All Bolts Present: YES NO (*Describe below*)

Well Condition

Well Cap: PVC Slip Cap Pressure-fit Cap None

Well Vent: Slot Cut in Riser Vent Hole in Cap None Not Applicable (*Flush Mount Well*)

Reported Well Riser Stickup (feet): _________ (*use negative number if below grade*)

Measured Well Riser Stickup (feet): _________ (*use negative number if below grade*)

Depth to Water (feet from Top of Well Riser): __________ *-or-* DRY

Depth to LNAPL (feet from Top of Well Riser): __________ *-or-* NONE

Depth to DNAPL (feet from Top of Well Riser): __________ *-or-* NONE

Reported Total Depth of Well (feet below grade): ___________

Measured Total Depth of Well (feet below grade): ___________

Well Obstructed: YES NO If yes, list depth in feet from Top of Well Riser: _________

Well Bottom: SOFT (*contains sediment*) FIRM (*no sediment*)

Recommendations

Repair Concrete/Surface Completion: YES NO

Re-Survey Well: YES NO If yes, list date performed: ____________

Remove Sediment and Re-Measure Depth: YES NO If yes, list date performed: ____________

Replace Well Cap: YES NO If yes, list date performed: ____________

Replace Bolts: YES NO If yes, list date performed: ____________

Other/Miscellaneous Observations: ____

____

____

____

____

Inspector(s):______________________________________

Appendix AA

Groundwater Sampling Procedures Using Passive-Diffusion Bags

Groundwater Sampling Procedures Using Passive-Diffusion Bags

I. Introduction

Groundwater samples will be collected from monitoring wells to evaluate groundwater quality. The protocol presented in this appendix describes the procedures to be used to collect groundwater samples from monitoring wells using passive-diffusion bag samplers. Passive-diffusion sampling of groundwater using a semipermeable membrane is a patented technology [U.S. Patent Number 5,804,743 held by Don A. Vroblesky (U.S. Geological Survey) and William T. Hyde (General Electric Company)]. Licensing information can be obtained from the USGS Technology Enterprise Office at 703-648-4450.

Similar to the procedures for low-flow and traditional groundwater sampling from monitoring wells, no well will be sampled until well development has been performed in accordance with the procedures presented in Appendix S to the FSP/QAPP, unless that well has been sampled or developed within the prior one year time period. Groundwater samples will not be collected within a one-week time-period following well development.

Passive-diffusion sampling of groundwater using a semipermeable membrane was initially studied and described by Vroblesky and Hyde (1997). The method is based on the principle that volatile organic compounds (VOCs) in groundwater migrate via molecular diffusion through a semipermeable membrane such as polyethylene until the concentrations on either side of the membrane reach equilibrium. Analyte-free water sealed within a semipermeable passive-diffusion bag serves as the sample medium, which is placed in the open interval of the monitoring well and removed after an equilibration period. Certain types of VOCs (e.g., some ketones) do not equilibrate rapidly enough for practical sampling using passive bags. Passive-diffusion bags (PDB) have been successfully benchmarked, however, for many common VOCs including aromatics and chlorinated ethenes and ethanes. Comparative passive-diffusion sampling for other groups of analytes (e.g., semivolatile organic compounds, pesticides, polychlorinated biphenyls, and inorganics) has not been demonstrated.

With the passive-diffusion sampling method, it is assumed that the water inside the open interval of a monitoring well represents ambient groundwater quality without any purging. This assumption is based on the fact that the wellbore hydraulic conductivity is higher than that of the surrounding formation in almost all geologic settings. Thus, the open interval of the well is assumed to be constantly flushed with ambient groundwater. Vroblesky and Hyde (1997) presented results comparing passive-diffusion groundwater sampling results for VOCs to those obtained using other methods, including submersible pump, bladder pump, and bailer. They concluded that the passive-diffusion sampling results were similar to those obtained using the other purging and sampling methods.

II. Materials

Specific to this activity, the following materials shall be available:

- Site plan, well construction records, prior groundwater sampling records (if available);
- Passive-diffusion bag sampling record (provided as Attachment AA-1)
- Passive-diffusion bag (PDB) sampler, consisting of low-density polyethylene (LDPE) lay-flat tubing and constructed as described below;
- Water level probe;
- Downhole temperature, pH, specific conductivity, ORP, and/or turbidity meters (optional);
- Appropriate water sample containers; and
- Appropriate blanks (trip blank supplied by the laboratory).

III. Procedures

Passive-Diffusion Bag Sampler Construction

Figure AA-1 illustrates a PDB sampler design. PDBs will be constructed in a clean laboratory or office environment by the vendor providing the bags (e.g. Eon Products Inc). Sampler size should be determined by the inner diameter of the well, volume of water needed for analysis, and the availability of saturated screen. If the samplers are not shipped pre-filled with laboratory grade analyte-free water (deionized water), field staff can fill and complete construction of the PDB sampler following the steps outlined below.

Step 1 - Fill PDB with deionized water in accordance with vendor/manufactures instructions, in a clean environment. Take care to completely fill sampler, in an effort to minimize headspace.

Step 2 - Attach a braided nylon rope (tether) to the upper nylon pull-tie and the top of the mesh sleeve using a stainless steel snap hook or nylon cable ties. Determine the depth of the desired sampling interval from the top of the well casing based on well construction data +/- geologic information. The entire length of the sampler should be submerged with the mid-point of the sampler being placed at the mid-point of the saturated screen. Utilizing historical groundwater elevation data for each location, approximate the proper placement of the passive diffusion sampler. With reference to the center of the PDB, measure an appropriate length of nylon rope to place the center of the PDB at the appropriate depth below the top of the well casing. Allowing approximately 3 to 5 extra feet of rope to extend above the top of the well casing (to secure the

rope at the top of the well and allow for field adjustment of PDB sampler after the water level is collected on the day of deployment), cut the rope and tie a reference knot corresponding to the top of the well casing. The reference knot facilitates placing the PDB at the proper depth within the monitoring well. Attach a stainless steel weight using a stainless steel clip or nylon cable tie to the tether below the PDB. Allow enough space between the weight and sampler as to not interfere with deployment, but not so much that the weight will touch the bottom of the well. Take care to decontaminate any used weights prior to attachment to tether.

Step 3 - Place each PDB sampler in a clean, labeled plastic bag, and transport it to the site in a cooler containing wet ice to minimize the formation of headspace inside the passive-diffusion bags.

Passive-Diffusion Bag Sampler Installation

Step 4 - Don appropriate personal protective equipment (as required by the Health and Safety Plan).

Step 5 - Place plastic sheeting around the well.

Step 6 - Clean the non-disposable, down-hole monitoring equipment (e.g., water-level probe, field parameter meters) according to the procedures in Appendix W to the FSP/QAPP. If the field parameters were collected during purging of well, then only water level needs to be recorded prior to deployment of sampler.

Step 7 - Open the well cover while standing upwind of the well. Remove well cap and place it on the plastic sheeting. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in the field log. If the well headspace reading is less than 5 parts per million (ppm), proceed. If the headspace reading is greater than 5 ppm, screen the air within the breathing zone. If the breathing zone reading is less than 5 PID ppm, proceed. If the PID reading in the breathing zone is above 5 ppm, move upwind from well for 5 minutes to allow the volatiles to dissipate, and then repeat the breathing zone test. If the reading is still above 5 ppm, don appropriate respiratory protection in accordance with the requirements of the HASP. Record all PID readings. For wells which are part of the regular weekly or monthly monitoring program and prior PID measurements have not resulted in a breathing zone reading above 5 ppm, PID measurements will be taken semi-annually.

Step 8 - Open well cover and measure the depth to water. Record water level on field log sheet prior to deploying sampler.

Step 9 - Remove the appropriate PDB sampler from the labeled plastic bag. Slowly lower the PDB sampler into the monitoring well until the mid-point of the bag is properly positioned within the saturated mid-point of the screen interval, with the reference knot even with the top of the well casing (make any field adjustments needed to account for the water level on the day of deployment).

Step 10 - Use the remainder of the rope, above the reference knot, to secure the rope to either the steel casing of the well or, for a flush-mounted well, the locking well cap.

Step 11 - Close and lock the well. Record time and date of sampler deployment on field long sheet, prior to departing well location.

Step 12 - Allow an equilibration period of 14 days or more before retrieving the PDB. If necessary, the well may be accessed briefly during the equilibration period (e.g., to obtain fluid water level measurements), provided that the reference knot remains at the top of the well casing throughout the equilibration period.

Passive-Diffusion Bag Sampler Retrieval and Sample Collection

Step 13 - After the equilibration period, unlock and open the monitoring well. Slowly remove the PDB sampler from the monitoring well.

Step 14 - Immediately following retrieval, use a discharge tube (straw-like device provided by vendor) to pierce bag, Allow a small amount of water to purge bucket to flush discharge tube. Fill required sample volume and discharge remaining water into purge bucket. Discharge tubes should be disposed of prior sampling at the next location to avoid cross-contamination.

Step 15 - Complete the sample label according to procedures in Appendix L to the FSP/QAPP, and cover the label with clear packing tape to secure the label onto the container.

Step 16 - Secure with packing material and store at 4°C in an insulated transport container provided by the laboratory.

Step 17 - Record on the field log or bound field book the time sampling procedures were completed, any pertinent observations of the sample (e.g., physical appearance, the presence of, or lack of, odors, sheens, etc.), and the values of field indicator parameters, if measured. PDB samples can be used to obtain an estimate for groundwater temperature. Other field parameters cannot be obtained using PDBs, but can be measured using an appropriate downhole instrument.

Step 18 - Place the PDB sampler (minus the used polyethylene bag) in a clean, new, dedicated, labeled plastic bag for storage until the next sampling event, secure the monitoring well, properly dispose of personal protective equipment (PPE+ and disposable equipment (see Section VI).

Step 19 - Complete the procedures for packaging, shipping, and handling with associated chain-of-custody (FSP/QAPP Appendix L).

IV. Field Quality Control

In addition to the quality control samples to be collected in accordance with Table 4 of the FSP/QAPP, the following quality control procedures should be observed in the field:

- Samples should be collected from monitoring wells in order of increasing concentration (i.e. from wells with the lowest concentrations to those with the highest concentrations), to the extent known.

- All monitoring instrumentation shall be operated in accordance with manufacturer instructions. Instruments should be calibrated at the beginning of each day, and the calibration should be verified at the end of each day.

- If passive-diffusion sampling is being benchmarked versus another groundwater sampling method (e.g., low-flow or traditional purge using a pump or bailer), the other sampling method should be performed on the same day that the PDB is retrieved and sampled at a given well. (This procedure will provide the best practicable comparison between the results of the different sampling methods.)

- Each passive-diffusion sampler (j-plug, nylon tether and stainless steel weight) should be dedicated to a single monitoring well. If a passive-diffusion sampler is being re-used following a period of storage, the length of the rope and position of the reference knot above the mid-point of the passive-diffusion bag should be verified prior to placement in the monitoring well.

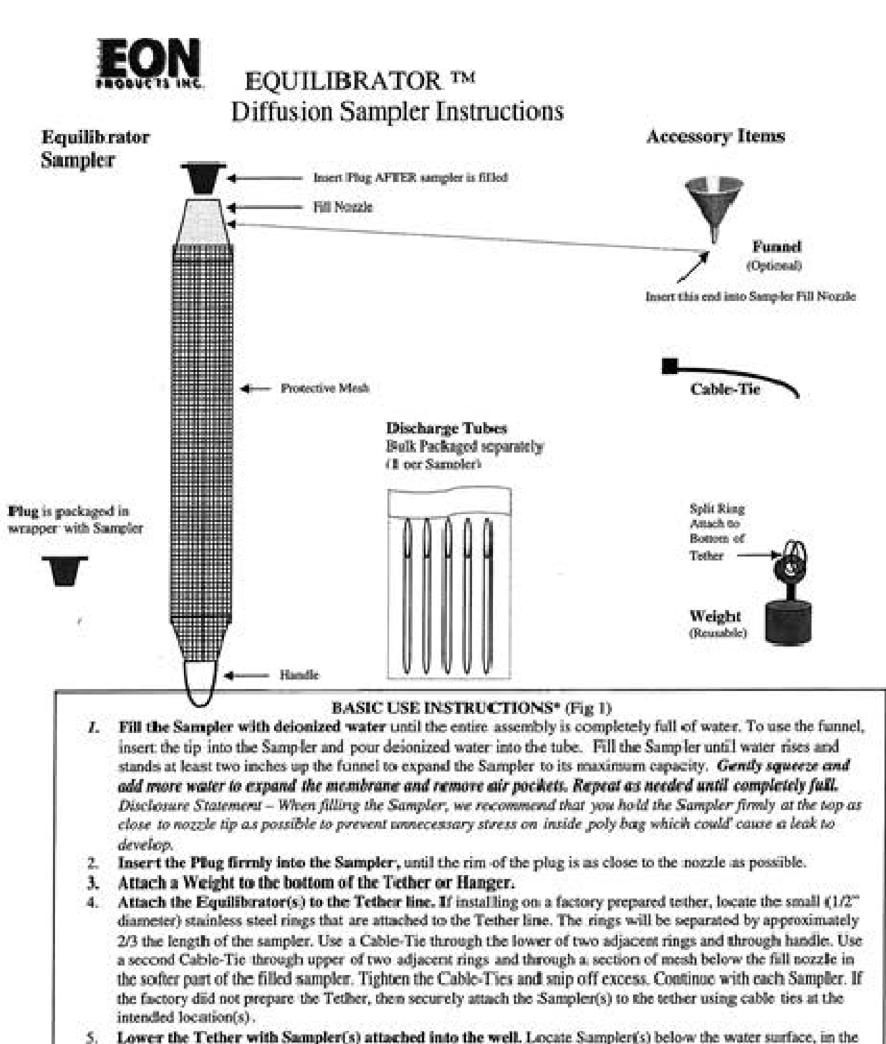
V. Equipment Cleaning

All groundwater monitoring equipment should be cleaned prior to use in the first well and after each subsequent well using procedures presented in Appendix W to the FSP/QAPP.

VI. Material Disposal

Materials generated during groundwater sampling activities, including disposable equipment, will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its ongoing disposal practices.

Figure AA-1. Passive-Diffusion Bag Sampler (*manufacturer's instructions provided by EON Products Inc. or similar)*



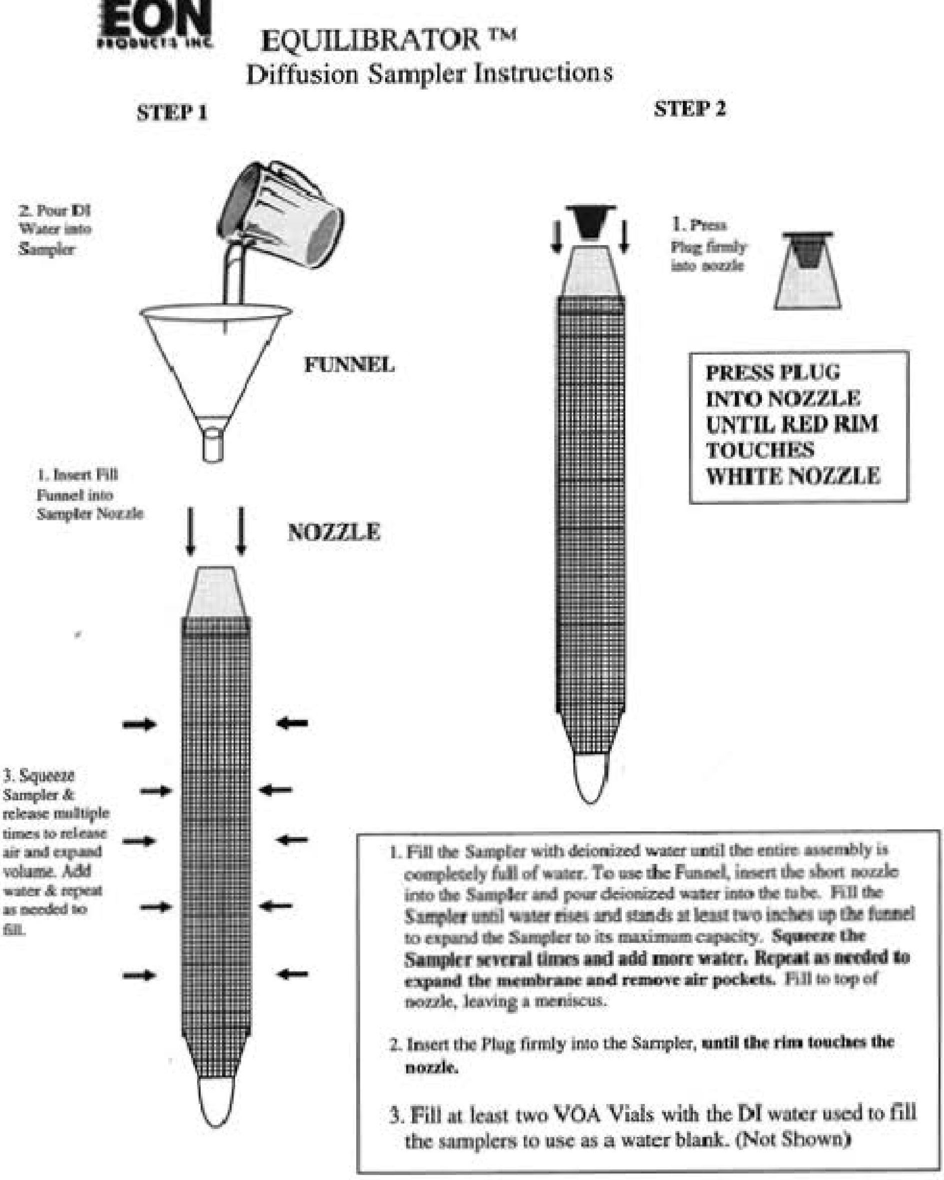
BASIC USE INSTRUCTIONS* (Fig 1)

1. **Fill the Sampler with deionized water** until the entire assembly is completely full of water. To use the funnel, insert the tip into the Sampler and pour deionized water into the tube. Fill the Sampler until water rises and stands at least two inches up the funnel to expand the Sampler to its maximum capacity. ***Gently squeeze and add more water to expand the membrane and remove air pockets. Repeat as needed until completely full.*** *Disclosure Statement – When filling the Sampler, we recommend that you hold the Sampler firmly at the top as close to nozzle tip as possible to prevent unnecessary stress on inside poly bag which could cause a leak to develop.*
2. **Insert the Plug firmly into the Sampler,** until the rim of the plug is as close to the nozzle as possible.
3. **Attach a Weight to the bottom of the Tether or Hanger.**
4. **Attach the Equilibrator(s) to the Tether line.** If installing on a factory prepared tether, locate the small (1/2" diameter) stainless steel rings that are attached to the Tether line. The rings will be separated by approximately 2/3 the length of the sampler. Use a Cable-Tie through the lower of two adjacent rings and through handle. Use a second Cable-Tie through upper of two adjacent rings and through a section of mesh below the fill nozzle in the softer part of the filled sampler. Tighten the Cable-Ties and snip off excess. Continue with each Sampler. If the factory did not prepare the Tether, then securely attach the Sampler(s) to the tether using cable ties at the intended location(s).
5. **Lower the Tether with Sampler(s) attached into the well.** Locate Sampler(s) below the water surface, in the screen flow zone of the well. Attach the top of the suspension cord to a well cap or other secure location at the top of the well. Leave Sampler in place for a time suitable for equilibration, a minimum of 2 weeks required.
6. **Upon retrieval: Discharge sample immediately** to avoid loss of volatile compounds. Select a point on the Sampler near the handle/bottom of sampler. Press one end of the Discharge Tube firmly into the clear polyethylene membrane at a downward angle until it pierces the membrane. ***Discharge small amount to waste to purge discharge tube.***

***Contact EON for detailed installation information and for factory prepared Tethers.**

800-474-2490

Figure AA-1 (cont'd)



Attachment AA-1

Passive Diffusion Bag Sampling Record

Attachment AA-1

GROUNDWATER SAMPLING LOG

Site/GMA Name ____________________

Deployment | Personnel ____________________

Date/Time ____________________

Weather ____________________

____

Well No. ____________________

Key No. ____________________

PID Background (ppm) ____________________

Well Headspace (ppm) ____________________

Sampling Personnel ____________________

Date ____________________

Weather ____________________

WELL INFORMATION

Reference Point Marked? Y N

Height of Reference Point ______ Meas. From ______

Well Diameter ______

Screen Interval Depth ______ Meas. From ______

Water Table Depth ______ Meas. From ______

Well Depth ______ Meas. From ______

Length of Water Column ______

Volume of Water in Well ______

Sample Time ____________________

Sample ID ____________________

Duplicate ID ____________________

MS/MSD ____________________

Split Sample ID ____________________

Required	Analytical Parameters:	Collected
( )	VOCs (Standard List)	( )
( )	VOCs (Expanded List)	( )
( )	SVOCs	( )
( )	PCBs (Unfiltered)	( )
( )	PCBs (Filtered)	( )
( )	Metals/Inorganics (Unfiltered)	( )
( )	Metals/Inorganics (Filtered)	( )
( )	Total Cyanide (Unfiltered)	( )
( )	Total Cyanide (Filtered)	( )
( )	PAC Cyanide (Filtered)	( )
( )	PCDDs/PCDFs	( )
( )	Pesticides/Herbicides	( )
( )	Natural Attenuation	( )
( )	Other (Specify)	( )

Reference Point Identification:

TIC: Top of Inner (PVC) Casing

TOC: Top of Outer (Protective) Casing

Grade/BGS: Ground Surface

Redevelop? Y N

Additional well maintenance needed? Y N (if yes, describe below)

PDB Information

PDB Length/diameter ____________________

PDB Material ____________________

PDBs Filled Lab / Field

Tether Assembled Lab / Field

Line/Tether Material diameter ____________________

Weight Type/Position ____________________

Water Quality Meters Types(s)/Serial Numbers:

____

____

____

PDB Collection				PDB replacement	
Depth	GW Appearance		Number of PDBs at	Depth	Number of
feet (bmp)	Color	Odor	Collection Depth	(ft bmp)	PDBs

Downhole Field Parameters

pH (SU) ______

Specific Conductivity (ms/cm) ______

ORP (mV) ______

Temperature (°C) ______

DO (mg/L) ______

Turbidity ______

OBSERVATIONS/SAMPLING METHOD DEVIATIONS ____________________

____

____

____

SAMPLE DESTINATION

Laboratory: ____________________

Delivered Via: ____________________

Airbill #: ____________________

Field Sampling Coordinator: ____________________

Appendix BB

Soil/Water Shake Test Procedures

Soil/Water Shake Test Procedures

I. Introduction

Soil samples are classified upon collection in the field based on a description of the soil characteristics and indications of contamination, such as the presence of non-aqueous phase liquid (NAPL) within a soil sample. The presence of minor quantities and/or different types of NAPL may not be visually evident on certain soil samples due to characteristics such as color, texture, and/or moisture content. In these situations, a soil/water shake test may be used to supplement other visual observations to possibly identify the presence of NAPL. It should be noted that the goal of this testing is to simply identify the presence of NAPL, not to determine its characteristics. For example, small quantities of dense non-aqueous phase liquid (DNAPL), which may separate from the soil and float on the water surface due to surface tension following performance of the soil/water shake test, should not be interpreted as light non-aqueous phase liquid (LNAPL). Laboratory density testing or observation of a NAPL layer within a monitoring well is required to definitively determine whether separate phase LNAPL or DNAPL are present.

II. Materials

The following materials will be available, as required, during soil sampling in areas where the presence of NAPL is suspected:

- Health and safety equipment (as required by the Site Health and Safety Plan);
- Clear glass soil sample jars;
- Potable or distilled water; and
- Field notebook.

III. Procedures

The detailed procedures for conducting a soil/water shake test on a soil sample are outlined below:

1. Half-fill a clean, clear glass jar with a representative portion of the soil sample. Label the boring identification and depth interval on the jar. If photoionization detector (PID) field screening is also being conducted, the samples utilized for PID screening may be utilized following headspace measurements with the PID.

2. Place a sufficient quantity of water into the sample jar to submerge the soil sample. Agitate the sample and set the jar on a stable surface and allow to settle. Depending on the nature of the soil sample, the settling time may range from several minutes to several hours.

3. Examine the saturated soil sample for evidence of NAPL or sheens on the water surface and within the settled soils.

4. Record all observations in the field notebook. Repeat the test if necessary.

Appendix CC

Basement Sump Sediment/Water Sampling Procedures

Basement Sump Sediment/Water Sampling Procedures

I. Introduction

This Appendix specifies the procedures for collecting sediment and water grab samples from basement sumps for chemical analysis. The wide variety of conditions existing at different sample locations requires that judgment be made regarding methodology and procedure for collection of representative samples. Sediment samples may be collected utilizing one or more of the following pieces of equipment: grab sampler (consisting of a wide-mouth container attached to a telescoping pole), hand-held dredge, peristaltic pump (equipped with silicone and Teflon® tubing), Lexan® tubing (with vacuum pump), hand bucket auger, or other appropriate device. Water samples may be collected utilizing a surface water grab sampler or peristaltic pump. The appropriate sampling method will be field determined at the time of sampling and will depend on the conditions encountered.

II. Equipment and Materials

The following materials will be available, as required, during sediment/water grab sampling:

- Health and safety equipment (as required by the Site Health and Safety Plan);
- Field notebook;
- Appropriate sampling containers and forms;
- Appropriate preservatives, as required;
- Cooler with ice or “blue ice;” and
- Appropriate sampling equipment.

III. Sampling Procedures

The typical procedure for assessing the integrity of a monitoring well is outlined below.

1. Identify grab sample location in the field notebook. Record the condition of the sump, including the presence and description of any standing water, drain lines connected to the sump, sump pump, etc.

2. Photograph the basement sump and draw a sketch, including dimensions, in the field notebook.

3. Don health and safety equipment (as required by the Health and Safety Plan).

4. Clean the sampling equipment in accordance with the procedures in Appendix W of the FSP/QAPP.

5. Collect sample with the appropriate field determined methodology.

6. If standing water is present, collect a sample with the appropriate field determined technology.

7. Transfer the sample(s) from the collection device to the appropriate sample container(s).

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

9. Label all sample containers as appropriate, as discussed in Appendix L of the FAP/QAPP.

10. Handle, pack, and ship the samples in accordance with the procedures in Appendix L of the FSP/QAPP.

IV. Equipment Cleaning

Equipment cleaning will occur at the beginning of each sampling event and between each sampling location, as described in Appendix W of the FSP/QAPP.

V. Disposal Methods

Rinse water, personal protective equipment, and other residuals generated during the equipment cleaning procedures will be placed in appropriate containers.

Appendix DD

Manhole/Catch Basin/Pipeline
Outfall Sediment/Water/NAPL
Sampling Procedures

Manhole/Catch Basin/Pipeline Outfall Sediment/Water/NAPL Sampling Procedures

I. Introduction

This Appendix specifies the procedures for collecting sediment, water, and non-aqueous phase liquid (NAPL) samples from manholes, catch basins, and pipeline outfalls for chemical analysis. The following procedures are to be utilized for remote sampling of manholes, catch basins, or similar pipeline features. These features may be considered confined spaces and should not be physically entered without proper authorization.

The wide variety of conditions that may exist at different sampling locations requires that judgment be made regarding methodology and procedure for collection of representative samples. Sediment samples may be collected utilizing one or more of the following pieces of equipment: grab sampler (consisting of a wide-mouth container attached to a telescoping pole), hand-held dredge, Lexan® tubing (with vacuum pump), hand bucket auger, or other appropriate sampling device. Water and NAPL samples may be collected utilizing a surface water grab sampler, bailer, submersible pump, or peristaltic pump. The appropriate sampling method will be field determined at the time of sampling and will depend on the conditions encountered.

II. Equipment and Materials

The following materials will be available, as required, during sediment/water/NAPL grab sampling:

- Health and safety equipment (as required by the *Site Health and Safety Plan* [HASP]);
- Field notebook;
- Appropriate access equipment;
- Appropriate sampling containers and forms;
- Appropriate preservatives, as required;
- Cooler with ice or "blue ice;" and
- Appropriate sampling equipment.

III. Procedures

1. Identify grab sample location in the field notebook. Record weather conditions (dry or wet period) in field notebook. Record the condition of the manhole or catch basin, including the presence and description of any covers, standing water, flow observations, depth to water, depth to bottom (using probe), depth/orientation of drain lines, staining or sheen observations, and PID readings.

2. Draw a sketch of the manhole or catch basin, including dimensions, in the field notebook.

3. Utilize health and safety equipment (as required by the HASP).

4. Open manhole or catch basin. If necessary, mark area with traffic cones and/or flagging tape.

5. Clean the sampling equipment in accordance with the procedures in Appendix W of the *Field Sampling Plan/Quality Assurance Project Plan* (FSP/QAPP).

6. Collect sample with the appropriate field determined methodology. The following are potential options for sample collection methods:

   - Sediment sample collection;

     - grab sampling with a wide-mouth container attached to a telescoping pole,
     - using a hand-held dredge,
     - pumping with vacuum pump and Lexan® tubing and,
     - using a hand bucket auger

   - Water and NAPL sample collection;

     - surface water grab sampling,
     - using a bailer,
     - pumping with a submersible pump and,
     - pumping with a peristaltic pump

7. Transfer the sample(s) from the collection device to the appropriate sample container(s).

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

9. Label all sample containers as appropriate, as discussed in Appendix L of the FSP/QAPP.

10. Close the manhole cover or catch basin grate and secure the area.

11. Handle, pack, and ship the samples in accordance with the procedures in Appendix L of the FSP/QAPP.

IV. Equipment Cleaning

Equipment cleaning will occur at the beginning of each sampling event and between each sampling location, as described in Appendix W of the FSP/QAPP.

V. Disposal Methods

Rinse water, personal protective equipment, and other residuals generated during the equipment cleaning procedures will be placed in appropriate containers.

Appendix EE

Electromagnetic Survey Procedures

Electromagnetic Survey Procedures

I. Introduction

The following methodologies will be utilized to perform electromagnetic surveys.

II. Selection of Electromagnetic Survey Equipment

The specific goal of the electromagnetic (EM) survey and the type of terrain over which it will be conducted should be considered prior to selection of the instrument to be utilized. In certain situations, multiple types of apparatus may be necessary. The following EM survey instruments or their equivalents may be utilized for a variety of situations:

- Geonics EM-31 Terrain Conductivity Meter – This device contains transmitter and receiver coils in either end of a 13-foot-long boom. The fixed inter-coil spacing allows the EM-31 to detect lateral variations in electrical conductivity while the instrument is carried along transects that are perpendicular to the centerline of the survey area. During the performance of any supplemental EM-31 geophysical survey, two components will be recorded: 1) EM quadrature phase conductivity values that respond to metallic and non-metallic sources of elevated conductivity; and 2) EM in-phase values that respond primarily to metallic objects. The effective depth of exploration of the EM-31 is approximately 15 to 18 feet. This multi-component measurement feature allows the EM-31 to be used for a variety of applications, such as identifying conductive groundwater contaminant plumes or delineating buried metallic objects. The instrument can be operated over rugged terrain or within brushy areas, but nearby cultural interferences such as fences, buildings, and power lines may impact the quality of the data.

- Geonics EM-34 Terrain Conductivity Meter – Similar to the EM-31, the EM-34 records EM quadrature phase conductivity values that respond to metallic and non-metallic sources of elevated conductivity. This device contains transmitter and receiver coils connected by cables that are supplied in 10-, 20-, or 40-meter lengths. The variable inter-coil spacing allows the EM-34 to detect variations in electrical conductivity at greater depths (over 100 feet) than obtainable with the EM-31.

- Geonics EM-61 Time Domain Metal Detector – This device contains vertically-aligned, one meter by one meter transmitter and receiver coils separated by 40 centimeters. The transmitter generates a pulsed primary magnetic field in the earth, which induces eddy current in nearby metallic objects and a secondary magnetic field that is measured by the receiver coil. The system can be trailer-mounted and pulled over the survey area, with readings collected approximately every 0.6 feet. The instrument can also be carried utilizing a shoulder harness and set to record up to three readings per second. The effective depth of exploration of the EM-61 is up to approximately 15 feet. The EM-61 is particularly suited to identify buried metallic objects due to the ease of data collection and interpretation and its relative insensitivity to nearby cultural interferences such as fences, buildings, and power lines. However, a generally flat and clear area is required to perform the survey.

III. Materials

The following materials will be available, as required, during electromagnetic surveys:

- Health and safety equipment (as required by the Site Health and Safety Plan);
- Geonics EM-31 terrain conductivity meter, Geonics EM-34 terrain conductivity meter, Geonics EM-61 time domain metal detector, or equivalent;
- Omnidata Polycorder Datalogger, Juniper Systems Datalogger, or equivalent (optional with EM-31 or EM-34, required with EM-61);
- Trimble AG-132 Global Positioning System (GPS), or equivalent (optional with EM-31, EM-34, and EM-61);
- Measuring tapes (100- to 300-foot lengths, as needed);
- Survey stakes, marking paint, traffic cones, or other visual marker aids; and
- Appropriate forms/field notebook.

IV. Procedures

1. Identify the traverse location on the appropriate form or in the field notebook, along with other appropriate information.
2. Don personal protective equipment (as required by the Health and Safety Plan).
3. Establish a grid system by standard surveying techniques to document the location of each grid point or column. The grid spacing should be sufficient to detail the site location, boundaries, and survey targets. Since target objects will be identified by an observed variation in ground conductivity, it is important to extend the survey grid into presumed “neutral areas”, if possible, in order to delineate the boundaries where such variations occur.
4. Calibrate the EM instrument in accordance with the manufacture’s operating manual and record values in field book. Note any abnormalities in the calibration of the instrument in the field book.
5. Utilizing the selected calibrated electromagnetic instrument, conduct the survey. Operate the instrument in accordance with the manufacturer’s operating manual.
6. Record all observations in the field notebook and/or electronic data logger. In particular, note the location of any observed metallic surface debris, site features that would effect EM measurements (e.g., utilities), and changes in terrain conditions (e.g., proceeding from paved to unpaved areas) to aid in the interpretation of any apparent anomalies in the data.

7. Review the raw data for any anomalous readings. If such readings were recorded, examine the location for any apparent source of the data, such as metallic items on the ground surface, nearby fencing, or power lines. If necessary, collect additional data in the vicinity of the anomaly at reduced grid spacing, or extend the survey area to assess anomalies that are located at or near the edge of the established survey grid.

8. Download the digital field data from the data logger to a computer using the appropriate software supplied by the manufacture. Data should be downloaded on a daily basis, if possible, and checked for errors and/or omissions.

9. Upon completion of the electromagnetic survey, plot the data set utilizing appropriate contouring software (e.g., Golden Software, Inc. – Surfer, or equivalent).

Appendix FF

Test Pit Excavation Procedures

Test Pit Excavation Procedures

Test pits will be excavated using a decontaminated, rubber-tired backhoe. Test pits may be utilized to identify subsurface structures, to facilitate the collection of soil samples that cannot be collected by soil borings, and/or to characterize subsurface conditions for the design or implementation of response actions. Personnel should stand upwind of the excavation area to the extent possible. Continuous air monitoring will be conducted as indicated in the *Site Health and Safety Plan* (HASP). Excavation will be conducted at the selected locations that have been cleared for utilities until the target depth, groundwater, or bedrock is encountered, or to within the physical limits of the backhoe. Test pit materials and samples will be visually observed and described with respect to depth and location. Photographs of the excavation and of the removed soil will be taken and referenced by location and direction for future use. In addition, results of soil head space screening will be recorded. Field activities and observations will be logged in a bound field logbook or on a test pit log form, including a plan view of the test pit and cross-sections of the excavation walls, where appropriate. Where necessary to characterize subsurface soil conditions, soil will be collected in one of two ways. If the excavation is less than 3 feet deep, the sample may be collected directly from the sidewall of the test pit with a decontaminated stainless steel shovel, scoop, or hand auger. If the test pit is deeper than 3 feet, the soil sample will be collected from the backhoe bucket, either directly or with a decontaminated stainless steel scoop or trowel. Samples should be homogenized, if appropriate. Samples collected for VOC analysis will be collected following the procedures in Appendix A.

Material removed from the test pit during excavation will generally be placed on polyethylene sheeting. If such material has been previously characterized for chemical constituents *in situ,* its subsequent disposition (e.g., replacement in the test pit, consolidation at GE's On-Plant Consolidation Areas, off-site disposal) will be based on the results of that sampling, in accordance with applicable requirements. If the material has not previously been chemically characterized, it will be so characterized *ex situ* as necessary to determine appropriate disposition, and its disposition will be based on those characterization results, in accordance with applicable requirements.

To facilitate surveying, the location of the pit will be marked with stakes after it has been backfilled. Stakes should be placed at the ends of the test pit and at any significant bend or corner, as appropriate.

Appendix GG

Monitoring Well Decommissioning Procedures

Monitoring Well Decommissioning Procedures

I. Introduction

This Standard Operating Procedure (SOP) describes the procedures for the decommissioning of groundwater monitoring wells. Monitoring wells may be decommissioned when it is found that they are no longer suitable for collection of groundwater data (i.e., groundwater quality or groundwater elevation) due to damaged and/or questionable construction, when they must be removed to avoid interference to/from other construction activities in the area, or when groundwater monitoring is no longer required at the location. Such wells will be permanently decommissioned in accordance with procedures described in Section 4.6 of the *Massachusetts Department of Environmental Protection Standard References for Monitoring Wells*. The purpose for decommissioning monitoring wells no longer in use is to:

- Eliminate physical hazards associated with an out-of-use monitoring well;
- Conserve the yield and hydrostatic head of confining aquifers;
- Prevent the intermingling of separate aquifers; and
- Remove a potential conduit for the vertical migration of constituents in groundwater along the well casing.

This SOP covers the decommissioning of single-cased overburden monitoring wells when a replacement well will not be installed within the same borehole. Three potential decommissioning methods (i.e., plugging in place, casing removal, and overdrilling) are described below. The well decommissioning procedures described below will be carefully adhered to and will be conducted under the supervision of an experienced geologist, engineer or other qualified individual. If the overdrilling decommissioning method is utilized, drilling activities will be conducted by a registered Massachusetts well driller.

Although these procedures are generally applicable for the decommissioning of double-cased monitoring wells or wells installed within bedrock, in most cases a decommissioning strategy should be developed on a well-by well basis. Additional information regarding potential methods to decommission these types of wells may be found in the *Massachusetts Department of Environmental Protection Standard References for Monitoring Wells*, or in ASTM D5299-92, *Standard Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities*.

II. Equipment and Materials

The following materials, as required, shall be available during pre-decommissioning and decommissioning activities:

- *Site Health and Safety Plan* (HASP);
- Health and safety equipment, as required in the HASP (e.g., air monitoring equipment, personal protective equipment);
- Information concerning the construction of the well to be decommissioned;
- Appropriate field forms or field notebook;
- Well keys;
- Water level probe;
- Cleaning materials (as required in Appendix W to the *Field Sampling Plan/Quality Assurance Project Plan* [FSP/QAPP]);
- Drill rig with Massachusetts registered well driller and experienced personnel, if overdrilling method is utilized;
- Containers for collecting spoils; and
- Any necessary specialized well drilling/decommissioning equipment.

III. Calculation/Verification of Volumes

To assure that a well is properly plugged and that there has been no bridging of the plugging materials, verification calculations and measurements are required to determine whether the volume of material placed in the well/borehole equals or exceeds the volume of the void that is being filled. Some useful formulas for calculating well and material volumes are provided below.

- *7.481 gallons = 1 cubic foot*
- *202.0 gallons = 1 cubic yard*
- *Volume of well/borehole* ***(in gallons) = π TIMES*** *well/borehole radius* ***(in feet)*** *squared* ***TIMES*** *length of well/borehole* ***(in feet) TIMES*** *7.481* ***(gallons per cubic foot)***

IV. Monitoring Well Decommissioning Procedures – Plug-in-Place Method

The plug-in-place method is applicable at locations where available information indicates that the annular space contains an adequate seal and vertical migration of constituents across a confining layer is not a concern in the well casing and screen interval, or if other considerations (e.g., double-cased well construction) preclude removal of the well casing. The well screen is left in place and may be additionally perforated, along with the base of the well, to allow the grout seal to penetrate the surrounding filter pack. The decommissioning process will consist of the following steps:

Step 1 - Perform a search of available records concerning the well to be decommissioned. The following activities should be performed to identify the location, construction, and condition of the well, and to determine the appropriate equipment to utilize based on the depth, diameter, and access to the monitoring well:

- Review the existing monitoring well log to identify construction characteristics (e.g., total depth, casing diameter, initial borehole diameter, type of casing, type of material(s) used);
- Locate the monitoring well in the field;
- Identify if the decommissioning equipment can access the monitoring well and/or if special considerations (e.g., construction of an access road) are necessary to gain access;
- Conduct total depth measurements and water level measurements;
- Calculate volume of well that will need to be filled utilizing field measurements and formulas provided above; and
- Record all observations and measurements.

Step 2 - Remove the protective casing and well casing to a depth of approximately 3 to 4 feet below grade, if possible;

Step 3 - Perforate the base of the well screen, utilizing a length of drilling rod or other equipment;

Step 4 - Prepare a neat cement grout (Note: A neat cement grout is preferred for application through an in-place well, whereas a bentonite/cement grout or bentonite pellets may also be considered at locations where the well casing is removed or the well is overdrilled.);

Step 5 - Place the cement grout in the perforated well casing via tremie method (i.e., the grout will be pumped from the bottom of the well upward). The grout will be added until the well is filled to above the top of the well casing remaining in place (i.e., typically approximately 3 to 4 feet below ground surface). Verify that amount of grout added equals or exceeds the calculated volume of the void to be filled.

Step 6 - The grout will be allowed to set for a minimum of 24 hours and the remainder of the borehole will be filled with concrete and/or other surface finish materials (see Step 7 below);

Step 7 - Where appropriate, a concrete surface finish will be installed by constructing an above grade concrete slab a minimum of 6 inches thick, with a diameter at least 2 feet greater than the diameter of the borehole. If such a concrete surface finish is not compatible with the existing land use (e.g., roadway, parking lot, residential, etc.) the borehole shall be terminated with a minimum 1-foot thick concrete plug above the grout and the remaining portion of the borehole shall be filled flush with grade with material(s) compatible with the surrounding land surface (e.g., asphalt, gravel, topsoil, etc.).

Step 8 - An Overburden Well Decommissioning Record will be completed and submitted to EPA and the Massachusetts Department of Environmental Protection (MDEP). An example of this form is provided as Attachment GG-1.

V. Monitoring Well Decommissioning Procedures – Casing Removal Method

The casing removal method is applicable at shallow locations where vertical migration of constituents across a confining layer is not a concern and where the integrity of the borehole is reasonably expected to be maintained following removal of the well materials. The decommissioning process will consist of the following steps:

Step 1 - Perform a search of available records concerning the well to be decommissioned. The following activities should be performed to identify the location, construction, and condition of the well, and to determine the appropriate equipment to utilize based on the depth, diameter, and access to the monitoring well:

- Review the existing monitoring well log to identify construction characteristics (e.g., total depth, casing diameter, initial borehole diameter, type of casing, type of material(s) used);
- Locate the monitoring well in the field;
- Identify if the decommissioning equipment can access the monitoring well and/or if special considerations (e.g., construction of an access road) are necessary to gain access;
- Conduct total depth measurements and water level measurements;
- Calculate volume of well that will need to be filled utilizing field measurements and formulas provided above; and
- Record all observations and measurements.

Step 2 - Remove the protective casing, if possible;

Step 3 - Remove the well casing (riser and screen);

Step 4 - Examine removed well casing to ensure that the entire section has been removed. Also ensure that borehole has not collapsed and that tremie pipe will be able to be inserted to the base of well depth. Well decommissioning should be completed by using the overdrilling method if the well casing is broken below grade and cannot be retrieved, or if the tremie pipe will not reach the base of the well.

Step 5 - Prepare a neat cement grout, or a bentonite/cement grout that is compatible with the soil and groundwater conditions present at the monitoring well (Note: A neat cement grout or a bentonite/cement grout is preferred for this application. Bentonite pellets may also be considered if the entire well boring is overdrilled, similar to procedures used to abandon boreholes.);

Step 6 - Place the cement grout in the borehole via tremie method (i.e., the grout will be pumped from the bottom of the borehole upward). The grout will be added until the borehole is filled to approximately 3 to 4 feet below ground surface. Verify that amount of grout added equals or exceeds the calculated volume of the void to be filled.

Step 7 - The grout will be allowed to set for a minimum of 24 hours and the remainder of the borehole will be filled with concrete and/or other surface finish materials (see Step 8 below);

Step 8 - Where appropriate, a concrete surface finish will be installed by constructing an above grade concrete slab a minimum of 6 inches thick, with a diameter at least 2 feet greater than the diameter of the borehole. If such a concrete surface finish is not compatible with the existing land use (e.g., roadway, parking lot, residential, etc.) the borehole shall be terminated with a minimum 1-foot thick concrete plug above the grout and the remaining portion of the borehole shall be filled flush with grade with material(s) compatible with the surrounding land surface (e.g., asphalt, gravel, topsoil, etc.).

Step 9 - An Overburden Well Decommissioning Record will be completed and submitted to EPA and MDEP. An example of this form is provided as Attachment GG-1.

VI. Monitoring Well Decommissioning Procedures – Overdrilling Method

The over-drilling method is the most conservative decommissioning procedure, and should be utilized at locations where a well has penetrated a confining layer and there is no evidence that the annular space around the well casing was adequately sealed, or if attempts to remove the well casing are unsuccessful. The decommissioning process will consist of the following steps:

Step 1 - Perform a search of available records concerning the well to be decommissioned. The following activities should be performed to identify the location, construction, and condition of the well, and to determine the appropriate equipment to utilize based on the depth, diameter, and access to the monitoring well:

- Review the existing monitoring well log to identify construction characteristics (e.g., total depth, casing diameter, initial borehole diameter, type of casing, type of material(s) used);
- Locate the monitoring well in the field;
- Identify if a drill rig can access the monitoring well and/or if special considerations (e.g., construction of an access road) are necessary to gain access;
- Conduct total depth measurements and water level measurements;
- Calculate volume of well/borehole that will need to be filled utilizing field measurements and formulas provided above; and
- Record all observations and measurements.

Step 2 - Remove the protective casing, if possible;

Step 3 - If the protective casing has been removed, advance a hollow stem auger or other drill casing - with an outside diameter larger than the well diameter - over the well casing to the bottom of the original borehole;

Step 4 - Prepare a neat cement grout, or a bentonite/cement grout that is compatible with the soil and groundwater conditions present at the monitoring well. Alternatively, bentonite pellets may be used plug the borehole, similar to procedures used to abandon boreholes.

Step 5 - Place the cement grout in the borehole via tremie method (i.e., the grout will be pumped from the bottom of the borehole upward) at the same time the hollow-stem augers or drill casing are removed from the borehole. The grout will be added until the borehole is filled to approximately 3 to 4 feet below ground surface. Verify that amount of grout added equals or exceeds the calculated volume of the void to be filled. If bentonite pellets are utilized, measure deposition depth with a weighted tape as the hollow-stem augers or drill casing are removed from the borehole to ensure that bridging does not occur. At certain shallow well locations installed in competent formations, it may be possible to remove the hollow-stem augers or drill casing prior to installing the sealant. If this is attempted, confirmatory measurements must be taken to verify that borehole integrity was maintained prior to plugging the hole.

Step 6 - The grout will be allowed to set for a minimum of 24 hours and the remainder of the borehole will be filled with concrete and/or other surface finish materials (see Step 7 below);

Step 7 - Where appropriate, a concrete surface seal will be installed by constructing an above grade concrete slab a minimum of 6 inches thick, with a diameter at least 2 feet greater than the diameter of the borehole. If such a concrete surface seal is not compatible with the existing land use (e.g., roadway, parking lot, residential, etc.) the borehole shall be terminated with a minimum 1-foot thick concrete plug above the grout and the remaining portion of the borehole shall be filled flush with grade with material(s) compatible with the surrounding land surface (e.g., asphalt, gravel, topsoil, etc.).

Step 8 - An Overburden Well Decommissioning Record will be completed and submitted to EPA and MDEP. An example of this form is provided as Attachment GG-1.

Attachment GG-1

Overburden Well
Decommissioning Record

ATTACHMENT GG-1

ARCADIS

OVERBURDEN WELL DECOMMISSIONING RECORD

Well I.D.:	
Project:	Start Date:
	Finish Date:
Location:	Drilling Company & Driller:
Client:	Inspector:

Well Schematic & Construction Materials (not to scale)

Outer Casing

Steel ☐ Diameter (in) ______
Stainless Steel ☐ Length (ft) ______
PVC/HDPE ☐

Original Borehole

Diameter (in) ______
Total Depth (ft) ______

Surface Seal

Concrete ☐
Cement Grout ☐
Bentonite Grout ☐
Soil ☐
Other ☐ ______

Filter Pack Seal

Bentonite Pellets ☐
Bentonite Grout ☐
Cement Grout ☐
Other ☐ ______

Filter Pack

Sand ☐
Gravel ☐
Soil ☐
Other ☐ ______

Well Casing

Steel ☐
Stainless Steel ☐
PVC/HDPE ☐
Diameter (in) ______

Well Screen

Steel ☐
Stainless Steel ☐
PVC/HDPE ☐
Diameter (in) ______

Decommissioning Information

Casing Removal

Method Employed: ______

Grouting

Calculated Borehole Volume: ______

Cement Type: ______

Cement Quantity (lbs): ______

Bentonite Quantity (lbs): ______

Water Quantity (gal.): ______

Actual Grout Volume: ______

Surface Finish: ______

Comments: ______

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Appx GG - Atta GG-1.xls

Appendix HH

Determination of Total Organic Carbon (TOC) in Solid Samples

Determination of Total Organic Carbon (TOC) in Solid Samples

I. Introduction

This Appendix specifies the procedures for determining the Total Organic Carbon (TOC) content in soils and sediments. The Lloyd Kahn Method ("*Determination of Total Organic Carbon in Sediment*," Lloyd Kahn, USEPA Region II, Edison, NJ), as incorporated in a Standard Operating Procedure (SOP) provided by Pace Analytical Services, will be utilized. A copy of that SOP is provided as Attachment HH-1.

Attachment HH-1

SOP – Preparation of Analysis of Solid Samples for TOC in Triplicate by a Modified Lloyd Kahn Method (Provided by Pace Analytical)

STANDARD OPERATING PROCEDURE

PREPARATION & ANALYSIS OF SOLID SAMPLES FOR TOTAL ORGANIC CARBON IN TRIPLICATE BY A MODIFIED LLOYD KAHN METHOD

Reference Methods: EPA METHOD LLOYD KAHN (MODIFIED)

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Christina L. Braidwood Quality Manager	Date

PERIODIC REVIEW

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

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1.0 IDENTIFICATION OF TEST METHOD

1.1 This test method is to determine the average total organic carbon content by performing multiple replicates of a sample by high-temperature combustion.

2.0 APPLICABLE MATRIX AND MATRICES

2.1 This test method is applicable to aqueous, soil, sludge, sand, and other solid samples.

3.0 DETECTION LIMITS

3.1 Method detection limits are analyzed annually on each instrument. Please see **Attachment 23.4** for an example of MDL results.

4.0 SCOPE AND APPLICATION

4.1 This SOP describes the procedure for the preparation and analysis of solid samples for Total Organic Carbon (TOC) according to Determination of Total Organic Carbon in Sediment, Lloyd Kahn, U.S. EPA Region II Edison, NJ 1988.

4.2 Greater accuracy is achieved by performing multiple replicates and also allows for the level of non-homogeneity of a sample by noting the RSD of the sample.

5.0 SUMMARY OF TEST METHOD

5.1 TOC in solid samples is measured by high-temperature combustion followed by infrared detection. Organic carbon is converted to carbon dioxide (CO_2) by catalytic combustion. The CO_2 formed can be measured directly by an infrared detector. The amount of CO_2 is directly proportional to the concentration of carbonaceous material in the sample.

5.2 The fractions of total carbon (TC) are defined as:

5.2.1 Inorganic carbon (IC) - The carbonate, bicarbonate, and dissolved CO_2

5.2.2 Total organic carbon (TOC) – All carbon atoms covalently bonded in organic molecules

5.3 TOC in solid and sludge can be measured by utilizing the combustion-infrared method. The sample is homogenized and treated with acid and then heated to remove IC. The treated sample is placed into a heated reaction chamber packed with an oxidative catalyst such as cobalt oxide. The organic carbon is oxidized to CO_2 and H_2O. The sludge and sediment sampler combusts samples at $800^oC/900^oC$ in an oxygen atmosphere so that solids as well as liquids can be analyzed.

5.4 The sampler consists of either magnetically coupled boat inlet system or a push rod and boat holder system that delivers the sample to the high temperature furnace. The CO_2 from the oxidation of organic carbon is transported in the carrier-gas stream and is measured by means of a non-dispersive infrared analyzer (NDIR).

5.5 Each sample is analyzed in triplicate to gain the accuracy of multiple analyses and, if the RSD is less than 25%, then it is deemed accurate. If the RSD is greater than 25%, then a fourth replicate is analyzed, and the new average TOC reading is reported.

6.0 DEFINITIONS

6.1 **Analytical Batch** – The basic unit for an analytical quality control is the analytical batch. The analytical batch is defined as samples that are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time period.

6.2 **Calibration** – The establishment of an analytical curve based on the area counts of known standards. The calibration standards should be prepared using the same type of acid and reagents or concentration of acids, as used in the sample preparation.

6.3 **Calibration Blank** - A volume of reagent water that acts as a zero standard and is used to calibrate the instrument.

6.4 **Calibration Standard (CAL)** - A solution prepared from the dilution of stock standards solutions. The CAL solutions are used to calibrate the instrument response with respect to organic carbon concentrations.

6.5 **Calibration curve**- If the correlation coefficient is < 0.995, if the calculated recoveries for any of the calibration standards are not within 10% of the true value, if the analysis of each standard in duplicate is greater than 10%, repeat analysis of the outlying standards until curve is within acceptance criteria.

6.6 **Correlation Coefficient** – The correlation coefficient for the calibration curve must be greater than or equal to 0.995 according to NYSDOH requirements.

6.7 **Continuing Check Blank (CCB)**- Carbon free reagent water. Analyze CCB after each CCV to check system performance.

6.8 **Continuing Calibration Verification (CCV)** – A solution of a known organic carbon concentration that is from a different source than the CAL standards, which evaluates the performance of the instrument system.

6.9 **Matrix** – The predominant material of which the sample to be analyzed is composed.

6.10 **Matrix Spike (MS)** – An aliquot of the sample is spiked with a known concentration of organic carbon. The spiking occurs during sample preparation. A matrix spike is used to document the bias of a method in a given matrix.

6.11 **Method Detection Limit (MDL)** – The minimum concentration of organic carbon that can be identified, measured, and reported with 99% confidence that the organic carbon concentration is greater than zero.

6.12 **Material Safety Data Sheet (MSDS)** – OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs. The thrust of the law is to have those who make, distribute, and use hazardous materials responsible for effective communication.

6.13 **Practical Quantitation Limit (PQL)** – is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions.

6.14 **Relative Standard Deviation (RSD)** – To compare two or more values, the relative standard deviation is based on the mean of the values and the standard deviation of the values. RSD is reported as an absolute value, i.e., always expressed as a positive number or zero.

6.15 **Replicate** – Repeated operation occurring within an analytical procedure. Two or more analyses of a single sample constitute replicate analyses**.**

6.16 **Reagent Water** – Water in which interference is not observed at or above the minimum quantitation limit of the parameters of interest.

6.17 **Rounding Rules** – If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by one. If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number.

6.17.1 If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

6.18 **Sample Delivery Group (SDG)** – Unit within a single case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a case, received over a period of up to 14 calendar days (7 calendar days for 14-day data turnaround contracts). Data from all samples in an SDG are due concurrently.

6.19 **Stock Standard solution-** A concentrated solution containing organic carbon prepared in the laboratory or purchased from a reputable commercial source.

7.0 INTERFERENCE

7.1 Volatile organics in sediments may be lost in the de-carbonation step resulting in a low bias.

7.2 Bacterial decomposition and volatilization of the organic compounds are minimized by maintaining the sample at 4°C and analyzing within the specified holding times.

8.0 SAFETY

8.1 Safety glasses, lab coat or lab apron, and disposable gloves must be worn when handling chemicals and samples.

8.2 Personnel should familiarize with the necessary safety precautions by reading MSDS information covering any chemicals used to perform this SOP.

8.3 Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Specifically, concentrated nitric, sulfuric, and hydrochloric acids present various hazards and are moderately toxic and

extremely irritating to skin and mucous membranes. Use these reagents in a fume hood and if skin contact occurs, flush with large volumes of water.

8.4 Both instruments operate at high temperatures and care should be taken to avoid direct contact with the furnace or parts that enter or are near the furnace.

9.0 EQUIPMENT AND APPARATUS

9.1 Rosemont-Dohrman DC-190 IR-I NDIR detector module and TOC Boat Sampler Model 183.
9.2 Shimadzu TOC-V Analyzer, Solid Sample Module SSM-5000A, and Autosampler ASI-V.
9.3 Quartz boats. Dohrmann (p/n 899-624).
9.4 Ceramic boats. Shimadzu (p/n 638-92099).
9.5 Quartz wool. Dohrmann (p/n 511-735).
9.6 VWR Model 1370FM drying oven set at 104 ^{O}C.
9.7 Muffle oven.
9.8 Tweezers large and small.
9.9 Magnet.
9.10 Steel spatula or tongue depressors.
9.11 Mettler AG204 analytical balance.
9.12 Aluminum weigh dishes.
9.13 9" Pasteur glass pipettes.
9.14 White bulb.
9.15 10, 25, 50, 100, 500 mL Class A volumetric flasks.
9.16 Oxygen tank (purity of 2.6 or higher) with regulator.
9.17 Gray septum. Dohrmann (p/n 517-807).
9.18 Red/White septum. Dohrmann (p/n 511-914).
9.19 Cobalt catalyst. Dohrmann (p/n 511-883).
9.20 20-mesh tin. Dohrmann (p/n 511-876).
9.21 Copper. Dohrmann (p/n 511-895).
9.22 Pyrex wool. Dohrmann (p/n 511-895).
9.23 Cobalt oxide catalyst. Shimadzu (p/n 630-00566).
9.24 Platinum catalyst. Shimadzu (p/n 017-42801-01)
9.25 Metal screen for catalyst. Shimadzu (p/n 638-58102).
9.26 Platinum mesh. Shimadzu (p/n 630-00105-01).
9.27 Pyrex baking dish.
9.28 Alconox soap.
9.29 Sonicator in biota room.

10.0 REAGENTS AND STANDARDS

10.1 LGR water: Carbon free water obtained from NEA's water system to be used as CCB. Laboratory research grade water system from U.S. Filter Water Systems Corporation.

10.2 ERA Demand TOC standard (p/n 516) to make DC-190 calibration standards.

10.3 TOC Calibration Standard for DC-190: ERA Demand stock standard. Prepare 6 standards of different concentrations ranging from ~70 ppm - ~11430 ppm. Record date and dilution information in the Inorganics standard logbook. Refer to **Attachment 23.1.**

10.4 High purity solid Potassium Hydrogen Phthalate (p/n 630-00635-01) to make Shimadzu calibration standards.

10.5 TOC Calibration Standard for Shimadzu: Potassium Hydrogen Phthalate solid that is made into a liquid standard. Prepare 5 standards of different concentrations ranging from ~100 ppm - ~ 400,000 ppm. Record date and dilution information in the Inorganics standard logbook.

10.6 High Purity Standards TOC standard (p/n CWW-TOC-G) to make Shimadzu spiking solution.

10.7 1:1 Nitric Acid (7.9 N). Dilute 50mL of ACS grade concentrated nitric acid (p/n A200SI-212) to a final volume of 100 mL

10.8 1:5 Hydrochloric Acid (2.4 N). Dilute 20mL of ACS grade concentrated hydrochloric acid (p/n A144SI-212) to a final volume of 100mL.

10.9 1:4 Phosphoric Acid (10.75 N). Dilute 125ml of ACS grade concentrated phosphoric acid (p/n JT0260-2) to a final volume of 500mL.

10.10 CCV for TOC on both instruments is 1000mg/L VWR Organic Carbon Standard (p/n VW3880-2).

10.11 Sparging Fluid (DC-190) – fill a volumetric flask most of the way with DI H_2O and then add a drop of Phosphoric Acid and test pH until it has a pH of 2.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

11.1 Solid samples require no preservation prior to analysis other than store at 4 ^{o}C and have a holding time of 28 days.

12.0 QUALITY CONTROL

12.1 Method Detection Limits should be determined annually or when a change in instrument hardware or operating conditions dictates they need to be re-determined as judged by the analyst. It is performed by running 7 or 8 replicates of the lowest standard of each instrument on its respective instrument.

12.2 Precision and Accuracy tests should be performed annually by the analysts to demonstrate their ability to reproduce an unknown sample four times accurately while following the SOP for each applicable matrix. All results should be within vendor's acceptance range and the RSD should be less than 10%.

12.3 CCV

12.3.1 Analyze immediately after calibration of an instrument. The CCV should come from a different source than the CAL standards.

12.3.2 Analyze at the beginning and end of each anlaysis. It is also analyzed every 20 sample injections.

12.3.3 Recovery of the CCV should be within 85-115% of the known value.

12.4 CCB is a blank that is analyzed immediately after every CCV that is analyzed. The CCB area should be lower than the first standard.

12.5 Each sample is run in triplicate and the RSD is then calculated. If the RSD is less than 25%, analysis continues for the next sample. If it is greater than 25%, then analyze the sample a fourth time and continue with the next sample.

12.6 Matrix Spike is run once every 20 samples, and is usually analyzed on the first sample of the batch. The solution for the DC-190 is 250 ul of the CCV solution and for the Shimadzu, is 500 ul of the CCV solution diluted from High Purity Standards stock solution. The recovery should be within 75-125% of the known value.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Calibration of the DC-190

13.1.1 A new calibration should be run every four months. It should be run sooner if CCV is repeatedly outside of limits or if the instrument has had major repairs.

13.1.2 The calibration curve is based on ‘ug of carbon’ versus ‘area’.

13.1.3 A Calibration Blank should be obtained as well as the ERA standard. The ERA standard is then diluted appropriately in volumetrics to concentrations of approximately 5, 10, 100, 250, 500, 800 ug of carbon after 70 ul of each are injected. Please see **Attachment 23.1** for details.

13.1.4 The Calibration Blank and each standard should be analyzed in duplicate. Each standard should be duplicated until they are reproducible within 10% of each other. The procedures for analyzoing each sample can be found in **Section 14**.

13.1.5 The blank area should be subtracted from the average area of each calibration standard.

13.1.6 Each calibration should begin with a quartz boat filled with new quartz wool and freshly burned in the furnace until the baseline has stabilized.

13.1.7 The area counts obtained for each replicate of each standard should be entered into the excel spreadsheet S:\Lab Data\Metals\TOC\Data(year)\tocDC190calibration(date) . The ‘Calculate Curve Click Here’ tab should then be pressed. Be sure the R square is >0.995, the recovery of each standard is within 90-110%, and the RPD is < 10% for each standard (the first standard can be within 80-120%).

13.1.8 The curve is then entered into LIMS by going to the Wetlab department and selecting TOC. In the Prep Book tab, create a new batch for the day and select the DC-190 as the Instrument. Press the Blank button twice and then the ICx button, which then adds the standards into the Prep Book. Enter the area counts into the system as well as Std Lot, Blank Area, Slope, Low Std Concentration, and Calibration Date. To finalize the calibration in LIMS select all injections and press the ‘calculate and send’ button on top (a hand with a paper in it).

13.2 Calibration of the Shimadzu

13.2.1 A new calibration should be analyzed every four months. It should be analyzed sooner if CCV is repeatedly outside of limits, or if the instrument has had major repairs.

13.2.2 The calibration curve is based on ‘ug of carbon’ versus ‘area’.

13.2.3 A Calibration Blank should be obtained as well as preparing the Potassium Hydrogen Phthalate standard at 40,000 ppm. The standard is then diluted down appropriately in a volumetric to concentrations of 100 ppm, 400 ppm and 4,000 ppm. Clean boats are then injected with different volumes of the 100 ppm, 400 ppm, 4,000 ppm, and 40,000 ppm standards and analyzed. The final concentrations analyzed are 10, 40, 400, 4,000, and 40,000 ug of carbon. Please see further details in **Attachment 23.1**.

13.2.4 To analyze the curve on the Shimadzu, create a new Sample Run and save it as (date)TOC. Then select Insert – Calibration Curve. Select new curve.cal and then analyze according to **Section 14**.

13.2.5 The program will automatically zero shift the curve.

13.2.6 Each calibration should begin with a ceramic boat filled with new ceramic fibers and freshly burned in the furnace until the baseline has stabilized.

13.2.7 The curve is then entered into LIMS by going to the Wetlab department and selecting TOC. In the Prep Book tab, create a new batch for the day and select the TOC-V as the instrument. Press the ICx button, which then adds the standards into the Prep Book. Enter the area counts into the system as well as Std Lot, Blank Area, Slope, Intercept, Low Std Concentration, and Calibration Date. To finalize the calibration in LIMS select all injections and press the ‘calculate and send’ button on top (a hand with a paper in it).

14.0 PROCEDURE

14.1 Procedures for the DC-190

14.1.1 Instrument preparation

14.1.1.1 Refer to the instrument manual for specific instructions and part numbers for all components.

14.1.1.2 In general, a portion of a sample is weighed into a quartz boat where it is acidified and dried. The boat is then placed in the boat port of the sampler, and it is moved into the combustion chamber. Gas from the combustion tube flows into the right flask where it passes through the sparging fluid. It then travels to the left flask where excess water is removed before traveling through the tin and copper scrubber to the detector.

14.1.1.3 To prepare the tin/copper scrubber (annual maintenance):

14.1.1.3.1 Insert a tuft of Pyrex wool at the bottom of one half of the U-shaped scrubber tube.

14.1.1.3.2 Weigh out 10g of tin and pour into the scrubber tube and secure with another tuft of Pyrex wool.

14.1.1.3.3 Insert a tuft of Pyrex wool at the bottom of the other half of the U-shaped scrubber tube.

14.1.1.3.4 Weigh out 10g of copper and pour into the scrubber tube and secure with another tuft of Pyrex wool.

14.1.1.3.5 Top both ends of the scrubber with a cored gray septum.

14.1.1.3.6 Check the scrubber periodically and change when about half of the tin has become discolored.

14.1.1.4 To prepare the combustion tube (quarterly maintenance):

14.1.1.4.1 Insert a tuft of quartz wool into the combustion tube and push it down to the dimple.

14.1.1.4.2 Insert about 3 inches of cobalt oxide catalyst and tap down to be sure it is uniformly packed.

14.1.1.4.3 Secure the cobalt oxide with another tuft of quartz wool. Verify the cobalt oxide will not move when the tube is turned horizontally.

14.1.1.4.4 Insert combustion tube into the furnace and secure tightly in large bolt near boat port. Also recap the left end with the cored gray septum.

14.1.1.5 The main NDIR detector should be turned on by the switch in the rear of the instrument and allowed to stabilize for a few hours. This module is generally left running and only shut off to reboot the instrument when necessary.

14.1.1.6 The Analysis Mode should be set to TC and the Inlet Mode should be set to Boat.

14.1.1.7 At the start of each day carefully check all components of both the main unit and boat sampler for wear. Check the level of acidified water in the right flask to be sure it is above the sparging finger. Empty any fluid in the left flask into acid waste. Turn the power on to the TOC Boat Sampler so the furnace begins to heat up and turn the oxygen tank on. Be sure the oxygen is set to 30 psi. Check the right flask to be sure a vigorous flow of gas is emitted from the sparging finger. If not bubbling check lines for gas leaks.

14.1.1.8 The boat sampler must warm up until the Furnace light is green, the Carrier Gas light is green, and the baseline is stabilized at a number

less than 3, which is displayed in the lower right-hand side of the main unit's display screen.

14.1.2 Solid samples preparation

14.1.2.1 Create a new batch in LIMS

14.1.2.1.1 Under "To Be Done" tab, pick "DC-190" from instrument drop down box, and then press "NEW" button.

14.1.2.1.2 Add the assigned QC sample first into the newly created batch by selecting it in the To Be Done tab, right-click and append to PrepBatch.

14.1.2.1.3 Select the QC sample under the "WetLab Book" tab, and add CCV and CCB by press the "SET" button.

14.1.2.1.4 Move the CCV and CCB set to the top of the batch by pressing the arrow button.

14.1.2.1.5 Add a MS to selected QC sample by pressing "MS" button.

14.1.2.1.6 Add samples.

14.1.2.1.7 Add QC set (CCV and CCB) for every twenty samples or less.

14.1.2.1.8 Assign blank IDs by selecting rows, right clicking mouse, and then following instruction.

14.1.2.1.9 Assign batch QCs by right clicking mouse, and then follow instructions.

14.1.2.2 Weigh samples

14.1.2.2.1 Lay out aluminum pans on a tray in labeled numerical order.

14.1.2.2.2 Place a clean quartz boat in each pan.

14.1.2.2.3 Assign each pan to every sample. Type the label numbers in the prep batch in LIMS.

14.1.2.2.4 Place aluminum pan on the analytical scale. Tare the scale.

14.1.2.2.5 Open the sample jar and stir with a spatula to homogenize the sample.

14.1.2.2.6 Weigh each sample amount between 0.005g and 0.01g into a quartz boat. Each sample is prepared with four replicates. The fourth may or may not be used depending on the RSD.

14.1.2.3 Acidify all samples with 2-3 drops of 1:1 HNO_3. Moisten the entire sample if more drops of acid are needed.

14.1.2.4 Place samples in the drying oven until the samples have dried (approximately 5-10 mins).

14.1.2.5 Spike the MS sample with 250 uL of CCV solution and dry in the oven for another 10 minutes.

14.1.2.6 Total solids should also be determined as per SOP NE090.

14.1.3 Sample analysis

14.1.3.1 When the instrument and samples are ready, the analysis will begin by entering data in the Wetlab department in LIMS. Select the TOC tab on the left.

14.1.3.2 Place your cursor in the Area box of the sample you intend to run (when you start should be a CCV).

14.1.3.3 Shut the oxygen off using the black toggle switch and toggle down on the boat sampler. Slide the boat under the red septum using the magnet, open the red septum, and pipette 70 ul of the CCV solution into the boat. Turn on the oxygen by moving the black toggle switch up. If the Start light is lit, then press Yes; and if the Start light is not lit, press Start, 1, Enter. Once the instrument makes a short noise, use the magnet and slide the boat into the furnace.

14.1.3.4 When the instrument makes a short noise again, use the magnet to slide the boat out from furnace. The area should now be in the LIMS Area box for CCV and has automatically moved down to the CCB Area box. To be sure the CCV has passed, select the line and press the 'calculate and send' button and scroll over to check the % Recovery column.

14.1.3.5 Once the boat has cooled, the CCB liquid sample can be added to the boat and measured likewise. Be sure the cursor is in the Area box for CCB before starting.

14.1.3.6 To add a solid sample, first shut off oxygen using the black toggle, and then open the boat port by pushing the red handle upward. Remove the CCV boat by using small tweezers and attach the sample boat to the hook of the wire. Close the boat port and toggle on the oxygen. Start the instrument and continue running samples likewise. Be sure to wait for the baseline to settle back down to a value less than 3 before starting a new sample.

14.1.3.7 For each sample, analyze the first three replicates and verify the weights are entered in LIMS; then press the 'calculate and send' button. If the calculated RSD is less than 25%, continue on to the next sample, but if the RSD is greater than 25%m then anlayze the fourth replicate.

14.1.3.8 If at any point the concentration of the sample is higher than the calibration range, re-prep the sample as noted above, but weigh out a smaller amount, which should not go below 0.001g.

14.1.3.9 When finished with analysis, verify all samples have been calculated and sent. Select each CCV/CCB set and the following 20 sample injections, right click, and assign Blank ID. Then, select each LRF and the CCV/CCB sets and MS assigned to it; right click and save selected QC injections for LRF. Fill in the parameters directly above the analysis log with the pressure of the oxygen tank for oxygen flow, select the HIGH setting for range, and then the number the baseline stabilized at throughout the day as the baseline.

14.1.3.10 By selecting the entire analysis of the day and then pressing the Print button, a Logbook page can be printed to place into the folder and package.

14.1.3.11 Shut off the oxygen tank and power off the boat sampler at the end of a day. Leave the main unit running.

14.1.4 Boat cleaning: place dirty boats in a jar and fill with water and a small dash of Alconox. Shake vigorously to make water sudsy and mix with boats. Place the jar in the sonicator and turn on sonicator for 20 minutes. Remove from sonicator and rinse with water multiple times until water is not soapy. Dry boats in drying oven first, and then burn in the muffle oven at 900° C for 20 minutes.

14.2 Procedures for the Shimadzu:

14.2.1 Instrument preparation

14.2.1.1 Refer to the instrument manual for specific instructions and part numbers for all components. Also you can refer to the online Virtual Advisor on the Shimadzu website to watch repair videos and troubleshoot problems. The website is http://www.ssi.shimadzu.com/toc_virtualadvisor/.

14.2.1.2 A portion of sample is weighed into a ceramic boat where it is acidified and dried. The boat is placed on the boat holder of the TC port and it is moved into the combustion chamber. Gas from the combustion tube flows through a condensation coil, dryer, and cooling coil into the main unit to the detector.

14.2.1.3 To prepare the TC catalyst of the SSM:

14.2.1.3.1 Verify the instrument is turned off and cooled down to room temperature. Then, remove the cover and unscrew the boat port. Gently remove the small end of the catalyst tube from the nut and slowly remove the catalyst tube from the furnace.

14.2.1.3.2 In a separate container, mix two containers of cobalt oxide catalyst (50g total) and one container of platinum catalyst (20g).

14.2.1.3.3 Fold the catalyst support screen and insert into tube with the folds facing towards the thin opening of the catalyst tube.

14.2.1.3.4 Place about 2 mm of quartz wool on top of the screen.

14.2.1.3.5 Pour in the mixed catalyst and tap gently to settle it down. Verify the catalyst is about 75 mm in height and well settled.

14.2.1.3.6 Add another 2 mm of quartz wool on top of the catalyst.

14.2.1.3.7 Fold another catalyst support screen and place it on top of the quartz wool. Verify the folds are facing towards the large opening of the catalyst tube.

14.2.1.3.8 Gently slide the catalyst tube into the furnace and slide the thin tube into the nut. Reattach the boat port by screwing on the adapter and the small knobs that hold it in place. Replace the cover and turn on the instrument.

14.2.1.3.9 Allow the instrument to heat to temperature with oxygen running through the system for 2-3 hours to stabilize the instrument by burning off any particles on the catalyst.

14.2.1.4 To prepare the main unit TC catalyst:

14.2.1.4.1 Verify the instrument is turned off and has cooled to room temperature. Open the door of the main unit and remove the small top panel.

14.2.1.4.2 Disconnect the carrier gas tubing on the right and loosen all the thumbscrews and remove the injector block.

14.2.1.4.3 Gently remove the bottom end of the combustion tube from the nut and slowly pull the tube up and out of the furnace.

14.2.1.4.4 Remove the catalyst and clean the tube if necessary.

14.2.1.4.5 Place two platinum mesh screens on the bottom of the tube and add 5 mm of quartz wool.

14.2.1.4.6 Add 100mm of the regular sensitivity catalyst, which is platinum catalyst only. Tap gently to be sure it is settled.

14.2.1.4.7 Replace the tube into the furnace and connect to bottom nut.

14.2.1.4.8 Reattach the injector block and check that it is centered over the catalyst tube. Reconnect the carrier gas tubing line.

14.2.1.4.9 Close the instrument top. Turn on the instrument and allow the catalyst to burn off particles for 2-3 hours.

14.2.1.5 Turn on the main unit. Press the button on the bottom right corner on the front of the main unit. The main unit has the NDIR Detector and should warm up for an hour or two. This unit is generally left on and only shut off to reboot the instrument.

14.2.1.6 Turn on the SSM using the switch on the right side towards the back. This unit is generally left on but can be shut off. Warm it up long enough for the temperature to rise to its set point and the baseline to settle.

14.2.1.7 At the start of each day check the moisturizer level to be sure it is within the fill range and the humidifier to verify the water level is above the end of the outlet tube on the main unit. Turn on the oxygen tank. Verify the oxygen is set to 60psi on the regulator. On the SSM, check the drain separator on the right side of the system to be sure it is not bubbling; fill with water to stop bubbling.

14.2.1.8 Open the TOC-Control V program. Select the Sample Table Editor. Press the New button (piece of white paper) and select Sample Run. Select the TOC-V+SSM as the instrument and name the file as the (date)toc. Press the connect button (lightning bolt) and select use settings on PC. The instrument will now connect with the computer and SSM. When it is fully initialized you can watch the baseline by going to Instrument and Background Monitor.

14.2.1.9 The instrument is ready to run when in the Background Monitor all lights are green in both the SSM and TOC tabs.

14.2.2 Solid sample preparation

14.2.2.1 Create a new batch as stated in **Section 14.1.1** but choose a different instrument (TOC-V CSH or CSH2).

14.2.2.2 Place a pre-numbered ceramic boat on the analytical scale and tare the scale.

14.2.2.3 The sample jar is then opened and stirred with a spatula to homogenize.

14.2.2.4 Each sample is weighed into a pre-numbered ceramic boat to weigh about 0.05g, and is prepared with four replicates in four individual boats. For the first sample a fifth boat is used to perform the MS. Weigh out all samples in a batch at the same time.

14.2.2.5 The ceramic boats are then placed carefully into a Pyrex dish. The dish is then carefully moved near the drying oven where each boat is acidified with 3-4 drops of 1:5 HCl being sure to moisten the entire sample.

14.2.2.6 The dish is then placed into the drying oven until the samples have dried (approximately 5-10 mins). At this point, the MS should be spiked with 500 uL of a 1000 ppm CCV solution and dried for 10 minutes.

14.2.2.7 Total solids should also be determined as per SOP NE090.

14.2.3 Sample analysis

14.2.3.1 Open the LIMS's batch, select all rows and press: "Save selected rows to file" button. Select method of the jumped window, and then type in a file name as mmddyyTOC4.

14.2.3.2 Open a new file in TOC Control-V program and go to Edit and select Import. Select the file that was just created. All the samples should then appear in the sample table as they did in LIMS.

14.2.3.3 Place the boat in the boat holder and close the boat port. It will then ask you to enter the weight of the sample. If you are running CCV or CCB you enter the volume (should be 100 ul) by un-checking the By Weight box. If you are running solid samples enter the weight as recorded in LIMS. Slide the boat in when the instrument asks you to by pushing the blue push bar.

14.2.3.4 After the sample is measured a window will pop up that asks you to move the boat back to the cooling position and then again to the stop point. Press next to continue running the next sample.

14.2.3.5 After three replicates you will want to check the % Recovery or RSD when the sample is measured press Stop and then go into LIMS. From LIMS highlight the sample you want to check on and press the 'Read Shimadzu data' button (an open book). After a few seconds the information will appear in LIMS and can be calculated and sent (using the hand and paper button) to see the calculations.

14.2.3.6 To pick up your run after checking on data just select the next sample to be run and press the green stoplight button. If the fourth replicate does not need to be run highlight the sample and press the scissors button to remove it from the run. Be sure to remember to remove it from the LIMS run also.

14.2.3.7 As you go you will have to make multiple files to be read into the Shimadzu program after you determine whether you are running 3 or 4 samples. Be sure they each get different names like (date)toc4-2.

14.2.3.8 When the entire run is completed. Go into LIMS highlight all samples that have not previously been read into LIMS, read them in from the shimadzu, and then press the 'calculate and send' button.

14.2.3.9 Select each CCV/CCB set and the following 20 samples and right click and assign blank ID. Then select all QC and right click and Save selected QC for each LRF. Set the parameters to 30 psi for oxygen flow, Low range, and '-' for baseline and save parameters. Check that the calibration information is correct.

14.2.3.10 Select the analysis for the day in LIMS and print a logbook (printer button).

14.2.4 Clean the ceramic boats: scrap all the ash from the boats. Then place the boats in a jar with 2M HCl and cap. Shake the jar gently and allow to sit for at least a half hour. Carefully remove the boats from the jar with tweezers and rinse thoroughly with tap water and then DI water. Then, place the boats in the drying oven to dry. Once dry, they can be placed in the muffle at 900° C for 10 minutes.

15.0 CALCULATIONS

15.1 RSD Calculation: $\frac{\text{(Standard Deviation of runs)}}{\text{(Average of runs)}} *100$

15.2 Spike Added Calculation: $\frac{\text{(volume of spike)*(spike concentration)}}{\text{(total solids)*(weight of sample)}}$

15.3 % Recovery of Spike: $\frac{\text{(TOC concentration of MS) – (TOC concentration of first run)}}{\text{Spike Added}} *100$

15.4 TOC Concentration Calculation for DC-190: $\frac{\text{(Area-Blank Area)* (Inverse Slope)}}{\text{(sample weight)*(total solids)}}$

15.5 TOC Concentration Calculation of Shimadzu: $\frac{\text{(Area – Intercept)}}{\text{(Slope) * (sample weight)}}$

15.6 Final TOC Concentration Calculation for Shimadzu: $\frac{\text{(TOC Concentration)}}{\text{(total solids)}}$

15.7 Average TOC Concentration: $\frac{\text{(Final Conc. 1) + (Final Conc. 2) + (Final Conc. 3)}}{3}$

or: $\frac{\text{(Final Conc. 1) + (Final Conc. 2) + (Final Conc. 3) + (Final Conc. 4)}}{4}$

15.8 % Recovery of CCV: $\frac{\text{(TOC Concentration of CCV)}}{\text{(True Value of CCV)}} *100$

15.9 PQL Calculation: $\frac{\text{(concentration of lowest standard in ug)}}{\text{(sample weight in g) * (total solids)}}$

15.10 % Total Solids: $\frac{\text{(Dry sample weight) – (dish weight)}}{\text{(Wet sample weight) – (dish weight)}} *100$

16.0 METHOD PERFORMANCE

16.1 Method Detection Limit

16.1.1 The MDL was previously discussed in **Section 12**. The MDL will vary but presently the DC-190 is at 68.2 mg/kg, TOC-V CSH is at 136 mg/kg and TOC-V2 is at 140 mg/kg. All data files from previously performed MDLs can be found in the QA office.

16.2 Initial Demonstration

16.2.1 Initial demonstrations must be performed on each instrument upon first receiving it to demonstrate its capabilities with different matrices. It requires a check against outside standards of both liquid and solid nature.

17.0 POLLUTION PREVENTION

17.1 Refer to SOP NE168 for instructions for pollution prevention.

18.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QC MEASURES

18.1 Calibration Assessment

18.1.1 All standards are analyzed in duplicate with RPD < 10%

18.1.2 Linear Regression should be $\geq$ 0.995

18.1.3 Recovery of the calculated concentration for each standard should be 90 – 110%. The lowest standard can have a recovery between 80 – 120%.

18.2 CCV recovery should be 85 – 115%

18.3 RSD for the first three replicates should be less than 25%. If greater than 25%, there should be a fourth replicate analysis.

18.4 MS recovery should be 75 –125%

18.5 CCB should have an area count lower than the mean area count of the first calibration standard.

18.6 All samples should have area counts lower than the mean area count of the fifth calibration standard.

19.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

19.1 Calibration must meet all requirements in **Section 18**. If it does not, the analyst can re-analyze a standard that is outside the curve, re-prepare the solution, and re-analyze as well. If requirements are still not obtained, begin troubleshooting for mechanical issues. Begin with an air flow check and catalyst performance check.

19.2 If CCV does not pass, the boat should be burned in the furnace for 5 minutes and then cooled. Retry the CCV, and if it still does not pass and there are no pipette issues (tip not on tightly, air bubble when solution sucked into tip) new solution should be obtained and analyzed. If it still does not pass, then air flow should be checked. If the recovery is too high, then there is too little flow or there is a catalyst issue; and if the recovery is too low consistently, then it may be a catalyst issue.

19.3 If the MS does not pass, then check that the amount being spiked with is at least 5 times greater than the PQL. If it is greater than 5X PQL, then re-prepare an MS sample and reanalyze. If it fails again, then write a case narrative explaining the issue. If it is less than 5X PQL, then write a case narrative explaining this and continue on with the anlaysis.

19.4 If the CCB does not pass, the boat should be burned in the furnace for 5 minutes and then cooled. Retry the CCB, and if it still does not pass, then air flow should be checked as well as the catalyst performance.

19.5 If a sample is found to be over the range of the calibration area count then the sample should be re-prepared at a smaller weight that would proportionally bring it into the range of the curve. The sample should still weigh more than 0.001g.

19.6 Also, some of these corrective actions can be seen in the flow chart **Attachment 23.5**.

20.0 CONTIGENCIES FOR HANDLING OUT-OF-CONTROL DATA

20.1 If the acceptance criteria for QC measures has been exceeded, the limits and the data must be reported, and the following procedures must be implemented:

20.1.1 The Quality Assurance Officer must be notified.

20.1.2 The data must be flagged with the appropriate qualifiers and a case narrative must be written.

20.1.3 The client must be notified about the data.

21.0 WASTE MANAGEMENT

21.1 All applicable federal and state rules and regulations governing hazardous waste will be followed when the disposal of laboratory waste is generated during the execution of this method.

21.2 Refer to SOP NE054 for instructions for the disposal of waste.

22.0 REFERENCES

22.1 "Determination of Total Organic Carbon in Sediment," Lloyd Kahn, U.S.E.P.A. Region II, Edison, NJ 1988.

22.2 "DC-190 High-Temperature TOC Analyzer Operation Manual," Rosemont, Inc. 1990,1991 Rev. C.

22.3 "TOC-V CPH/CPN & TOC-Control Software User Manual," Shimadzu Corporation 2004.

22.4 "SSM-5000A for TOC-V Series Total Organic Carbon Analyzers User Manual," Shimadzu Corporation 2001.

23.0 ATTACHMENTS

23.1 Curve Preparation and Information

23.2 Example of LIMS Logbook

23.3 MDL Results

23.4 Flowchart of Corrective Actions

Attachment 23.1
Curve Preparation and Information

DC-190 Curve

To calculate the standards to use for the DC-190 curve, there is an excel file located at:
S:\Lab Data\Metals\TOC\DC190 std calc

Enter the certified TOC concentration of the ERA standard and then adjust the Amount used column numbers until they match or are near the desired values. These end concentrations should then be adjusted in the calibration curve page when calibrating.

Below is a sample of the dilutions completed for one ERA standard stock solution.

ERA certified concentration: 90.2 ppm
Actual undiluted concentration: 18040 ppm

Standard Number	Amount used (ml)	Final Volume (ml)	Concentration (ppm)	Volume Used to calibrate (ml)	End Concentration (ug)	Desired Concentration (ug)
1	0.039	10	70.356	0.07	4.92	5
2	0.079	10	142.516	0.07	9.98	10
3	0.397	5	1432.376	0.07	100.27	100
4	0.99	5	3571.92	0.07	250.03	250
5	1.98	5	7143.84	0.07	500.07	500
6	3.167	5	11426.536	0.07	799.86	800

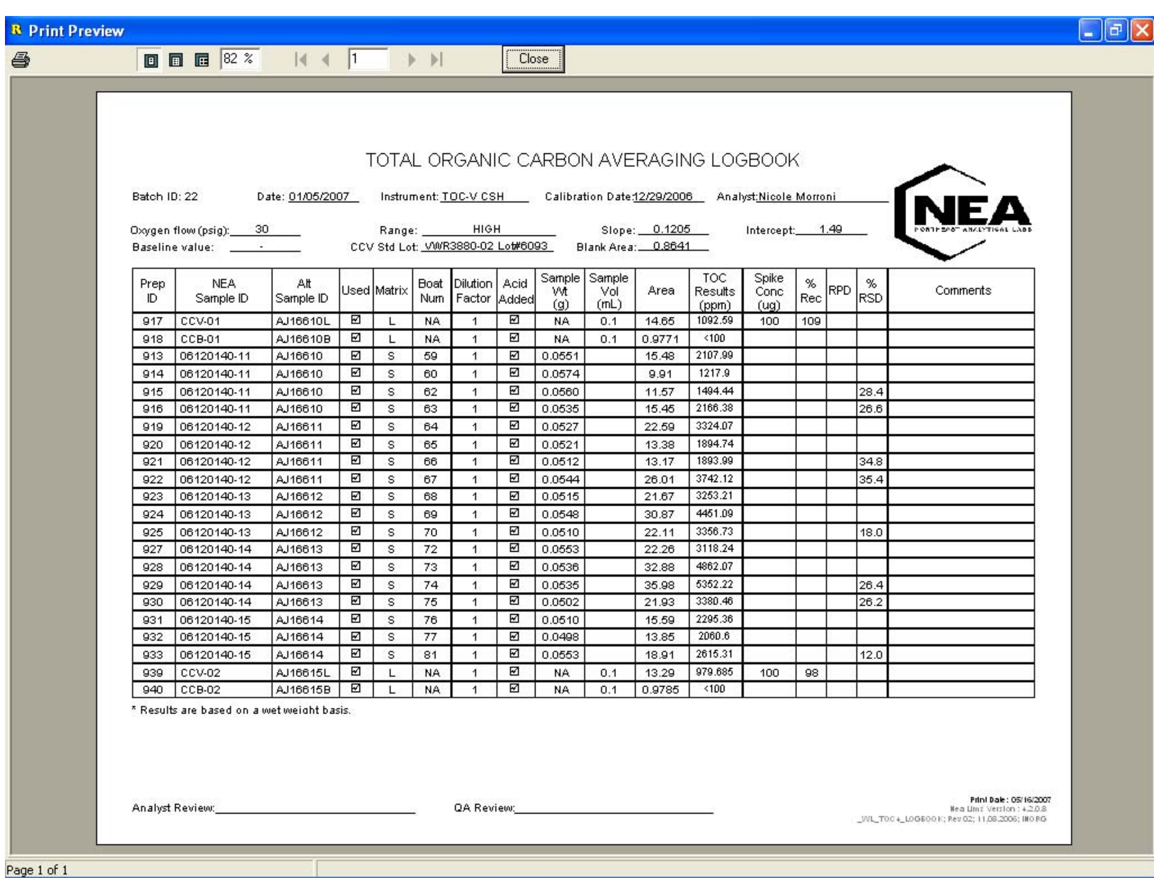
Shimadzu Curve

Begin by placing Potassium Hydrogen Phthalate in a clean jar and drying in the oven at 104 C for an hour. Once it is cooled, in the dessicator, 2.1266 g is weighed into a 25 mL volumetric. The volumetric is

then filled with fresh DI H_2O from the volatiles lab. Then, shake the volumetric until completely dissolved, and can be warmed to help this process. This solution has a concentration of 40,000 ppm. It can be diluted to 400 ppm by taking 0.5 mL and diluting it to 50 mL with fresh DI H_2O.

Standard 1 – 0 ppm – pipette 100uL of fresh DI H2O into a boat
Standard 2 – 10 ppm - pipette 25 uL of the 400 ppm standard into a boat
Standard 3 – 40 ppm - pipette 100 uL of the 400 ppm standard into a boat
Standard 4 – 400 ppm - pipette 100 uL of the 4,000 ppm standard into a boat
Standard 5 – 4,000 ppm - pipette 100 uL of the 40,000 ppm standard into a boat
Standard 6 – 40,000 ppm - pipette 1 mL of the 40,000 ppm standard into a boat and place it in the drying oven until it is completely dry, and then analyze in the Shimadzu.

Attachment 23.2
Example of LIMS Logbook



Print Preview

82 % | 1 | Close

TOTAL ORGANIC CARBON AVERAGING LOGBOOK

NEA NORTHEAST ANALYTICAL LABS

Batch ID: 22 Date: 01/05/2007 Instrument: TOC-V CSH Calibration Date: 12/29/2006 Analyst: Nicole Morroni

Oxygen flow (psig): 30 Range: HIGH Slope: 0.1205 Intercept: 1.49
Baseline value: - CCV Std Lot: VWR3880-02 Lot#6093 Blank Area: 0.8641

Prep ID	NEA Sample ID	Alt Sample ID	Used	Matrix	Boat Num	Dilution Factor	Acid Added	Sample Wt (g)	Sample Vol (mL)	Area	TOC Results (ppm)	Spike Conc (ug)	% Rec	RPD	% RSD	Comments
917	CCV-01	AJ16610L	☑	L	NA	1	☑	NA	0.1	14.65	1092.59	100	109			
918	CCB-01	AJ16610B	☑	L	NA	1	☑	NA	0.1	0.9771	<100					
913	06120140-11	AJ16610	☑	S	59	1	☑	0.0551		15.48	2107.99					
914	06120140-11	AJ16610	☑	S	60	1	☑	0.0574		9.91	1217.9					
915	06120140-11	AJ16610	☑	S	62	1	☑	0.0560		11.57	1494.44				28.4	
916	06120140-11	AJ16610	☑	S	63	1	☑	0.0535		15.45	2166.38				26.6	
919	06120140-12	AJ16611	☑	S	64	1	☑	0.0527		22.59	3324.07					
920	06120140-12	AJ16611	☑	S	65	1	☑	0.0521		13.38	1894.74					
921	06120140-12	AJ16611	☑	S	66	1	☑	0.0512		13.17	1893.99				34.8	
922	06120140-12	AJ16611	☑	S	67	1	☑	0.0544		26.01	3742.12				35.4	
923	06120140-13	AJ16612	☑	S	68	1	☑	0.0515		21.67	3253.21					
924	06120140-13	AJ16612	☑	S	69	1	☑	0.0548		30.87	4451.09					
925	06120140-13	AJ16612	☑	S	70	1	☑	0.0510		22.11	3356.73				18.0	
927	06120140-14	AJ16613	☑	S	72	1	☑	0.0553		22.26	3118.24					
928	06120140-14	AJ16613	☑	S	73	1	☑	0.0536		32.88	4862.07					
929	06120140-14	AJ16613	☑	S	74	1	☑	0.0535		35.98	5352.22				26.4	
930	06120140-14	AJ16613	☑	S	75	1	☑	0.0502		21.93	3380.46				26.2	
931	06120140-15	AJ16614	☑	S	76	1	☑	0.0510		15.59	2295.36					
932	06120140-15	AJ16614	☑	S	77	1	☑	0.0498		13.85	2060.6					
933	06120140-15	AJ16614	☑	S	81	1	☑	0.0553		18.91	2615.31				12.0	
939	CCV-02	AJ16615L	☑	L	NA	1	☑	NA	0.1	13.29	979.685	100	98			
940	CCB-02	AJ16615B	☑	L	NA	1	☑	NA	0.1	0.9785	<100					

* Results are based on a wet weight basis.

Analyst Review:__________ QA Review:__________

Print Date: 05/16/2007
Nea Lims Version: 4.2.0.8
_WL_TOC4_LOGBOOK; Rev 02; 11.08.2006; INORG

Page 1 of 1

Attachment 23.3
MDL Results

Northeast Analytical, Inc.

File Name: Q:\IDL\TOC\011206DC190.XLS

Instrument Detection Limits

Date: 12-Jan-06

Instrument Detection Limit (IDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	Total Organic Carbon	Analysis:	EPA Lloyd Kahn
Matrix:	Soil/Solid	Instrument:	DC 190
Spike conc:	4.99 ug	Method:	Boat Injection/IR

	NEA Sample ID	Preparation Date	File Name	Analysis Date	Measured Concentration ug	Percent Recovery (%)
1	Run #1	01/05/04	Run #1	01/12/06	3.96	79.4%
2	Run #2	01/05/04	Run #2	01/12/06	3.38	67.7%
3	Run #3	01/05/04	Run #3	01/12/06	3.31	66.3%
4	Run #4	01/05/04	Run #4	01/12/06	3.18	63.7%
5	Run #5	01/05/04	Run #5	01/12/06	3.40	68.1%
6	Run #6	01/05/04	Run #6	01/12/06	3.82	76.6%
7	Run #7	01/05/04	Run #7	01/12/06	3.20	64.1%
8	Run #8	01/05/04	Run #8	01/12/06	3.33	66.7%
				Number (n):	8	
	One sided Student's t values (t)			AVG:	3.45	ug

at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

STD (s):	0.29	ug
%RSD:	8.30%	
MDL:	0.86	ug
VALID ?:	Valid	

IDL calculations: IDL = t * s

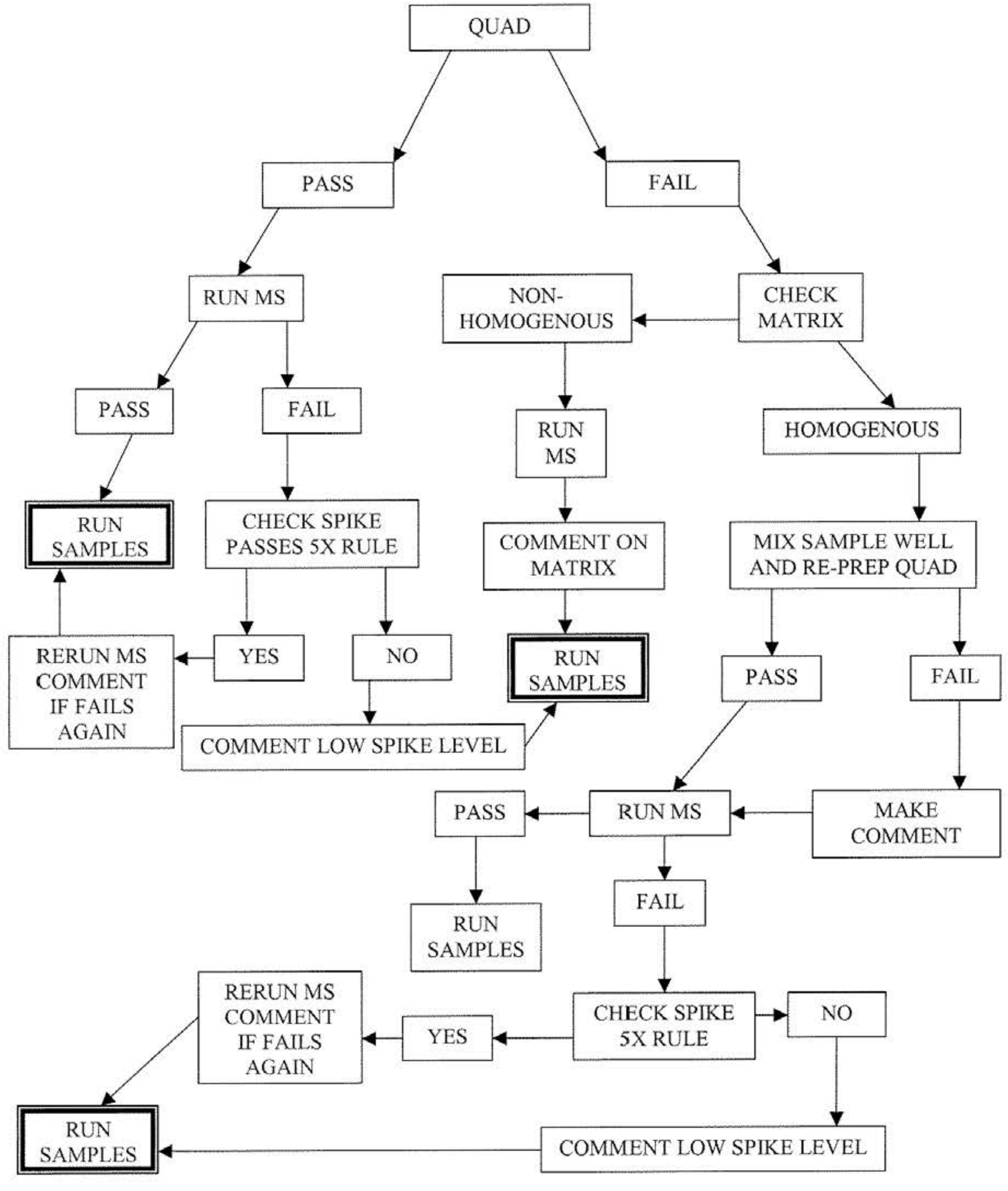
Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

Sample Preparation Chemist:	CJA	Date:	11/14/2005
Instrument Analyst:	CJA	Date:	01/12/2006

TOC FLOW CHART



STANDARD OPERATING PROCEDURE REVIEW

SOP Name	Review Number	Reviewers	Title	QAO Approval	Effective Date
NE177_04	00	Christina L. Braidwood Robert E. Wagner	QAO Lab Director	Christina Braidwood	10/18/10

Appendix II

Vibracore Sediment Collection Procedures

Vibracore Sediment Collection Procedures

I. Scope and Application

The general procedures to be utilized in obtaining Vibracore sediment samples from the river are outlined below. Aluminum or Lexan® tubing will be the primary method used to collect sediment cores.

Following collection, the sediment cores will be transferred to a processing area.

II. Personnel Qualifications

Not applicable.

III. Equipment List

The following equipment will be required for use during collection procedures:

- personal protective equipment (PPE), as required by the Health and Safety Plan (HASP);
- navigation and site maps;
- boat equipped with 90HP outboard motor;
- Vibracore device (Rossfelder P-3C or equivalent);
- Lexan® and aluminum tubing with end caps;
- calibrated rod for sediment depth measurement;
- 6-foot (minimum) rule and survey rod;
- duct tape;
- camera; and
- field notebook.

The following equipment list contains materials that may be needed to process the cores following collection:

- PPE, as required by the HASP;
- brushes;
- wash/rinse tubs;

- low phosphate detergent;
- decontamination equipment (e.g., brushes, wash/rinse tubs, detergents, and cleaning solvents), as required;
- deionized water;
- tap water;
- core rack;
- table for processing cores;
- ruler or measuring tape;
- hacksaw;
- electric sheet metal shears or similar device;
- sampling equipment (e.g., stainless steel utensils and bowls);
- sample bottles for chemical analyses;
- refrigerator (at 4°C);
- camera;
- Unified Soil Classification System (USCS) charts;
- photoionization detector (PID); and
- field notebook.

IV. Health and Safety Considerations

Not applicable.

V. Sampling Procedures

1. Maneuver the sampling vessel to within 2 feet of the target sample location. Secure the vessel in place using spuds, anchors, or tie lines.

2. Use a calibrated steel rod to probe the sediment surface 3 to 5 feet away from the target location to determine the sediment thickness, type, and presence of debris or obstructions.

3. Once the targeted area is deemed suitable for core collection, select an appropriate 3-inch (outside diameter) core tube type (Lexan® or aluminum) and length based on the probing information. Use Lexan® tubing in soft sediments and aluminum tubing for coarse sediments. Deeper sediments will be sampled with core tubes custom cut on the boat from 12-foot tube sections.

4. Mount a clean coring tube into the Vibracore device.

5. Lower the coring apparatus with the core tube attached vertically through the water column tube end first, until the river bottom is reached.

6. Vibrate the core into the sediment to refusal. Measure and record the depth of core tube penetration into the sediment in the field notebook.

7. Pull the apparatus upward out of the river bottom (using a winch) and raise it to the surface while maintaining the core in a vertical position.

8. Before the bottom of the tube breaks the water surface, place a cap over the bottom to prevent loss of material from the corer. The cap will be placed on the core by reaching down into the water from the center of the sample vessel. Secure the cap in place with duct tape when brought on board the vessel.

9. Water overlying the core tube in the coring apparatus will be allowed to drain prior to removal of the core tube.

10. Estimate the recovered length of the sediment core and note it in the field notebook.

    - The length of the cores recovered in the Lexan® tubing will be determined by visual observation and direct measurement.

    - The approximate length of the cores recovered in the aluminum tubing will be determined indirectly by tapping the core with a metal rod. The spot where the pitch of the sound changes corresponds to the approximate top of the recovered core. The distance between the top of the sediment in the core tube and the bottom of the coring tube corresponds to the estimated length of the recovered core.

11. Compare the measured length of the recovered core with the core penetration depth.

    - If the recovered length of the sediment core is more than 60% of the penetration depth, keep the core for analysis.

    - If an insufficient amount of material is recovered, set the core tube to the side and prepare to make an additional attempt.

- An additional attempt will be made at a minimum distance of 2 feet from the previously attempted location.

- A maximum of three attempts to collect a core will be made for a given location ID.

- If all three attempts to collect a core are unsuccessful based on recovery alone (i.e., less than 60% recovery), retain the longest core for analysis and indicate that the targeted recovery was not achieved. Discard the usable cores into a re-sealable 5-gallon pail for subsequent disposal.

12. After core recovery, enter additional information into the field notes:

    - date;
    - time of recovery;
    - sample position;
    - water depth (feet);
    - core tube material (aluminum or Lexan®);
    - core penetration depth (inch); and
    - observation, including probing results.

13. Remove the core tube from the Vibracore device and place a second cap on top of the core tube labeled with the site location ID and the word "top." Secure the cap in place with duct tape. Rinse the outside of the core tube with a small amount of river water.

14. Draw an arrow on the core tube with permanent marker to mark the top of the core. Label the core with permanent marker indicating station ID, date, and time.

15. Store the core vertically while on the vessel and transport it to the processing area.

VI. Core Processing Procedures

The general procedures to be utilized for the processing of Vibracore sediment cores and the extraction of samples for chemical analyses are outlined below. Core processing includes observational and photographic logging of the cores and collection of samples from the cores for chemical analyses.

Pre-Processing the Core Prior to Sample Extraction

The following procedures will be followed to prepare the core for logging and sample extraction:

1. Gather the necessary decontaminated sampling equipment and sample jars to collect sediment samples from the core.

2. Transfer the core to the processing area. The core should be maintained in a vertical position and kept cold while in transit to the core processing area prior to processing.

3. Upon delivery of the core to the processing area, transfer the field sampling information from the sampling personnel to the processing personnel. If applicable, chain-of-custody forms will be signed by the sampling personnel and processing personnel.

4. Maintain the core vertically in the core rack and dry the surface of the core tube with clean paper towels.

5. While the core tube is vertical in the core rack, remove the top cap from the core and inspect the sediments within the core to determine if they are comprised of loose, watery sediments (that would slump if placed horizontally) or cohesive sediments. Remove any loose sediment with a stainless steel utensil while maintaining the core tube in a vertical position.

6. Place the core horizontally on the core processing table and cut the core tube open lengthwise. Split the core in half. If PID screening is being performed for selection of Appendix IX+3 samples, take a PID reading along the length of the opened core as soon as possible after opening.

7. Mark the sample interval ranges on the outside of the core tube.

8. Describe the core while the core is split open on the core processing table. Record the description of the sediment type using the USCS in the field notebook. The description should include such information as approximate grain size (silt, clay, fine, medium or coarse sand, or gravel), the presence of organic matter, or biota, odor, and color. Record any unusual observations in the field notebook. Identify changes in the sediment (such as sediment type or grain size) within the core.

9. Photograph the opened core. In the photograph, include a ruler or measuring tape for scale and mark the top and bottom ends of the core. Photograph any foreign objects or gaps. Record the photograph number and a description of each photograph in the field notebook.

Sample Extraction

The procedures to collect sediment from the core for chemical analysis are presented below. In addition, if samples are being collected for analysis of volatile organic compounds (VOCs) or volatile/extractable petroleum hydrocarbons (VPH/EPH), the collection of those samples should also incorporate the procedures presented in Appendices A and B for VOCs and VPH/EPH, respectively.

1. Prior to collecting sediment for chemical analysis, remove the smear zone (i.e., the portion of the sediment core that comes into contact with the core tube) over the interval to be sampled, to the extent practical.

2. For each sample interval, remove sediment from the open core tube using a decontaminated stainless steel utensil and place the sediment in a dedicated, decontaminated stainless steel bowl.

3. Using a stainless steel utensil, thoroughly mix the sample in the center of the stainless steel bowl. Homogenize the sediment until the color and texture differences are no longer observable. Samples collected for certain analyses (e.g., volatile organic analyses samples collected with Encore samplers) may be required to be collected prior to homogenization.

4. Fill the appropriate, pre-labeled sample jars with the homogenized sediment for chemical analyses.

5. Handle, pack, and ship the samples using the chain-of-custody procedures in accordance with Appendix L.

6. Cleaning of sampling equipment is to follow the procedures specified in Appendix W. The sampling equipment is to be cleaned prior to the start of sampling activities, between samples, and following the completion of sampling activities.

7. Rinse water, PPE, and other residuals generated during the equipment cleaning procedures will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its ongoing disposal practices.

Appendix JJ

Pore Water Sample Collection Procedures

Pore Water Sample Collection Procedures

I. Introduction

Pore water samples will be collected from sediment cores to evaluate the concentration and/or partitioning of constituents within the sediment matrix. The protocol presented in this Appendix describes the procedures to be used to collect pore water samples.

II. Materials

Specific to this activity, the following materials (or equivalent) shall be available:

- Site Plan;
- Lexan® tubing (typically 3-inch outside diameter) and end caps;
- PVC core driver system;
- camera;
- Global Positioning System (GPS) equipment (if sampling locations not previously surveyed);
- appropriate sample containers and forms;
- centrifuge;
- stainless steel pressure filtration apparatus; and
- onsite processing laboratory.

III. Sampling Procedures

Locations will be recorded at the time of sampling by GPS if not previously surveyed. A sediment-coring device made up of a 7.6 centimeter (cm) (3-inch outside diameter) Lexan® coring tube will be used with a PVC core driver system to collect sediments. The corer will be driven a minimum of 24 inches into the sediment bed. The distance of core penetration will be recorded. After driving the coring tube to the required depth, the core will be slowly extracted from the bottom of the water body, keeping the core perpendicular. A rubber stopper or other similar device may be utilized to develop a slight vacuum with the corer to help maintain maximum core integrity. A plastic end cap will be used to seal the bottom of the core tube to enclose the sediment as soon as possible after the core is extracted from the water. A plastic cap will be used to seal the top of the core and will be labeled with the site location ID and the word “top.” An observational description of each core will be logged and the core photographed. Cores will be maintained vertically during all handling, transport, and storage steps to minimize disruption.

A minimum of three sediment cores will be collected from each pore water sampling location using the methods described above to provide adequate pore water sample volume and material for additional tests that may be conducted (e.g., sequential batch tests). Additional sediment cores may be required depending on the number of analyses to be performed. It is estimated that two sediment cores (after removal of aliquots for bulk sediment analyses) will provide 235 to 470 milliliter (ml) of pore water when pore water constitutes 50% of the bulk sediment, and when 25 to 50% of the available pore water can be collected from the centrifugation and filtration processing (see filtration description below).

IV. Sample Processing Procedures

Prior to their processing, cores will be stored at the approximate temperature of the water body during core collection. The actual storage temperature will be recorded. Cores will be processed as soon as possible, but no later than one working day after sample collection.

At the onsite sample processing area, the cores will be drained of overlying surface water through a hole drilled/cut in the core tube and extruded using a piston-driven core extruder. After removal of the overlying water, each core will be measured from the top of the sediment surface to 0.5 cm (0.2 inches) and cut using a pre-cleaned hacksaw. Due to concerns regarding mixing with the overlying water and other boundary conditions at the sediment/water interface, the top 5 cm of the sediment will be extruded and not used to determine pore water constituent concentrations. This upper 5 cm can, however, be used to provide a corresponding measurement of surface layer constituent concentrations for a comparison to the underlying sediment that will be centrifuged. The top 5 cm of sediment from each core will be extruded into a pre-cleaned stainless steel bowl, mixed using stainless steel utensils, placed in sample jars, and transferred to a laboratory (onsite or offsite) for analysis.

The 5-cm to 30-cm sediment interval will next be extruded into a clean stainless steel mixing bowl and homogenized using stainless steel utensils (see Section 4.3 of EPA, 2001). Assuming that sediments from two of the three 3-inch Lexan® tubes are used for sediment collection at each location, the 25-cm long segments used will have an approximate total volume of 2,250 ml. Previous sampling of Silver Lake sediments, for example, indicated an average bulk density of 71 pounds per cubic foot and an average specific gravity of 2.4. Assuming no significant change in density as a result of compaction during sample collection and handling, the sample would contain approximately 600 grams of solids and 1,750 grams of water (although only half of this is expected to be available for extraction). After homogenization, sample aliquots for analysis of sediment constituent concentrations will be prepared.

The homogenized bulk sediment will be centrifuged in 285 ml stainless steel centrifuge bottles, four equal mass aliquots (approximately 250 grams each +/- 1 gram) will be placed into stainless steel centrifuge bottles. An IEC Centra 8R centrifuge with a #216 swinging bucket rotor and 378S cups (or equivalent) shall be utilized for the centrifugation. The sealed bottles will be placed in the centrifuge and centrifuged at 2,500 revolutions per minute (rpm) for 20 minutes at the temperature of the core when collected. This speed is 90% of the maximum speed recommended by the manufacturer for the selected equipment.

The resulting supernatant will be transferred from the centrifuge tubes using disposable pipettes or glass syringes into a 750 ml stainless steel pressure filtration apparatus. The supernatant will then be pressure-filtered through a pre-cleaned steel 120 ml diameter, 0.7 micrometer glass-fiber filter (e.g., Whatman GF/F) using high-purity nitrogen gas pressurized up to 15 pounds per square inch gauge (psig). The glass-fiber filter will be pre-cleaned by the laboratory by heating to a minimum of 450° Celsius for 1 hour in a PCB-free environment (supplied and stored in Petri dishes). The laboratory will run at least one filter blank per batch of filters cleaned to confirm the lack of PCBs.

The first 10 to 15 ml of filtrate passing through the filter will be discarded to allow the filter media to establish equilibrium with the sample. The remaining pore water filtrate from each sample location will be collected into a single 500 ml glass sample bottle. This sample bottle will be used to fill the analytical sample containers that will be submitted for analysis (no further filtration by the laboratory). The temperature and approximate volume of sample collected will be recorded. Samples will be stored at 4° Celsius until analysis. Analysis will be conducted within 48 hours of extraction.

Poor centrifugal separation of samples with high levels of fine particles may require multiple filters. Up to three filters may be used to complete the filtration of each pore water sample. The use of nitrogen at a pressure of up to 15 psig in concert with the large diameter of the filters is expected to complete the filtration with one filter. The actual number of filters used for each sample will be recorded. Decontamination of the filtration unit between the replacement of clogged filters for each sample will not be necessary as the constituents will have equilibrated with the surfaces of the housing. Each filter will have the first 10 to 15 ml of filtrate discarded.

The filtration unit will be decontaminated with an Alconox-water solution followed by a four-step rinsing procedure of acetone, hexane, acetone, and deionized water between each sample location. Between each sample, the centrifuge bottles will be emptied of the compacted sediments; brush-cleaned with an Alconox-water solution; and then rinsed in sequence with tap water, hexane, isopropyl alcohol, and deionized water.

V. Reference

EPA, 2001. *Methods for Collection, Storage, and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual*. EPA-823-B-01-002, 20

Appendix KK

Sequential Batch Leach Test Procedures

Sequential Batch Leach Test Procedures

I. Introduction

The sequential batch leach test is performed on residual sediments remaining in centrifuge tubes after centrifugation during pore water analysis procedures. Pore water sample collection procedures are described in Appendix JJ. The protocol presented in this Appendix describes the procedures to be used to conduct sequential batch leach tests.

II. Materials

Specific to this activity, the following materials (or equivalent) shall be available:

- distilled/deionized (DDI) water;
- appropriate sample containers and forms;
- centrifuge; and
- stainless steel pressure filtration apparatus.

III. Procedures

1. Collect pore water samples from sediment cores as described in Appendix JJ.

2. After the supernatant has been removed from the centrifuge for further processing in the pore water analysis, DDI water will be added to each of the centrifuge tubes to bring the final water-to-solids ratio to approximately 4:1 (by mass). Depending on the mass and bulk density of sediment initially placed in each tube, the extraction of material, re-mixing, and re-addition of the sediments into the centrifuge may be required. Assuming that centrifugation results in a 50% solids content, the addition of 100 grams of the sediment mixture with an additional 150 milliliters (ml) of DDI water would achieve the desired 4:1 ratio in each centrifuge tube.

3. After addition of the DDI water, the centrifuge tubes should be weighted to ensure that opposing pairs of tubes in the centrifuge are within a few grams. The centrifuge tubes are then capped, sealed, and placed in a tumbler or similar device to provide for thorough agitation. After 24 hours of tumbling, the centrifuge tubes are removed and placed in the centrifuge at 2,500 revolutions per minute (rpm) for 20 minutes.

4. Filtration procedures are similar to those used for pore water analysis. As with the pore water samples, the resulting supernatant will be transferred from the centrifuge tubes using disposable pipettes or glass syringes into a 750 ml stainless steel pressure filtration apparatus. The supernatant will then be pressure-filtered through a pre-cleaned stainless steel 120 ml diameter, 0.7 micrometer glass-fiber filter using high-purity nitrogen gas pressurized up to 15 pounds per square inch gauge (psig).

5. The first 10 to 15 ml of filtrate passing through each new filter will be discarded to allow the filter media to establish equilibrium with the sample. If poor centrifugal separation of samples with high levels of fine particles requires multiple filters, decontamination of the filtration unit between the replacement of clogged filters for each sample will not be necessary as the constituents will have equilibrated with the surfaces of the housing.

6. The remaining pore water filtrate from each sample location will be collected into a single 500 ml glass sample bottle. This sample bottle will be used to fill the analytical sample containers that will be submitted for analysis (no further filtration by the laboratory). The temperature and approximate volume of sample collected will be recorded. Samples will be stored at 4° Celsius until analysis.

The procedure, starting with Step 2 (addition of DDI water to produce a 4:1 water to solids ratio), is then repeated three more times.

Appendix LL

Seepage Meter Usage Procedures

Seepage Meter Usage Procedures

I. Introduction

A seepage meter is used to collect groundwater that is flowing through the sediments and into a water body. The seepage meter is placed into the sediments for a known period of time; the volume of water collected in an expandable bag attached to the meter is proportional to the surface area covered by the meter and the groundwater discharge rate. The times of instrument installation and sample collection are recorded, as is the volume of water collected in the expandable bag. A volumetric flow rate can then be determined from these measurements. A seepage velocity is determined based on the change in the volume of water collected in the bag over time and the cross-sectional area of the meter exposed to the sediment bed.

II. Materials

The following materials will be available, as required, during seepage meter installation and water collection:

- personal protective equipment (PPE), as required by the Health and Safety Plan (HASP);
- boat and/or waders;
- diving equipment, as necessary;
- Buoy marker, rope, anchor (cinder block);
- seepage meter, protective crate, elastic cord;
- water collection bags (polyethylene);
- flexible tubing and clamps;
- measuring tape;
- graduated cylinder;
- funnel and tubing;
- field notebook and camera;
- waterproof watch;
- waterproof marker; and
- potable water.

III. Seepage Meter Construction

The most commonly used seepage meter is referred to as a Lee Meter. The seepage meter, as designed by Lee (1977), consists of a cut 55-gallon drum with two fittings cut into the bottom of the drum. The surface area of a 55-gallon drum is approximately 405 square inches (2,600 square centimeters). The size of the drum being checked and the area used in calculation of the seepage rate can be adjusted as required by site conditions. Two small (0.5 to 1 inch) holes are cut into the drum bottom and leak-proof fittings affixed in these small holes. On one hole, a pressure relief valve is installed. On the other hole, a flared fitting and a valve are affixed so that flow through the fitting can be turned on and off, and an expandable bag can be attached to allow measurement of changes in water volume. The accumulation bag will also be fitted with a valve, as well as a quick-release fitting to attach it to the flared fitting. Several varieties of bags designed for medical applications are well-suited to this application and come with suitable fittings attached. The partial drum/seepage meter is inverted and pushed into the bottom sediments and net change in water volume is monitored through time. A typical Lee seepage meter design is shown on attached Figure LL-1.

IV. Field Procedures

The following are the general steps for seepage meter installation (it should be noted that some of the specific procedures related to the coupling and uncoupling of the collection bag are subject to modification based on the fitting or clamps used in the final construction of the meter assembly):

1. If a boat is needed, position it in the desired location.

2. Lower the seepage meter into the water and invert the meter below the water to eliminate possible air entrapment. In water greater than approximately 4 to 5 feet deep, a diver may be required. Lower the meter to the bottom of the water body.

3. Push the meter (with tubing and bag **not** attached) approximately 8 to 10 inches into undisturbed sediment. Tilt the seepage meter slightly so that the connection between the meter and collection bag will be higher than the rest of the seepage meter (this will minimize possible air bubbles which might dislodge the meter). Mark the seepage meter location with a labeled buoy anchored adjacent to the meter. Record the depth of the water and the general nature of the sediments at the seepage meter location.

4. Once the meter is in place for at least 48 hours (preferably 72 hours), allowing pore pressures to equalize and air to vent, the collection bag will be attached. Above the surface, purge the water collection bag of all air and water. Place a known volume of water (e.g., 200 milliliters [ml]) in the collection bag. A funnel with tubing to fit the inlet valve can be used. Close the inlet valve to the bag. Remove the funnel tubing from the valve and replace with tubing for connection to the meter. The tubing is clamped prior to attachment to the meter. Record the volume of water added. Attach the collection bag to the fitting on the meter. Unclamp the flexible tubing so that water from the seepage meter may enter the collection bag. Record the sampling initiation time.

5. Attach the protective crate over the meter and bag assembly.

6. Return approximately one week later for collection. The collection time may vary depending on collection bag size and seepage rate.

For water collection, the steps are as follows:

7. If a boat is needed, position it in the desired location. Enter water and locate the meter.

8. Observe the general area for notable conditions (e.g., turbidity, movement or tilting of the meter, groundwater leakage around the meter).

9. Remove protective crate.

10. Check the water collection bag for obvious changes in water volume. If the collection bag requires changing (e.g., if it is between 25 and 75% of its capacity), clamp the flexible tubing and remove the collection bag. If several weeks of monitoring result in continued low groundwater flux rates (i.e., collection bag is less than 25% of its capacity), the monitoring need not be continued.

11. If desired, install new collection bag following procedures given above and replace the protective crate.

12. Return to the surface with collection bag.

13. Record the volume of water collected, as well as collection time. If upon collection the collection bag is less than 25 or more the 75% of its capacity, then the measurement shall be repeated for a longer/shorter duration, or with a smaller/larger collection bag, as appropriate.

14. Compute the volumetric flow rate from the change in the volume of water in the collection bag divided by the time of collection (see Section V below).

V. Calculations

To determine the specific discharge or apparent groundwater velocity (i.e., the rate of discharge of groundwater per unit area of a porous medium perpendicular to the direction of flow), divide the volumetric flow rate by the cross-sectional area:

$$\textbf{Specific Discharge} = \frac{__V \times __t}{A}$$

Where ___**V** equals the measured change in volume of the water in the collection bag (in cubic centimeters);

___**t** equals the time since the collection bag was attached; and

A equals the cross-sectional area (approximately 2,600 square centimeters if a cut 55-gallon drum is utilized for construction of the seepage meter).

VI. Reference

Lee, D.R. 1977. "A Device for Measuring Seepage Flux into Lakes and Estuaries." *Journal of Limnology and Oceanography*. January 1977, pp 140-147.

Figure LL-1

Typical Seepage Meter

Protective Crate

Flexible Plastic Collection Bag

Valve

End Section of 55 Gallon Drum

Vent (Valve)

River/Lake Bottom

GENERAL ELECTRIC COMPANY
PITTSFIELD, MASSACHUSETTS
FIELD SAMPLING PLAN/ QUALITY ASSURANCE PROJECT PLAN

TYPICAL SEEPAGE METER

06/14/04 SYR-D85-DJH
C:20457010/20457g01.cdr

Appendix MM

Storm-Duration Flow-Composite
Water Sampling Procedures

Storm-Duration Flow-Composite Water Sampling Procedures

I. Introduction

This appendix specifies several types of water sampling/monitoring procedures. These include procedures for collecting storm-duration flow-composite water samples for subsequent chemical analysis; procedures for obtaining velocity profile measurements and flow volumes at selected sampling locations; and procedures for developing the composite samples based on flow volumes.

II. Storm-Duration Flow-Composite Water Sampling for Chemical Analysis

This section specifies the procedures for collecting storm-duration flow-composite water samples for chemical analysis. The methods outlined in this SOP for collecting storm-duration flow-composite water samples have been tailored to the sampling of storm sewer outfalls but can be applied to various sampling scenarios.

Materials

The following materials will be available, as required, during storm-duration flow-composite water sampling.

- Health and safety equipment (as required by the Health and Safety Plan);
- Cleaning equipment (as required in Appendix W);
- Rope;
- Surveyor's rod and/or 6-foot rule;
- Duct tape;
- Open channel flow meter or electromagnetic velocity meter;
- Large glass mixing container;
- Medical-grade silicone tubing;
- Teflon® tubing;
- Teflon® stirring rod;
- Beaker or equivalent glass measuring device;
- Field notebook;
- pH meter;
- Appropriate blanks (trip), if necessary;

- Appropriate sampling containers and forms;
- Appropriate preservatives (as required);
- Coolers with ice or ice packs (e.g., blue ice); and
- Appropriate automatic water sampler as specified in the project-specific work plan, such as a Teledyne ISCO Sequential Sampler.

<u>Procedures</u>

A. The following procedures will be used to obtain samples:

Step 1 - Identify water sampling location on appropriate sampling log sheet (Attachment E-1) and/or field notebook along with other appropriate information.

Step 2 - Don health and safety equipment (as required by the Health and Safety Plan).

Step 3 - Clean the sampling equipment in accordance with the procedures in Appendix W.

Step 4 - Install the open channel flow meter or assemble the electromagnetic velocity meter to be used. Note that if an electromagnetic velocity meter is to be used in place of the open flow channel flow meter, water channel width and depth measurements will need to be collected and recorded with every velocity measurement.

Step 5 - Calibrate the open channel flow meter or the velocity meter as provided by the manufacturer.

Step 6 - Assemble the appropriate automatic water sampler adjacent to the sample location. Make sure that the sampling unit is securely positioned prior to operating. Units positioned in public areas should be secured to a non-removable structure to discourage theft.

Step 7 - Secure the Teflon® sample tubing at the water sampling location within the water column as provided in the project-specific work plan.

Step 8 - Program the automatic water sampler to collect samples at the frequency (i.e., sample collection interval) outlined in the project-specific work plan. Sample collection intervals should be consistent throughout the sample collection process.

Step 9 - Initiate flow monitoring activities using the open channel flow meter or an electromagnetic velocity meter (see Section III below for procedures associated with the use of an electromagnetic velocity meter).

Step 10 - Initiate sampling activities in accordance with the requirements outlined in the project-specific work plan.

Step 11 - Collect samples for analyses of volatile organic compounds (VOCs) and oil & grease samples prior to initiating composite grab sampling.

When sampling for VOCs, water samples should be collected at the sample collection point (e.g., location where water sampler collection tubing is located) as a discrete grab sample just prior to initiating the composite grab sampling. A Teflon® lined bailer should be used to retrieve an appropriate sample volume, which should be immediately transferred to 40 mL vials with Teflon® liners.

When sampling for oil and grease, surface water should be collected at the sample collection point (e.g., location where water sampler collection tubing is located) as a discrete grab sample prior to initiating the composite grab sampler. Oil and grease samples should be collected directly in 1-liter amber glass vessels with Teflon® liners.

Step 12 - If an electromagnetic velocity meter is being utilized, continue to collect and record velocity and water channel width and depth measurements throughout the sampling period.

Note: Sample collection equipment should be inspected, installed, and tested prior to the sample collection period to ensure all equipment is in good operating condition.

B. The following procedures will be used to develop a storm-duration flow-composite water sample:

Step 1 - If an open channel flow meter was used, retrieve the flow information from the unit.

If a velocity meter was used, calculate the flow volume for each velocity measurement collected during the sampling intervals (refer to Section III below for procedures associated with the calculation of flow using an electromagnetic velocity meter).

Step 2 - Calculate the total flow that was recorded during each sample collection interval using the flow monitoring information developed in Step 1 above.

Step 3 - Calculate the percent flow contribution for each sample interval based on the amount of flow recorded within the sample interval. The percent flow contribution is the recorded flow for a sample interval divided by the total flow recorded over the sample period.

Step 4 - Based on the total volume needed for the analysis, determine the appropriate sample volume from each sample interval needed to develop the composite sample. The appropriate volume is the percent flow contribution multiplied by the required sample volume.

Step 5 - Using a beaker or graduated cylinder measure the appropriate sample volume from each sample collection vessel (see Step 4) and transfer the volume into a mixing container with sufficient capacity to hold the entire composite sample volume.

Step 6 - Repeat Step 5 for all sample intervals (i.e., each sample collection vessel) collected during the sampling event.

Step 7 - Mix the entire sample volume with the Teflon® stirring rod and transfer the appropriate volume into the laboratory sample jar. (Volatile samples and oil and grease samples will not be homogenized.) Preserve samples as specified in Table 1 of the FSP/QAPP.

Step 8 - The sample collection order (as appropriate) will be as follows:

1. VOCs;
2. TOC;
3. SVOCs;
4. Metals and cyanide; and
5. Others.

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

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

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

Step 12 - Label all sample containers as appropriate, as discussed in Appendix L.

Step 13 - After sample containers have been filled, fill a beaker or glass container with the water sample and measure the pH, as discussed in Appendix O.

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

Step 15 - Handle, pack, and ship the samples in accordance with the procedures in Appendix L.

III. Velocity Profile Measurement Procedures

The following materials will be required for this activity:

- Health and safety equipment (as required by the Health and Safety Plan);
- Field notebook and pen;
- Calculator;
- Rope;
- Surveyor's rod;
- Duct tape;
- Measuring tape; and
- Electromagnetic velocity meter.

The following procedures will be used to determine the velocity profile at channel cross sections:

Step 1 - Don personal protective equipment (as required in the Health and Safety Plan).

Step 2 - Extend rope across the channel.

Step 3 - Measure the width of the channel, then divide and mark into equally spaced measurement locations. For channels less than 5 feet in width, a minimum of three measurements should be collected. For channels less than 30 feet in width, the spacing should be 5 feet. For channels between 30 feet and 100 feet in width, the spacing should be 10 feet; and for channels greater than 100 feet in width, the spacing should be 20 feet.

Step 4 - Calibrate velocity meter as per manufacturer's specifications.

Step 5 - Lower the surveyor's rod and measure and record the water depth to the nearest 0.1 foot at each measurement location.

Step 6 - Velocities will be determined using the two-point method. Attach the velocity meter probe to the surveyor's rod, measure, and record the velocity in feet per second at depths equaling 0.2 and 0.8 times the total river depth at each measurement location. If the measurement of velocities requires wading into the channel, position the probe upstream of wading field personnel to avoid disruption of flow during measurement. Average the two velocity measurements to obtain the average velocity for that vertical section.

Step 7 - Record all measurements in field notebook.

Step 8 - Calculate the channel flow rate by multiplying the average velocity reading for a particular vertical section times the area represented by the portion of the total cross-section extending half-way to the adjacent vertical sections (i.e., the velocity-area method). The total flow rate is the sum of the flow of the partial sections.

$$Q_T = V_1 A_1 + V_2 A_2 + \ldots + V_n A_n$$

Where: Q_T = Total flow in cubic feet per second

$V_{1\text{-}n}$ = Average velocity for a vertical section in feet per second

$A_{1\text{-}n}$ = Cross-section area extending half-way to the adjacent vertical sections in square feet.

IV. Survey

A field survey control program will be conducted using standard instrument survey techniques to document the water sampling locations when necessary to have record of the exact location. Generally, to accomplish this, a local control baseline will be set up. This local baseline control may then be tied into the appropriate vertical and horizontal datum such as the National Geodetic Vertical Datum of 1929 and the State Plane Coordinate System.

V. Equipment Cleaning

Equipment cleaning will occur at the beginning of each sampling event and between each sampling location as described in Appendix W.

VI. Disposal Methods

Rinse water, personal protective equipment (PPE), and other residuals generated during the equipment cleaning procedures will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its ongoing disposal practices.

Appendix NN

Soil Vapor Point Installation and Tracer Gas Leak Testing Procedures

Soil Vapor Point Installation and Tracer Gas Leak Testing Procedures

I. General

This protocol provides a list of the necessary equipment and detailed instructions for the installation of soil vapor points, as well as the implementation of tracer gas leak test prior to soil vapor sampling.

For all vapor point installations, the following cautions should be considered prior to installing a soil vapor point:

- When installing any vapor point, be mindful of utilities that may be in the area. Always complete utility location, identification and marking before installing ports. Be aware that public utility locator organizations frequently do not provide location information within buildings so alternative lines of evidence (e.g., GPR, magnetometers, air knifing) must be used. If the driller is concerned about a particular location, consult the project manager about moving it to another location. Don't be hesitant to use Stop Work Authority. If something doesn't seem right stop and remedy the situation.

- Sampling personnel should not handle hazardous substances (such as gasoline), permanent marking pens, wear/apply fragrances, or smoke cigarettes/cigars 24 hours before and/or during the sampling event.

- Vapor point installation should be completed 24 hours before or after any indoor air sampling to avoid cross-contamination of the indoor air samples.

- Field personnel will properly seal the vapor probe at the slab surface to prevent leaks of atmosphere into the soil vapor probe during purging and sampling. Temporary points should be fit snug into the pre-drilled hole using Teflon® tape or modeling clay and sealed with hydrated bentonite, clay not containing volatile organic compounds (VOCs), or natural bees wax at the surface. If this is not done properly, the integrity of the sample port may be compromised.

- Modeling clay or other materials used should only be obtained from an approved source and should not be purchased off the shelf from an unapproved retail source. Data indicate that some modeling clays may contain significant levels of VOCs that can affect sample results

- If possible, have equipment shipped two to three days before the scheduled start of the sampling event so that all materials can be checked. Order replacements if needed.

II. Permanent Vapor Point Installation

Permanent soil vapor points (other than sub-slab vapor points, discussed in Section IV below) should be installed using a hand auger, air knife (if utility clearing is required), or drill rig equipped with direct push capabilities. Using a photoionization detector (PID) capable of reading parts per billion (ppb), removed soils should be logged (if possible) and screened for VOCs during the point installation to identify potential contamination that could impact sample data. If using an air knife for utility clearing, soils should be removed via hand auger or direct push beginning 1 foot above the top of the zone to be screened, (generally this would be approximately 2.5' above the water table). All well construction details should be described on Attachment NN-1. The equipment and procedures required for the installation of the permanent vapor point are presented below.

A. Installation Equipment and Materials

- Appropriate personal protective equipment (PPE) (as presented in the site-specific HASP and the Job Safety Analysis [JSA]))
- Boring equipment: hand auger, air knife with Vac-Truck, and/or drill rig equipped with direct push capability;
- 3/8" outer diameter Teflon®-lined polyethylene tubing;
- 0.5' long stainless steel mesh screen;
- PID capable of parts per billion (ppb) readings (e.g., ppbRAE);
- Swagelok® stainless steel adapter to fit Teflon® tubing;
- Appropriate-sized open-end wrench (typically 9/16-inch, 1/2-inch , and 3/4-inch);
- Clean filter silica well sand (#2 or larger);
- Granular bentonite and non-shrink grout cement;
- Field notebook and/or field forms (Attachment NN-1); and
- 6 to 8-inch bolting road box (if installed outside of a high traffic building).

B. Installation Procedures

Step 1 - If the depth to the water table has been predetermined, wearing proper PPE, advance the borehole tooling to 1 foot above the highest water table. If the use of an air knife is required to clear the location of utilities to 5 feet, and the vapor point is to be installed anywhere between 0 and 6.5 feet below ground surface, the point maybe air knifed to 1 foot above the top of the intended screen zone. For example, if the point is to be installed from 6 to 6.5' below ground surface, air knife to a maximum of 5' below ground surface. The additional distance should be hand augured or direct pushed to set the point at the desired screen interval. If the boring contacted the water table, backfill the borehole to 1 foot above the water table with granular

bentonite (this step is not required if the depth to groundwater was predetermined). Record in the field log the soil type and PID readings that were collected from soil cuttings removed during installation. .

Step 2 - After reaching the desired depth, install a 1/2-inch outside diameter (OD) stainless steel (SS) screen connected to 3/8-inch OD Teflon®-lined polyethylene tubing. Place the screen and tubing into the open borehole or inside the direct push drill pipe along with the required amount of clean silica sand (as specified in Step 3).

Step 3 - Install the screen such that there are 3 inches of clean silica sand above the screen and 3 inches of such sand below the screen, along with the clean silica sand surrounding the screen, so that there is total of 12 inches of such sand.

Step 4 - Withdraw the drill pipe and place 0.5 foot of dry granulated bentonite on top of the clean sand layer, followed by a hydrated powder bentonite slurry to one foot below ground surface.

Step 5 - Install and cement in a bolting water tight road box around the vapor tubing.

Step 6 - Fill the remaining annulus of the boring with non-shrink grout cement to 0.5-foot below ground surface.

Step 7 - Place the 3/8-inch Swagelok® adapter over the Teflon® tubing with Swagelok® two-way valve or plug, leaving enough tubing to reach a SUMMA® canister (2 to 3 feet). Let the point equilibrate and cement cure a minimum of 24 hours before leak testing and sampling.

Step 8 - See Soil Vapor Sampling SOP (Appendix I) for sample collection procedure.

III. Temporary Sub-Slab Vapor Point Installation

Temporary sub-slab soil vapor probes are installed using equipment and procedures that allow the point to be installed quickly and abandoned after an initial sample is collected. These procedures are not recommended if the probe is to be sampled more than once. Under those conditions, refer to Section IV below for permanent sub-slab soil gas installations.

A. Installation Equipment and Materials

The equipment required to install a temporary sub-slab vapor point is presented below:

- Appropriate personal protective equipment (PPE) (as required by the HASP and the JSA);
- Hammer drill (Hilti, Bosch Hammer, or equivalent);
- 1/2 inch-diameter concrete drill bit (drill bit length contingent on slab thickness);

- Hand tools including open-end wrench (typically 9/16-inch), pliers, channel lock pliers, etc;
- 1/4-inch or 3/8-inch OD tubing (Teflon®, or Teflon®-lined polyethylene);
- Teflon® tape;
- Clean cut-resistant gloves;
- Nitrile gloves;
- Hydrated bentonite, VOC-free modeling clay that complies with ASTM D4236 (McMaster Carr 6102T11 recommended) or natural bees wax to seal drill hole (see Installation procedures, Step 10 note discussing precautions when using heated bees wax);
- Whisk broom and dust pan;
- Bottle brush;
- Ground fault circuit interrupter (GFCI);
- Extension cords rated for amperage required for hammer drill;
- Shop vacuum with clean HEPA particle filter and disposable bag;
- Rigid wire with a small 90-degree bend on the end (or equivalent);
- Plastic sheeting; and
- Hot plate and melting pot (If using bees wax).

B. Installation Procedures

Step 1 - Wearing proper PPE, complete a Utility mark-out prior to drilling activities.

Step 2 - Make a copy of Attachment I-1, and update required fields throughout the installation process remove, only to the extent necessary, any covering on top of the slab (e.g., carpet).

Step 3 - Lay down plastic sheeting to keep the work area clean. Check to make sure shop vacuum is working properly and all filters are correctly installed and functioning.

Step 4 - Drill a 1/2-inch-diameter hole into the concrete slab using the electric hammer drill. Do not fully penetrate the slab at this time. If possible stop drilling approximately 1 inch short of penetrating the slab.

Step 5 - Use the shop vacuum, and/or bottle brush with dust broom to clean up the work area and material that may have fallen into and around the drill hole.

Step 6 - Advance the ½--inch drill bit the remaining thickness of the slab and approximately 3 inches into the sub-slab material to create an open cavity. Additionally, note (if possible) from the drill cuttings any evidence for the types of materials in the immediate sub-slab – i.e. moisture barriers, silt, sand, gravel, shrinkage gap.

Step 7 - Use the bottle brush, whisk broom, and dust pan to quickly clean surface around and within the top of the hole. The hole should not be left open for any extended length of time to ensure that VOCs below the slab do not migrate into indoor air. Do not use the shop vacuum to clean out the drill hole after the full thickness of the slab has been penetrated.

Step 8 - To ensure that the hole is open and remains clear for sampling, either re-drill the ½-inch hole or using a piece clean sample tubing, push down to the bottom of the open annulus. Using a piece of clean rigid wire (or equivalent) with a small 90-degree bend on the end, measure the total length of the slab and penetration depth to make sure that there is a 3-inch void below the slab . Document the approximate slab thickness and total drill depth on Attachment I-1 or field book.

Step 9 - Insert the tubing approximately 2 to 3 inches into the slab; tubing should be wrapped with clean Teflon® tape so tubing will not come in contact with the material beneath the slab. Tubing extended above the slab should be capped or another fitting added so it does not provide a pathway for vapor movement.

Step 10 - Prepare a clean hydrated granular bentonite mixture and apply bentonite at slab surface around the tubing. If bentonite is unavailable, either VOC-free modeling clay (McMaster-Carr #6102T11) or bees wax (usually used when samples are to be collected for over 4 hours) may be used for the temporary seal around the tubing where it enters the slab.

Note: when melting bees wax on a hot plate, the hot plate and melting pot must be paced on a flat non-flammable surface away from all foot traffic. Extra caution should be taken to prevent overheating and burns. When heating or plugged in, the hot plate should not be left unattended. When task is completed the melting pot and hot plate should be left to cool for a minimum of 30 minutes and coned out if possible.

Step 11 - See Section V for Leak Testing and Appendix I for Soil Vapor Sampling procedure.

Step 12 - Following sample collection, remove the tubing and fill up the drilled hole in the slab with hydrated quick-setting hydraulic cement mix. This step must be done carefully to ensure that the abandoned sampling point does not become a preferential flow pathway.

Step 13 - Replace the surface covering (e.g., carpet) to the extent practicable. Sample collection location should be returned to pre-sampling conditions.

IV. Permanent Sub-Slab Vapor Point Installation

Permanent sub-slab soil vapor probes are installed using an electric hammer drill and manual placement of the probe. After a dry fit, the vapor probe is inserted into the hole and grouted with a hydrated quick-setting non-shrink grout mix. The vapor probe is equipped with a plug. The cap is removed and a compression fitting nut and ferrules are used to allow collection of a soil gas sample through Teflon® tubing. The vapor probe and tubing will be purged with a portable sampling pump prior to collecting the soil gas sample.

A. Installation Equipment and Materials

- Appropriate personal protective equipment (PPE) (as required by the HASP and the JLA);
- Hammer drill (Hilti, Bosch Hammer, or equivalent);
- 3/8-inch and 1-inch-diameter concrete drill bit (drill bit length contingent on slab thickness);
- Decontaminated stainless steel vapor probe (typically 1/4-inch inside diameter [ID] stainless steel tubing, 1/4-inch Swagelok® by 1/4-inch Swagelok® female coupling, (or equivalent), 1/4-inch Swagelok® cap); See Figure NN-1;
- Extra 1/4-inch OD Swagelok® front and back compression sleeves (ferrules);
- Stainless steel washers;
- Safety blade box cutter;
- Clean metal pipe cutter with cutting wheel;
- Hand tools including open-end wrench (typically 9/16-inch), pliers, channel lock pliers, etc;
- Teflon® tape;
- Clean cut resistant work gloves;
- Nitrile gloves;
- Quick-setting non-shrink grout cement;
- Distilled water for mixing cement;
- Disposable cups and spoons for mixing grout;
- Spray bottle with distilled water;
- Whisk broom and dust pan;
- Bottle brush;
- Ground fault circuit interrupter (GFCI);
- Surge strip and extension cords rated for amperage required for hammer drill;

- Shop vacuum with clean HEPA particle filter and disposable bag; and
- Plastic sheeting.

B. Installation Procedures

Step 1 - Wearing proper PPE, complete a Utility mark-out prior to drilling activities.

Step 2 - Make a copy of Attachment I-1, and update required fields throughout the installation process. Assemble the sample port assembly as shown on Figure NN-1 (stainless steel tubing, stainless steel Swagelok® coupling, Swagelok® nut). Teflon® tape should never be used with Swagelok® connections.

Step 3 - Using the safety blade box cutter remove, only to the extent necessary, any covering on top of the slab (e.g., carpet). Lay down plastic sheeting to keep the work area clean. Check to make sure shop vacuum is working properly and all filters are correctly installed and functioning.

Step 4 - Advance the 1-inch drill bit approximately 1½ inches into the slab. This hole is drilled deep enough to permit the top of the coupling to be set flush with the slab when the 3/8-inch probe is inserted into the 3/8-inch hole drilled under Step 7 below. Clean up cuttings with shop vacuum, bottle brush, and dust pan.

Step 5 - Drill a 3/8-inch diameter hole into the concrete slab using the electric hammer drill. Do not fully penetrate the slab at this time. Stop drilling approximately 1 inch short of penetrating the slab. Use the shop vacuum, and/or bottle brush with dust broom to clean up the work area and material that may have fallen into and around the drill hole.

Step 6 - Advance the 3/8-inch drill bit the remaining thickness of the slab and approximately 3 inches into the sub-slab material to create an open cavity. Using a piece of clean rigid wire (or equivalent) with a small 90-degree bend on the end, measure the total length of the slab and penetration depth to make sure that there is a 3-inch void below the slab. Document the approximate slab thickness and total drill depth on Attachment I-1 or field book. Additionally, note (if possible) from the drill cuttings any evidence for the types of materials in the immediate sub-slab – i.e. moisture barriers, silt, sand, gravel, shrinkage gap.

Step 7 - Use the bottle brush, whisk broom, and dust pan to quickly clean surface around the top of the hole. The hole should not be left open for any extended length of time to ensure that VOCs below the slab do not migrate into indoor air. A shop vacuum can be used to lightly clear the cutting from the 3/8'' hole. Caution must be taken not to create a seal between the vacuum and floor surface. Doing so will prevent any potential soil vapor from being pulled into the indoor air.

Step 8 - Using an assembled sample probe assembly as shown on Figure NN-1, test fit the components so that the proper length of 3/8-inch probe and depth of the 1-inch hole provides enough space for the stainless steel coupling. Adjust so that the coupling will lie flush with the slab surface and does not create a tripping hazard.

Step 9 - Re-check the measured depth (to confirm that it matches the measurement collected in Step 6) using the piece of clean rigid wire (or equivalent) that has a small 90-degree bend on the end. If it is less than the required 3 inches below the slab, push the wire down into the sub-surface or, if necessary, re-drill the 3/8-inch hole to ensure that the void remains clear.

Step 10 - Wrap the sample probe assembly with Teflon® tape, to the extent necessary, for a snug fit of the assembly and hole. Teflon® tape or stainless steel washers can also be used to achieve the proper depth of the sample port assembly. Ensure that Teflon® tape or stainless steel washers do not interfere with the cement that will be used to permanently fix and seal the sample probe.

Step 11- Prepare a mixture of non-shrink cement and water according to the manufactures directions in a disposable cup using a plastic spoon for mixing.

Step 12 - Before cementing in the sample probe, moisten the bore hole with the spray bottle to provide better adhesion

Step 13 - Cement in the sample probe using the plastic spoon to apply the cement into the annular space between the coupling and the 1-inch drill hole

Step 14 - Replace the surface covering (e.g., carpet) if warranted. Sample collection location should be returned to pre-sampling conditions to the extent feasible given the presence of a permanent probe.

Step 15 - All permanent sub-slab sample probes should be allowed to cure and equilibrate for a minimum of 24 hours before proceeding to sample collection.

Step 16 - See Soil Vapor Sampling SOP (Appendix I) for sample collection procedure.

V. Tracer Gas Evaluation and Testing

When collecting subsurface vapor samples as part of a vapor intrusion evaluation, a tracer gas serves as a quality assurance/quality control method to verify the integrity of the vapor port seal and the numerous connections comprising the sample train. Without the use of a tracer, verification that a soil vapor sample has not been diluted by ambient or indoor air is difficult.

This standard operating procedure (SOP) focuses on using helium as a tracer gas. However, depending on the nature of the contaminants of concern, other compounds can be used as a tracer including sulfur hexafluoride (SF6), butane and propane (or other gases). In all cases, the protocol for using a tracer gas is consistent and includes the following basic steps: (1) enrich the atmosphere in the immediate vicinity of the sample port where ambient air could enter the sampling train during sampling with the tracer gas; and (2) measure a vapor sample from the sample tubing for the presence of elevated concentrations (> 10%) of the tracer. A new plastic pail, bucket, garbage pail can serve to keep the tracer gas in contact with the port during the testing.

There are two basic approaches to testing for the tracer gas:

1. Include the tracer gas in the list of target analytes reported by the laboratory; and/or

2. Use a portable monitoring device to analyze a sample of soil vapor for the tracer prior to sampling for the compounds of concern. (Note that tracer gas samples can be collected via syringe, Tedlar bag, etc. They need not be collected in SUMMA® canisters or minicans.)

This SOP focuses on monitoring helium using a portable sampling device, although helium can also be analyzed by the laboratory along with other volatile organic compounds (VOCs). Real-time tracer sampling is generally preferred as the results can be used to confirm the integrity of the port seals prior to formal sample collection. During the initial stages of a subsurface vapor sampling program, tracer gas samples should be collected at each of the sampling points. If the results of the initial samples indicate that the port seals are adequate, the number of locations at which tracer gas samples are used in future monitoring rounds maybe reduced. At a minimum, at least 5% of the subsequent samples should be supported with tracer gas analyses. When using permanent soil vapor points as part of a long-term monitoring program, the port should be tested prior to the first sampling event. Tracer gas testing of subsequent sampling events may often be reduced or eliminated unless conditions have changed at the site. Soil gas port integrity should certainly be rechecked with Tracer gas if land clearing/grading activities, freeze thaw cycles, or soil desiccation may have occurred. Points should also be rechecked if more than 2 years have elapsed since the last check of that port.

A. Installation Equipment and Materials

- Appropriate PPE for site (as required by the HASP);
- Helium (laboratory grade);
- Regulator for helium tank;
- Shroud (plastic bucket, garbage can, etc);
  - The size of the shroud should be sufficient to fit over the sample port.
  - The shroud will need to have three small holes in it on the sides (one for the helium detector probe, one for the sample train tubing, and one for the helium line), as described further below.

- The shroud should ideally enclose the sample port and as much as possible of the sampling train.

- Helium detector capable of measuring from 1 - 100% (Dielectric MGD-2002, Mark Model 9522, or equivalent);
- Tedlar bags;
- Seal material for shroud (rubber gasket, non-VOCs modeling clay, hydrated bentonite, etc) to keep helium levels in shroud. (Although the sealing material is not in direct contact with the sample if leakage does not occur, sealing materials with high levels of VOC emissions must be avoided, since they could contaminate a sample if a leak occurs.)
- Sample logs; and
- Field notebook.

Note: Helium is an asphyxiant! Be cautious with its use indoors! Never release large volumes of helium within a closed room! Compressed gas cylinders should be handled with caution; see attachment on the use and storage of compressed gasses before beginning field work. Care should be taken not to pressurize the shroud while introducing helium. If the shroud is completely air tight and the helium is introduced quickly, the shroud can be over-pressurized and helium can be pushed into the ground. Provide a relief valve or small gap where the helium can escape.

Because minor leakage around the port seal should not materially affect the usability of the soil vapor sampling results, the mere presence of the tracer gas in the sample should not be a cause for alarm. Consequently, portable field monitoring devices with detection limits in the low ppm range are more than adequate for screening samples for the tracer. If high concentrations (> 10%) of tracer gas are observed in a sample, the port seal should be enhanced and fittings within the sampling train should be should be checked and/or tightened to reduce the infiltration of ambient air and the tracer test re-administered. If the problem cannot be rectified, a new sample point should be installed or an alternate sampling train used.

B. Tracer Gas Procedure

The procedure used to conduct the helium tracer test should be specific to the shroud being used and the methods of vapor point installation. The helium tracer test can be conducted when using temporary or permanent sampling points and inside or outside a facility. When using the tracer gas within indoor areas you must provide adequate ventilation as helium is an asphyxiant.

Step 1 - Make a copy of Figure NN-2 and Attachment I-1 (from Appendix I), and update required fields throughout the testing process. If testing is performed on a flush permanent sub-slab point, attach Teflon® or Teflon® lined polyethylene sample tubing to the sample point. This can be accomplished utilizing a number of different methods depending on the sample install (i.e., most typically Swagelok® brand compression fittings, but some quick release fittings could also be used etc.). If there is tubing capable of extending more than 2 feet of above ground surface, proceed to Step 2.

Step 2 - Use Figure NN-2 as a guide when completing the following steps. While wearing proper PPE, drill three holes in the shroud (bucket, clean trash can), one hole should be 1/4 to 3/8 inch in diameter and is for the sample tubing to extend from. The two additional holes will be 1/2-inch in diameter, one drilled near the top of one side, and one near the bottom on the other side of the shroud. Each 1/2-inch-diameter hole should then have a valve assembly consisting of a 1/2-inch threaded male fitting and a 1/4 or 3/8-inch quick-connect adapter on opposite ends. The threaded side of the valve should be inserted into the 1/2-inch hole in the shroud. Each threaded end should be fitted with a rubber washer and stainless steel or brass bolt to hold the valve securely against the shroud.

Step 3 - On the valve assemblies, install 6-inches of Teflon®-lined tubing (1/4 or 3/8-inch depending on quick connect size) into each ball valve quick connect and 3 inches of lab grade silicone tubing to the end of the Teflon® tubing not inserted into the valve. The bottom valve will be for the detector and helium OUT and top valve will be for the helium IN. Place the shroud over the sample point and pull the sample tubing through the appropriate hole.

Step 4 - Seal opening at top and bottom of shroud with hydrated bentonite or non-VOC emitting modeling clay. You may place weight on top of shroud to help maintain a good seal with the ground.

Step 5 - Connect helium regulator to silicon tubing on the top valve assembly and helium detector probe into silicone tube on the bottom valve assembly. An additional seal consisting of modeling clay to prevent leaks may be used if the rubber seals are not tight against the sides of the shroud.

Step 6 - Open both ball valves on the shroud, begin to slowly fill shroud with helium allowing atmospheric air to escape.

Step 7 - Use the helium detector to monitor helium concentration within the shroud. Helium should be added until the environment inside the shroud has > 60% helium. Once the desired concentration is reached. Shut off the helium regulator and close both ball valves. Document the concentration within the shroud.

Step 8 - Begin to purge the sample point through the sample tubing into a Tedlar bag using a hand held sampling pump or battery operated pump using a flow regulator. The purge rate should at least match the sample collection rate but not exceed 100 ml/min. Test the air in the Tedlar bag for helium using the portable helium detector. If the point is free of leaks there should be very low helium in the purge air from the soil. The natural concentration of helium in the atmosphere is 0.00052% by volume and there are few if any natural sources of helium to soil gas.

Step 9 - If > 10% helium is noted in purge air, add more bentonite clay or other material to the seal the sample port and repeat the testing procedure. If the seal cannot be fixed, reinstall sample point.

Step 10 - Monitor and record helium level in shroud before, and after tracer test.

Step 11 - Monitor and record helium level in purge exhaust.

Upon successful installation of the soil vapor point and completion of tracer gas evaluation with seal testing, samples maybe collected from the soil vapor point; see Soil Vapor Sampling Procedures for TO-15, TO-13A and TO-17 SOP (Appendix I). The shroud should remain in place throughout the sample collection process, following sampling collection the helium shroud maybe removed but caution should be taken to not stress the sample tubing and any established seals. Additional tracer gas measurements should be collected from the sample tubing and shroud following the completed sampling event. This information should be documented on the sampling forms in Attachment I-1.

Figures NN-1 and NN-2

CITY:SYRACUSE DIV/GROUP:ENVCAD DB:R. BASSETT LD:A.SCHILLING PIC:(Opt) PM:(Reqd) TM:D.ZUCK LYR:(Opt)ON=*;OFF=*REF*
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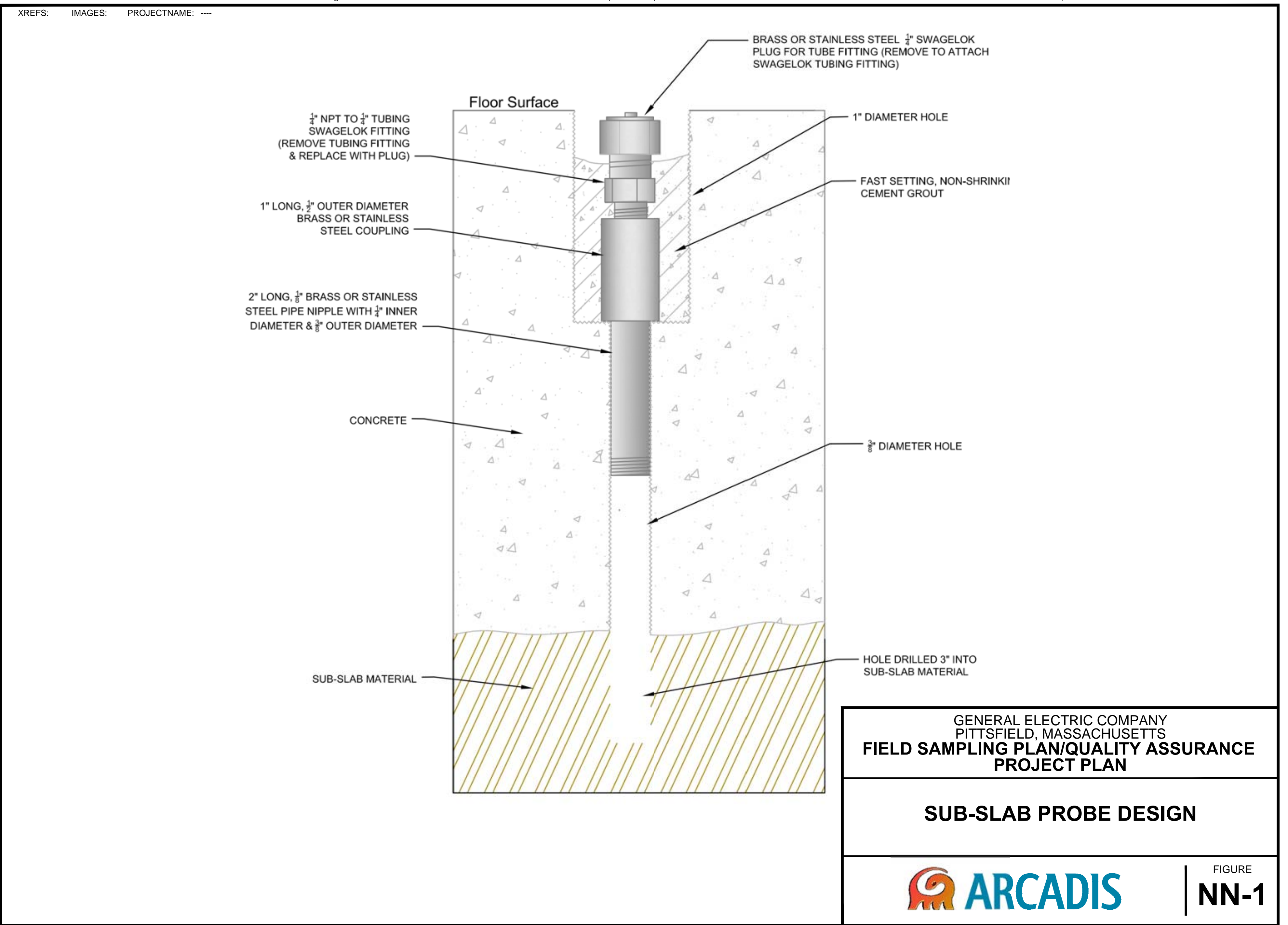

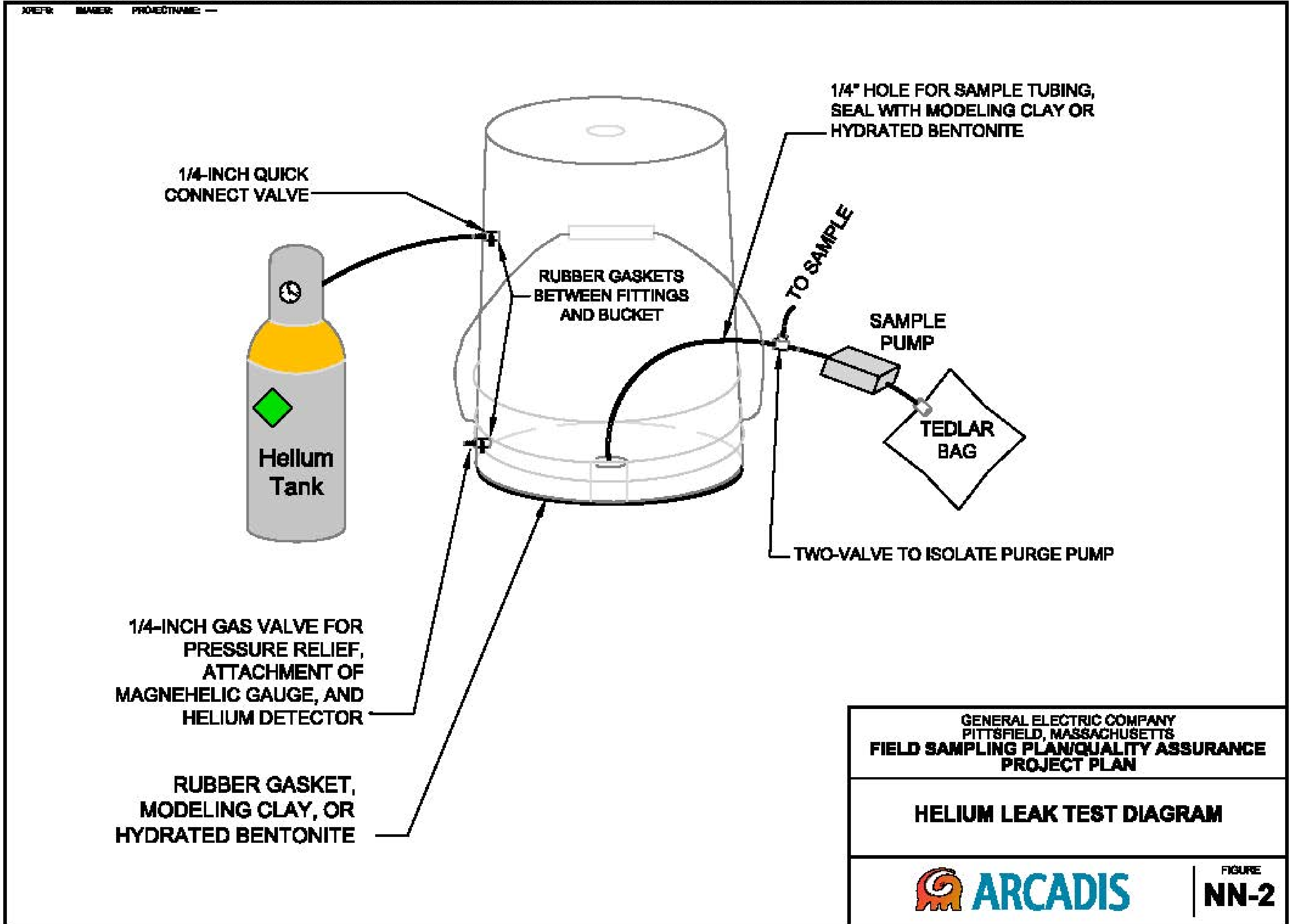

1/4-INCH QUICK CONNECT VALVE
Helium Tank
RUBBER GASKETS BETWEEN FITTINGS AND BUCKET
1/4" HOLE FOR SAMPLE TUBING, SEAL WITH MODELING CLAY OR HYDRATED BENTONITE
TO SAMPLE
SAMPLE PUMP
TEDLAR BAG
TWO-VALVE TO ISOLATE PURGE PUMP
1/4-INCH GAS VALVE FOR PRESSURE RELIEF, ATTACHMENT OF MAGNEHELIC GAUGE, AND HELIUM DETECTOR
RUBBER GASKET, MODELING CLAY, OR HYDRATED BENTONITE
GENERAL ELECTRIC COMPANY
PITTSFIELD, MASSACHUSETTS
FIELD SAMPLING PLAN/QUALITY ASSURANCE PROJECT PLAN
HELIUM LEAK TEST DIAGRAM
ARCADIS
FIGURE
NN-2

Attachment NN-1

Permanent Soil Vapor Point Construction Log

Attachment NN-1

ARCADIS

Permanent Soil Vapor Point Construction Log

Surface completion
Type: " MH

Tubing Length

LAND SURFACE

Cement Type: ________
Concrete (to) ft*

Tubing Size:
________ inch diam.,

___ inch diam. Drilled hole
(to) ft*

Slury (Y / N) (to) ft*

Granular Bentonite:
Hydrated ☐ (to)'
Dry ☐ (to)'

______ ft* Top of Bentonite

______ ft* Bottom of Bentonite

______ ft* Top of screen

Well Screen (inch):
________ , type:
Stainless Steel

Filter Pack: #
(to) ft*

_____inch diam. Drilled hole
(to) ft*

______ ft* Bottom of screen

Project Name and No.: General Electric -

Point ID: ________ Address: ________

Town/City: Pittsfield State: MA

Land-Surface Elevation and Datum:

________ feet ☐ Surveyed ☐ Estimated

Coordinates- Northing: ________ Easting: ________

Installation Date(s): ________

Drilling Contractor:
(Diller/Helper) ________

Installation Method: ________

Equipment Used: ________

Groundwater Information:

Well ID: ________

Well Screen Setting: ________

Static Depth to Water: ________

Vapor Point Purpose: ________

Remarks: Soils: ________

**Measuring Point is Top of Well Casing Unless Otherwise Noted.

Prepared by ________