General Electric Company
Pittsfield, Massachusetts

Field Sampling Plan/ Quality Assurance Project Plan

Volume II of III

Originally submitted September 2000
Revised March 2007

Volume II - Standard Operating Procedures for Field Based Activities

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Appendices

Appendix A

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

Appendix A

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 EnCoreJ , SoilCoreJ , 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 EnCoreJ , SoilCoreJ , 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;
- EnCoreJ 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, EnCoreJ , SoilCoreJ , or equivalent Sampler); and
- Appropriate transport containers (coolers) with water ice and appropriate labeling, packing, and shipping materials.

III. Field Sampling

<u>Option 1 - Collection of Unpreserved Sample</u>

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 - EnCoreJ Sampler

Step 1 - Place EnCoreJ sample container into the EnCoreJ T-handle.

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

Step 3 - Wipe the outside of the EnCoreJ 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 EnCoreJ 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 EnCoreJ 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 - SoilCoreJ Sampler

Step 1 - Collect soil sample by pressing the SoilCoreJ 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 SoilCoreJ sample container with a handiwipe or lint-free paper towel to ensure an adequate seal.

Step 3 - Cap the filled end of the SoilCoreJ sample container.

Step 4 - Fill the opposite side of the SoilCoreJ sample container by pushing the empty side of the SoilCoreJ 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 SoilCoreJ 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 SoilCoreJ 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 each 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.**

NOTE 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)

Appendix B

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 EnCoreJ, SoilCoreJ, 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;
- EnCoreJ Sampler T-handle;
- EnCoreJ, SoilCoreJ, 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

<u>VPH</u>

<u>Option 1 - Field Preservation</u>

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 - EnCoreJ Sampler

Step 1 - Place EnCoreJ sample container into the EnCoreJ T-handle.

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

Step 3 - Wipe the outside of the EnCoreJ 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 EnCoreJ 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 - SoilCoreJ Sampler

Step 1 - Collect soil sample by pressing the SoilCoreJ 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 SoilCoreJ sample container with a handiwipe or lint-free paper towel to ensure an adequate seal.

Step 3 - Cap the filled end of the SoilCoreJ sample container.

Step 4 - Fill the opposite side of the SoilCoreJ sample container by pushing the empty side of the SoilCoreJ 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 SoilCoreJ 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 SoilCoreJ 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

Appendix C

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

Appendix D

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* (USEPA SOP No. GW0001; July 30, 1996) (Attachment D-1). In addition, should GE desire to change to a markedly different sampling methodology (e.g., discrete interval samplers, passive diffusion bags, or a yet to be developed technique), 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 using a 0.45-micron disposable filter. No wells will be sampled until well development has been performed in accordance with the procedures presented in Appendix S, 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., ISCO Model 150); and/or
  - Bladder pump (e.g., Marschalk System 1);
- Teflon® tubing or Teflon®-lined polyethylene tubing of an appropriate size for the pump being utilized;
- Water level probe (e.g., Solinst Model 101);
- Water quality (temperature/pH/specific conductivity/ORP/turbidity/dissolved oxygen) meter and flow-through measurement cell. Several brands may be utilized, including:
  - YSI 6-Series Multi-Parameter Instrument;
  - Hydrolab Series 3 or Series 4a Multiprobe and Display; and/or
  - Horiba U-22 Water Quality Monitoring System;

- Supplemental turbidity meter (e.g., Horiba U-22 or Hach 2100P). Turbidity measurements collected with multi-parameter meters have been shown to sometimes be unreliable due to fouling of the optic lens of the turbidity meter within the flow-through cell. A supplemental turbidity meter will be utilized to verify turbidity data during purging if such fouling is suspected. Note that industry improvements may eliminate the need for these supplemental measurements in the future;

- Appropriate water sample containers (supplied by the laboratory);

- Appropriate blanks (trip blank supplied by the laboratory);

- 0.45-micron disposable filters;

- Large glass mixing container; and

- Teflon® stirring rod.

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.

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 prior to placement of pumps. If a submersible or bladder pump is being utilized, slowly lower pump, safety cable, tubing, and electrical lines into the well to a depth corresponding to the 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.

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. 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, the well should be pumped dry and sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection of samples. 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 lower stabilization level cannot be maintained, 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 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 to reduce turbidity levels as low as possible. During extreme weather conditions, stabilization of field indicator parameters may be difficult to obtain. Modifications to the sampling procedures to alleviate these conditions (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 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 parameters have stabilized, 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, a filtered and unfiltered sample will be collected. If the sample cannot be transferred to the laboratory for filtering, sample filtration for the filtered sample will be performed in the field utilizing an in-line filtration system. Install an in-line, disposable 0.45-micron particle filter on the discharge tubing after the appropriate unfiltered groundwater sample has been collected. Continue to run the pump until an initial volume of “flush” water has been run through the filter in accordance with the manufacturer’s directions (generally 100 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), 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).

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 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 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.

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

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 the field indicator parameters have not stabilized, remove a maximum of five well volumes prior to sample collection. Alternatively, five well volumes may be removed with measurement of initial and final groundwater parameters, at a minimum.

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, a filtered and unfiltered sample will be collected. If the sample cannot be transferred to the laboratory for filtering, sample filtration for the filtered sample will be performed in the field utilizing 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 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, 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; and

- 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
USEPA Low-Stress (Low-Flow) Groundwater Purging and Sampling SOP

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I

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

July 30, 1996
Revision 2

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I

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

I. SCOPE & APPLICATION

This standard operating procedure (SOP) provides a general framework for collecting ground water samples that are indicative of mobile organic and inorganic loads at ambient flow conditions (both the dissolved fraction and the fraction associated with mobile particulates). The SOP emphasizes the need to minimize stress by low water-level drawdowns, and low pumping rates (usually less than 1 liter/min) in order to collect samples with minimal alterations to water chemistry. This SOP is aimed primarily at sampling monitoring wells that can accept a submersible pump and have a screen, or open interval length of 10 feet or less (this is the most common situation). However, this procedure is flexible and can be used in a variety of well construction and ground-water yield situations. Samples thus obtained are suitable for analyses of ground water contaminants (volatile and semi-volatile organic analytes, pesticides, PCBs, metals and other inorganics), or other naturally occurring analytes.

This procedure does not address the collection of samples from wells containing light or dense non-aqueous phase liquids (LNAPLs and DNAPLs). For this the reader may wish to check: Cohen, R.M. and J.W. Mercer, 1993, DNAPL Site Evaluation; C.K. Smoley (CRC Press), Boca Raton, Florida and U.S. Environmental Protection Agency, 1992, RCRA Ground-Water Monitoring: Draft Technical Guidance; Washington, DC (EPA/530-R-93-001).

The screen, or open interval of the monitoring well should be optimally located (both laterally and vertically) to intercept existing contaminant plume(s) or along flowpaths of potential contaminant releases. It is presumed that the analytes of interest move (or potentially move) primarily through the more permeable zones within the screen, or open interval.

Use of trademark names does not imply endorsement by U.S.EPA but is intended only to assist in identification of a specific type of device.

Proper well construction and development cannot be overemphasized, since the use of installation techniques that are appropriate to the hydrogeologic setting often prevents "problem well" situations from occurring. It is also recommended that as part of development or redevelopment the well should be tested to determine the appropriate pumping rate to obtain stabilization of field indicator parameters with minimal drawdown in shortest amount of time. With this information field crews can then conduct purging and sampling in a more expeditious manner.

The mid-point of the saturated screen length (which should not exceed 10 feet) is used by convention as the location of the pump intake. However, significant chemical or permeability contrast(s) within the screen may require additional field work to determine the optimum vertical location(s) for the intake, and appropriate pumping rate(s) for purging and sampling more localized target zone(s). Primary flow zones (high(er) permealability and/or high(er) chemical concentrations) should be identified in wells with screen lengths longer than 10 feet, or in wells with open boreholes in bedrock. Targeting these zones for water sampling will help insure that the low stress procedure will not underestimate contaminant concentrations. The Sampling and Analysis Plan must provide clear instructions on how the pump intake depth(s) will be selected, and reason(s) for the depth(s) selected.

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 remaining criteria in this procedure are met. If after 4 hours of purging indicator field parameters have not stabilized, one of 3 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 not meet the sampling objectives).

Changes to this SOP should be proposed and discussed when the site Sampling and Analysis Plan is submitted for approval. Subsequent requests for modifications of an approved plan must include adequate technical justification for proposed changes. All changes and modifications must be approved before implementation in field.

II.EQUIPMENT

A. Extraction device

Adjustable rate, submersible pumps are preferred (for example, centrifugal or bladder pump constructed of stainless steel or

Teflon).

Adjustable rate, peristaltic pumps (suction) may be used with caution. Note that EPA guidance states: "Suction pumps are not recommended because they may cause degassing, pH modification, and loss of volatile compounds" (EPA/540/P-87/001, 1987, page 8.5-11).

The use of inertial pumps is discouraged. These devices frequently cause greater disturbance during purging and sampling and are less easily controlled than the pumps listed above. This can lead to sampling results that are adversely affected by purging and sampling operations, and a higher degree of data variability.

B. Tubing

Teflon or Teflon lined polyethylene tubing are preferred when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics.

PVC, polypropylene or polyethylene tubing may be used when collecting samples for inorganics analyses. However, these materials should be used with caution when sampling for organics. If these materials are used, the equipment blank (which includes the tubing) data must show that these materials do not add contaminants to the sample.

Stainless steel tubing may be used when sampling for VOCs, SVOCs, pesticides, and PCBs. However, it should be used with caution when sampling for metals.

The use of 1/4 inch or 3/8 inch (inner diameter) tubing is preferred. This will help ensure the tubing remains liquid filled when operating at very low pumping rates.

Pharmaceutical grade (Pharmed) tubing should be used for the section around the rotor head of a peristaltic pump, to minimize gaseous diffusion.

C. Water level measuring device(s), capable of measuring to 0.01 foot accuracy (electronic “tape”, pressure transducer). 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 record.

D. Flow measurement supplies (e.g., graduated cylinder and stop watch).

E. Interface probe, if needed.

F. Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate the samples.

G. Indicator field parameter monitoring instruments - pH, Eh, dissolved oxygen (DO), turbidity, specific conductance, and temperature. Use of a flow-through-cell is required when measuring all listed parameters, except turbidity. Standards to perform field calibration of instruments. Analytical methods are listed in 40 CFR 136, 40 CFR 141, and SW-846. For Eh measurements, follow manufacturer's instructions.

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

I. Logbook(s), and other forms (for example, well purging forms).

J. Sample Bottles.

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

L. Sample tags or labels.

M. Well construction data, location map, field data from last sampling event.

N. Well keys.

O. Site specific Sample and Analysis Plan/Quality Assurance Project Plan.

P. PID or FID instrument (if appropriate) to detect VOCs for health and safety purposes, and provide qualitative field evaluations.

III.PRELIMINARY SITE ACTIVITIES

Check well for security damage or evidence of tampering, record pertinent observations.

Lay out sheet of clean polyethylene for monitoring and sampling equipment.

Remove well cap and immediately measure VOCs at the rim of the well with a PID or FID instrument and record the reading in the field logbook.

If the well casing does not have a 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.

A synoptic water level measurement round should be performed (in the shortest possible time) before any purging and sampling activities begin. It is recommended that water level depth (to 0.01 ft.) and

total well depth (to 0.1 ft.) be measured the day before, in order to allow for re-settlement of any particulates in the water column. If measurement of total well depth is not made the day before, it should not be measured until 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 are usually not needed unless analytical data or field head space information signal a worsening situation. Note: procedures for collection of LNAPL and DNAPL samples are not addressed in this SOP.

IV.PURGING AND SAMPLING PROCEDURE

Sampling wells in order of increasing chemical concentrations (known or anticipated) is preferred.

1. Install Pump

Lower pump, safety cable, tubing and electrical lines slowly (to minimize disturbance) into the well to the midpoint of the zone to be sampled. The Sampling and Analysis Plan should specify the sampling depth, or provide criteria for selection of intake depth for each well (see Section I). 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. Collection of turbid free water samples may be especially difficult if there is two feet or less of standing water in the well.

2. Measure Water Level

Before starting pump, measure water level. If recording pressure transducer is used-initialize starting condition.

3. Purge Well

3a. Initial Low Stress Sampling Event

Start the pump at its lowest speed setting and slowly increase the speed until discharge occurs. Check water level. Adjust pump speed until there is little or no water level drawdown (less than 0.3 feet). If the minimal drawdown that can be achieved exceeds 0.3 feet but remains stable, continue purging until indicator field parameters stabilize.

Monitor and record water level and pumping rate every three to 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 (for example, 0.1 - 0.4 l/min) to ensure stabilization of indicator

parameters. 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" as pump flow adjustments are made. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. Do not allow the water level to fall to the intake level (if the static water level is above the well screen, avoid lowering the water level into the screen). The final purge volume must be greater than the stabilized drawdown volume plus the extraction tubing volume.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (bladder, peristaltic), and/or the use of dedicated equipment. If the recharge rate of the well is lower than extraction rate capabilities of currently manufactured pumps and the well is essentially dewatered during purging, then the well should be sampled as soon as the water level has recovered sufficiently to collect the appropriate volume needed for all anticipated samples (ideally the intake should not be moved during this recovery period). Samples may then be collected even though the indicator field parameters have not stabilized.

3b. Subsequent Low Stress Sampling Events

After synoptic water level measurement round, 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). Perform purging operations as above.

4. Monitor Indicator Field Parameters

During well purging, monitor indicator field parameters (turbidity, temperature, specific conductance, pH, Eh, DO) every three to five minutes (or less frequently, if appropriate). Note: during the early phase of purging emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments. 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, taken at three (3) to five (5) minute intervals, are within the following limits:

turbidity (10% for values greater than 1 NTU),
DO (10%),
specific conductance (3%),
temperature (3%),
pH (± 0.1 unit),
ORP/Eh (± 10 millivolts).

All measurements, except turbidity, must be obtained using a 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 and may also cause an underestimation of turbidity values measured after 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.

The flow-through-cell must be designed in a way that prevents air bubble entrapment in the cell. 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 be submerged in water at all times. If two flow-through-cells are used in series, the one containing the dissolved oxygen probe should come first (this parameter is most susceptible to error if air leaks into the system).

5. Collect Water Samples

Water samples for laboratory analyses must be collected before water has passed through the flow-through-cell (use a by-pass assembly or disconnect cell to obtain sample).

VOC samples should be collected first and directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

During purging and sampling, the tubing should remain filled with water so as to minimize possible changes in water chemistry upon contact with the atmosphere. 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 one of the following procedures to collect samples: (1) add clamp, connector (Teflon or stainless steel) or valve to constrict sampling end of tubing; (2) insert small diameter Teflon tubing into water filled portion of pump tubing allowing the end to protrude beyond the end of the pump tubing, collect sample from small diameter tubing; (3) collect non-VOC samples first, then increase flow rate slightly until the water completely fills the tubing, collect sample and record new drawdown, flow rate and new indicator field parameter values.

Add preservative, as required by analytical methods, to samples immediately after they are collected if the sample containers are not pre-preserved. Check analytical methods (e.g. EPA SW-846, water supply, etc.) for additional information on preservation. Check pH for all samples requiring pH adjustment to assure proper pH value. For VOC samples, this will require that a test sample be collected during purging to determine the amount of preservative that needs to be added to the sample containers prior to sampling.

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 is required, and the filter

size (0.45 um is commonly used) should be based on the sampling objective. Pre-rinse the filter with approximately 25 - 50 ml of ground water prior to sample collection. Preserve 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 ground water for human health risk calculations.

Label each sample as collected. Samples requiring cooling (volatile organics, cyanide, etc.) 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.

6. Post Sampling Activities

If recording pressure transducer is used, remeasure water level with tape.

After collection of the samples, the pump tubing may either be dedicated to the well for resampling (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 is optional after the initial low stress sampling event. However, it is recommended if the well has a "silting" problem or if confirmation of well identity is needed.

Secure the well.

V. DECONTAMINATION

Decontaminate sampling equipment prior to use in the first well and following sampling of each subsequent well. Pumps will not be removed between purging and sampling operations. The pump and tubing (including support cable and electrical wires which are in contact with the well) will be decontaminated by one of the procedures listed below.

Procedure 1

The decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump or the pump can be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and isopropyl 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.

Flush with isopropyl alcohol (pesticide grade). If equipment blank data from the previous sampling event show that the level of contaminants is insignificant, then this step may be skipped.

Flush with distilled/deionized water. 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.

VI. FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the ground water samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples shall be collected for each batch of samples (a batch may not exceed 20 samples). Trip blanks are required for the VOC samples at a frequency of one set per VOC sample cooler.

Field duplicate.

Matrix spike.

Matrix spike duplicate.

Equipment blank.

Trip blank (VOCs).

Temperature blank (one per sample cooler).

Equipment blank shall include the pump and the pump's tubing. If tubing is dedicated to the well, the equipment blank will only include the pump in subsequent sampling rounds.

Collect samples in order from wells with lowest contaminant concentration to highest concentration. Collect equipment blanks after sampling from contaminated wells and not after background wells.

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

If split samples are to be collected, collect split for each analyte group in consecutive order (VOC original, VOC split, etc.). Split sample should be as identical as possible to original sample.

All monitoring instrumentation shall be operated in accordance with EPA analytical methods and manufacturer's operating instructions. EPA analytical methods are listed in 40 CFR 136, 40 CFR 141, and SW-846 with exception of Eh, for which the manufacturer's instructions are to be followed. Instruments shall be calibrated at the beginning of each day. If a measurement falls outside the calibration range, the instrument should be re-calibrated so that all measurements fall within the calibration range. At the end of each day, check calibration to verify that instruments remained in calibration. Temperature measuring equipment, thermometers and thermistors, need not be calibrated to the above frequency. They should be checked for accuracy prior to field use according to EPA Methods and the manufacturer's instructions.

VII. FIELD LOGBOOK

A field log shall be kept to document all ground water field monitoring activities (see attached example matrix), and record all of the following:

Well identification.

Well depth, and measurement technique.

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, and clock time, at the appropriate time intervals; calculated or measured total volume pumped.

Well sampling sequence and time of each sample collection.

Types of sample bottles used and sample identification numbers.

Preservatives used.

Parameters requested for analysis.

Field observations during sampling event.

Name of sample collector(s).

Weather conditions.

QA/QC data for field instruments.

Any problems encountered should be highlighted.

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

VIII. DATA REPORT

Data reports are to include laboratory analytical results, QA/QC information, and whatever field logbook information is needed to allow for a full evaluation of data useability.

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 (below MP) ________/________ of screen
top bottom
Pump Intake at (ft. below MP)________________
Purging Device; (pump type)__________________

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/ Eh[3] mv	DO mg/L	Turb- idity NTU	Comments

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 (stand in for Eh).

Attachment D-2
Groundwater Sampling Log

GROUNDWATER SAMPLING LOG

Well No. ______________________ **Site/GMA Name** ______________________
Key No. ______________________ **Sampling Personnel** ______________________
PID Background (ppm) ______________________ **Date** ______________________
Well Headspace (ppm) ______________________ **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

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]*

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]*

Attachment D-3
Representative Sample Label

Project #	Date
Sample I.D.	

Sample Type	Collection Mode	Time
☐ Soil/Sediment ☐ Water	☐ Composite ☐ Grab	

Analysis

Sampler(s)	Preservative

Appendix E

Surface Water Sampling Procedures

Appendix E

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; the procedures for obtaining velocity profile measurements at selected river/stream cross-sections; and the 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.

Materials

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 mixing container;
- Peristaltic pump;
- Medical-grade silicone tubing;
- Teflon7 tubing;
- Teflon7 stirring rod;
- 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 Ablue@ice; and
- Appropriate water sampler as specified in the project-specific work plan, which may include following:

- Surface water grab sampler (Attachment E-3) consisting of a 1,000 mL beaker, adjustable clamp, and two-or three-piece telescoping aluminum tube or an equivalent acceptable sampling device; or
- Peristaltic pump with a short piece of medical-grade silicone tubing and with Teflon7 tubing; or
- Kemmerer stainless steel bottle sampler (Attachment E-4).

Procedures

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-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 - Assemble the water grab sampler (Attachment E-3). 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 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 Teflon7 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 Teflon7 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 Teflon7 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 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.

Step 18 - Measure the water temperature about 1-foot below the surface 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, a peristaltic pump (Attachment E-4) will be used, and the following procedures will be followed:

Step 1 - Identify sampling location on appropriate sampling log sheet (Attachment E-2) and/or field notebook along with other appropriate information;

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

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

Step 4 - Install clean, medical-grade silicone tubing in the pump head, as per the manufacturer=s instructions. Allow sufficient tubing on the discharge side to facilitate convenient dispensation of liquid into sample bottles and only enough on the suction end for attachment to the intake line. This practice will minimize sample contact with the silicone pump tubing;

Step 5 - Select the length of Teflon7 tubing necessary to reach the required sample depth and attach to intake side of pump tubing. Taping the Teflon7 tube to a surveyor=s rod will facilitate reaching the required depth;

Step 6 - If possible, allow several liters of sample to pass through the system before actual sample collection. Collect this purge volume and then return to source after the sample aliquot has been withdrawn;

Step 7 - Using a graduated cylinder or beaker as a measuring device, collect equal volumes of water at depths equaling 0.2, 0.5, and 0.8 times the total water depth,

Step 8 - If sampling for VOCs, a Kemmerer stainless steel bottle sampler (as shown in Attachment E-4) will be used in place of the peristaltic pump. To collect the sample, lower the Kemmerer bottle to mid-depth and release the trigger. Raise the Kemmerer bottle from the water column with minimal disturbance.

Step 9 - If sampling for total and filtered metals, a filtered and unfiltered sample will be collected. Sample filtration for the filtered sample will be performed in the field utilizing a peristaltic pump prior to preservation. Install new medical-grade silicone tubing in the pump head. Place new Teflon**7** tubing into the sampling 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 - The samples taken from the three depths noted above will be combined into one composite sample for the given location;

Step 12 - After sample collection, follow steps 7 through 19 described in the preceding Section II.A; and

Step 13 - All tubing will be discarded between sampling locations and not re-used.

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 on-going disposal practices.

Attachment E-1
Surface Water Grab Sample Field Log

Surface Water Grab Sample Field Log

Client	____________	Project No.	____________
Site	____________	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 ____________ Suspended Soils ____________

Odor ____________ Film ____________

IV. Container Analysis

V. Surface Water Characteristics

Color	________	Suspended Solids	____________
Temperature	________	Film	____________
		Odor	____________

Attachment E-2
Peristaltic Pump Sampler Field Log

Peristaltic Pump Sampler Field Log

Location ___________ Date ____________, 19__ Sampled by: _____________

Location No. _______ Gauge Reading ___________________________

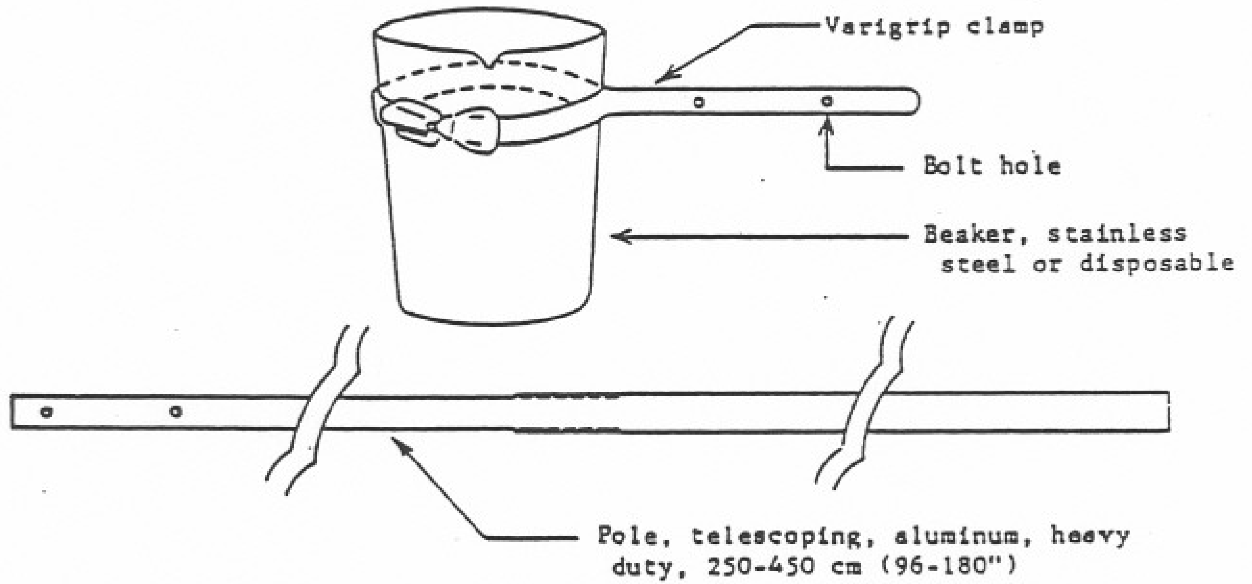
Measured Water Depth (D) ________ ft Air _______°F Water _________°F

Weather __________________________ Flow Conditions _______________

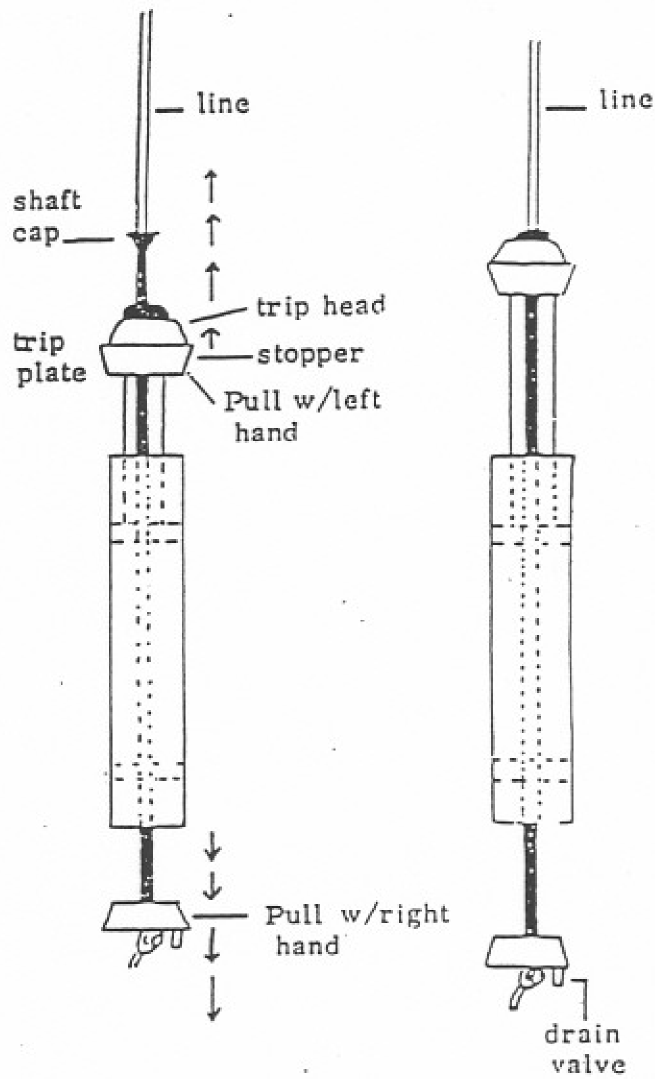
Sample ID	Date	Time	Sample Depth (feet off bottom)	Analysis

Comments ____

Attachment E-3
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-4
Kemmerer Bottle


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

Appendix F

Sediment Sampling Procedures

Appendix F

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 Procedures

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.

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

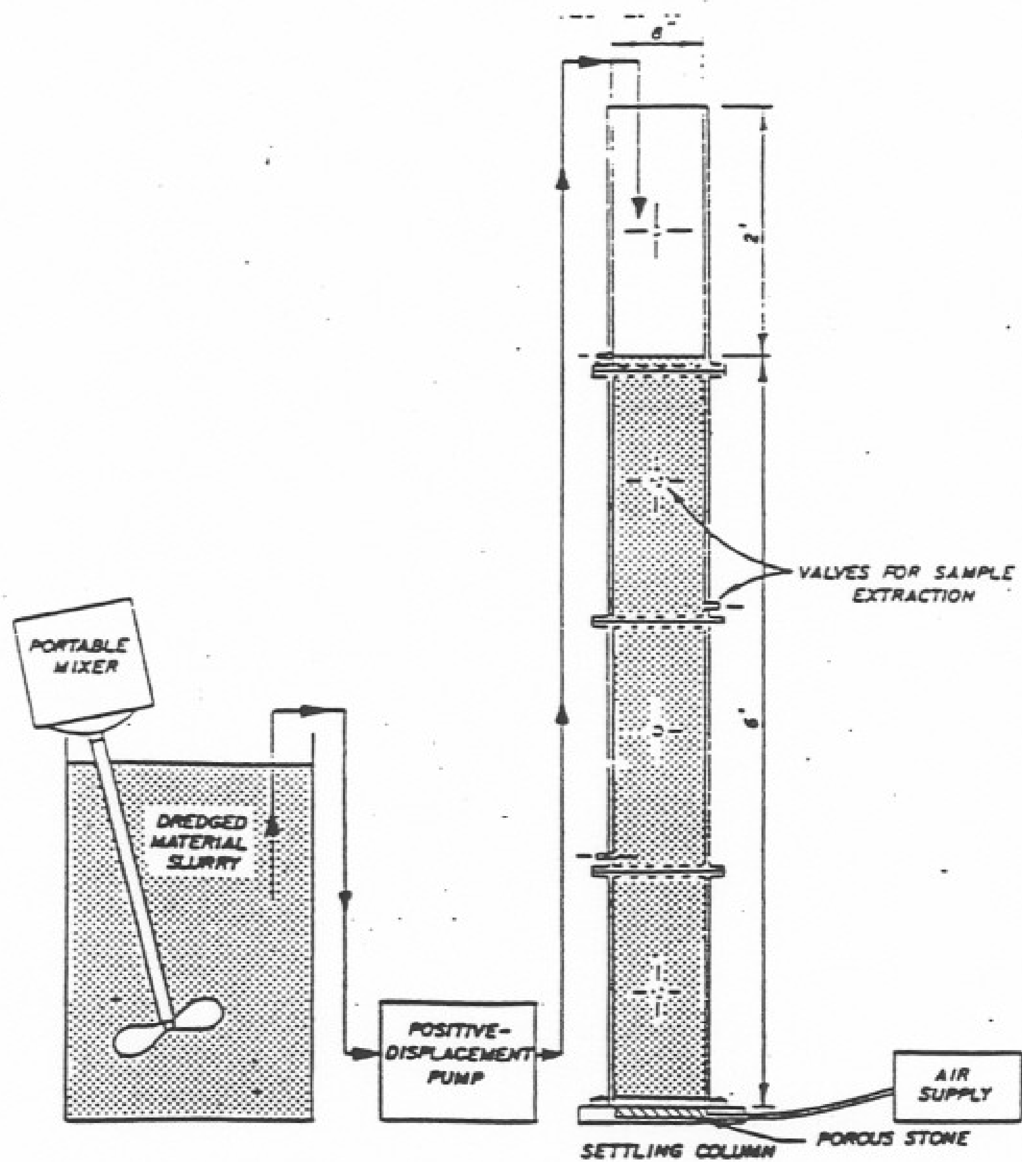
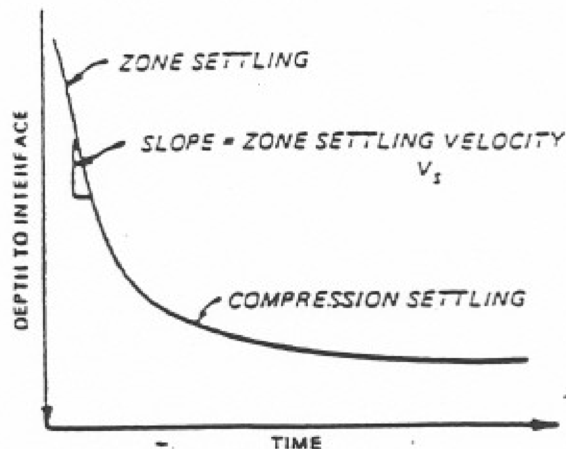


Figure 1. Schematic diagram of apparatus for settling tests

Figure A1. Conceptual plot of interface height versus time



Test Procedures

Pilot test

4. A pilot test conducted in a small graduated cylinder (1ℓ 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 6-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

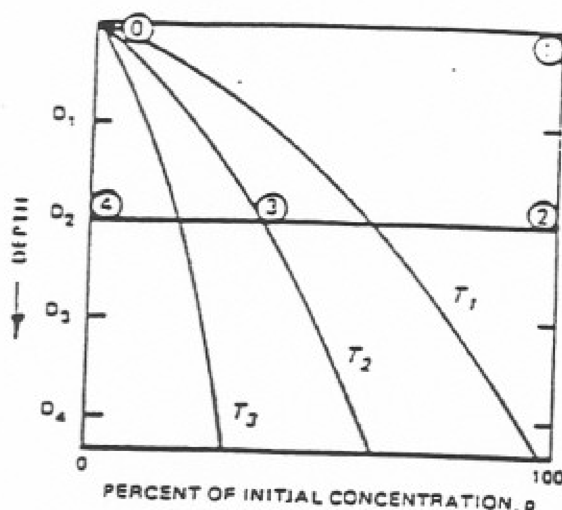


Figure A2. Conceptual concentration profile diagram

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.

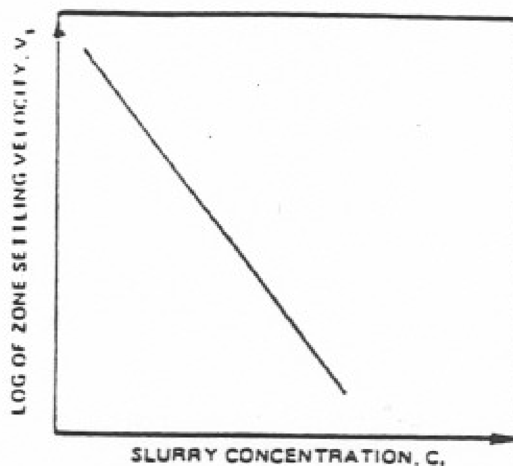


Figure A3. Conceptual plot of zone settling velocity versus concentration

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.

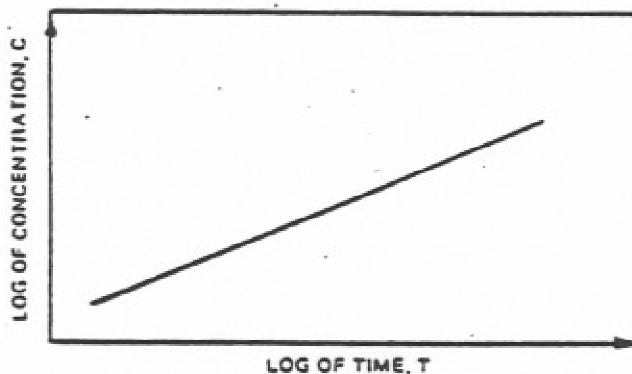


Figure A4. Conceptual time-versus-concentration plot

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

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

Appendix G

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

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 Procedures

Appendix H

Biota Sampling Procedures

I. Introduction

This Appendix specifies the procedures for certain types of biota sampling that may be conducted in Massachusetts pursuant to the Consent Decree for the GE-Pittsfield/Housatonic River Site, the Reissued RCRA corrective action permit for the Rest of the River portion of that Site, or the Administrative Consent Order (ACO) executed by GE and the Massachusetts Department of Environmental Protection (MDEP) for certain off-site properties. These include: (a) procedures for the collection and processing of samples of aquatic vertebrates (fish and bullfrogs) for chemical analysis of their tissue; (b) procedures for aging fish (if appropriate); (c) procedures for the installation, sampling, and processing of caged bivalves (mussels) to monitor bioaccumulation; and (d) procedures for conducting a survey of benthic macroinvertebrates in order to assess bioturbation potential. For other types of biota sampling (if appropriate), the sampling and analysis procedures will be presented in the work plan proposing such sampling (if such sampling is proposed to U.S. Environmental Protection Agency [EPA] or MDEP for approval) or in other documents relating to such investigations.

The procedures for collection and analysis of biota samples from the Connecticut portion of the Housatonic River will be those presented in Attachment H-2 to this Appendix, prepared by the Academy of Natural Sciences of Philadelphia (ANSP), or as specified in the applicable work plan or report prepared by the ANSP.

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

This part 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 fish and frog samples.

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. The directions for wrapping, labeling, and shipping the aquatic vertebrate samples are listed below:

   a. All samples must remain whole and ungutted.

   b. 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.

   c. 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 so as to limit the ability of the sampling study to determine differences between 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 other wise provided in a project-specific work plan.

   d. Wrap each sample (or composite sample) 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. Processing

Once the fish and/or frog samples have been transported to the analytical lab, the following processing procedures 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

   a. All samples will be prepared as one of the following samples types:

      – Scale and Fillet (SF): standard skin-on fillet with scales removed - used for adult bass, perch, trout, and other fish that could be consumed by humans. Each fish sample shall be placed on fresh aluminum foil following a thorough rinse with deionized water. A sterile disposable scalpel blade shall be used to remove the fillet (skin-on) from the carcass. The fillet from one site of each sample will be used for PCB and lipid analyses. The remaining fillet will be archived in a frozen state for future analysis as required in the project-specific work plan.

      – Composite: Used for young-of-the-year (YOY) fish. YOY fish will not be filleted. A composite sample of the whole body fish will be prepared using the grinding procedures identified below.

      – Legs Only (LO): (used for bullfrogs). Hind legs will be skinned and boned using sterile, disposable scalpel blades. Muscle meat from both legs will be used for PCB and lipid analyses. Sample aliquots shall be combined as described in the project-specific work plan.

   b. A new sheet of aluminum foil and a new disposable scalpel blade will be used for each sample. The scalpel handle 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.

III. 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.

IV. Caged Bivalve Bioaccumulation Study Procedures

Studies may be conducted to monitor the bioaccumulation of polychlorinated biphenyls (PCBs) in bivalves (mussels) installed in cages in the Housatonic River. Unless otherwise provided in the project-specific work plan, the procedures to be used to install such cages and to collect, process, ship, and prepare the mussel samples for chemical analysis will be the procedures specified in Attachment H-3 to this Appendix.

V. Sample Preparation and Analysis

Samples of the biota collected in Massachusetts may be analyzed for Aroclor-specific PCBs and/or polychlorinated dibenzo-p-dioxins (PCDDs)/polychlorinated dibenzofurans (PCDFs). Such analyses will be conducted following the methods presented in Table 1 of the FSP/QAPP. The procedures for preparation of fish samples for analysis will be those presented in the Standard Operating Procedure (SOP) on “Preparation of Biological Tissues for Analytical Determinations,” included in Attachment H-1 hereto, unless otherwise provided in the project-specific work plan. Bullfrog and caged mussel samples will be processed using similar procedures to those listed in Attachment H-1, with the exception that bullfrog samples will be prepared as the edible portion of the legs (boneless, skin-off) and caged mussel samples will be prepared as whole-body composite samples minus the shell (see Attachment H-3). The specific procedures for analysis of all such biota samples collected in Massachusetts for Aroclor-specific PCBs and lipid content will be those presented in the SOPs in Attachment H-1 on extraction of biological samples for PCBs, determination of lipid content, and analysis of PCBs, unless otherwise provided in the applicable project-specific work plan. If additional analyses are proposed, the protocols for such analyses will be presented in the applicable project-specific work plan.

As noted above, the procedures for preparation and analysis of biota samples collected from the Connecticut portion of the Housatonic River for PCBs and lipid content will be those presented in Attachment H-2, or as specified in the applicable work plan or report prepared by the ANSP.

VI. Benthic Invertebrate Survey Procedures

Studies may be conducted to evaluate the potential impact of bioturbation on a sediment cap surface. Bioturbation refers to processing, mixing, and/or resuspension of sediments by aquatic organisms (including benthic macroinvertebrates and other bottom-dwelling organisms) while burrowing, feeding, spawning, and/or undertaking other physiological activities. Unless otherwise provided in the project-specific work plan, the procedures to be used to collect, process, ship, and prepare benthic invertebrate samples will be the procedures specified in Attachment H-4 to this Appendix.

Attachment H-1

Preparation of Biological Tissue
Extraction of Biota Samples for PCBs
Determination of Lipid Content
Analysis of Aroclor-Specific PCBs

PROCEDURE TITLE: Preparation of Biological Tissues for Analytical Determinations

DEPARTMENT: General Laboratory

PROCEDURE SUMMARY:

This document outlines the preparation of biological tissue samples prior to laboratory analysis. These preparation steps are compatible with the analysis of volatile organics, semivolatile organics, pesticides/PCB's, herbicides and metals.

REVIEWED BY: Kevin Noltemeyer — 6-22-99
Kevin Noltemeyer
Extractions Groupleader
Date

REVIEWED BY: Gregory J. Graf — 6-22-99
Gregory J. Graf
Quality Assurance Officer
Date

APPROVED BY: Glen A. Coder — 6-24-99
Glen Coder
Laboratory Manager
Date

EQUIPMENT AND MATERIALS:

- Stainless steel spatula
- HDPE or stainless steel cutting board
- Knife, heavy blade (or meat cleaver)
- Mallet, plastic faces, 2 to 3-lbs
- Hobart Stainless Steel meat grinder
- Blender, stainless steel blade (glass or stainless steel blender container only, no plastic containers)
- 40 mL glass vials with Teflon septum caps
- Wide-mouth 4 to 16-oz glass jars with Teflon lined caps
- Dry ice or liquid nitrogen
- Methanol
- Canned tuna fish (if volatile analytes are of interest) preferably Chicken of the Sea in spring water
- Tekmar Tissumizer

PROCEDURE:

Note: Preparation steps must be done in a clean environment (i.e. not in an extraction lab) when the determination of volatile organics is to be determined. All efforts should be made to minimize sources of contamination.

1. All equipment to be used in the preparation of tissue samples should be soap and water washed and then rinsed with hot water and then a final rinse with methanol. This procedure should be repeated between samples before re-using the equipment.

   Some samples such as eggs, insects, or small livers etc.. may be prepped with the tissumizer in the jar they were sent in so as to avoid loss of sample. If this is done "sample prepped in jar" should be noted on the prep worksheet or in the lab notebook.

2. Fish samples may be prepared with the following specifications:

   - Whole Fish
   - Fillet of Fish
   - Skin On
   - Skin Off

   The type of preparation usually takes into account the size of the fish, the species being investigated and the class of the compound or compounds to be determined. Be sure to clarify the specific requirements with the project sponsor before proceeding in the preparation of the fish.

3. Unwrap and, if required, weigh each fish before any grinding or blending steps. Record this weight on a LIMS worksheet or in a bound lab notebook. Small fish, such as minnows, are usually collected as composites and will represent a single composite sample and therefore should be weighed as a single sample. A total weight of 250 grams is the preferred mass required when a complete screening of the fish is to be performed (volatile organics, semivolatile organics, pesticides, metals).

4. Chop larger fish into 2 to 3-inch cubes, using either a sharp knife and mallet or a butcher saw.

5. Grind the fish cubes in a large commercial meat grinder or industrial blender (Note: When dealing with small quantities of tissue such as minnows, use a blender instead of the meat grinder to minimize the loss of tissue in the grinding process itself).

6. Thoroughly mix and re-grind two additional times. Clean out any material remaining in the grinder; add this to the sample and mix well.

7. For Volatile Analysis: Weigh three 5.0 g portions of the sample into separate 40-mL glass vials. Cap the vial with a Teflon septum/screw cap and store in a freezer until ready for analysis. Document on the prep worksheet or lab notebook that these were prepared.

8. If samples are to be analyzed for volatile compounds, a volatile preparation blank must be prepared each day samples are prepared. The preparation blank consists of tuna fish put through the same preparation steps as the samples. Three 5 gram aliquots of this prepared tuna fish should be placed into separate 40-mL glass vials and labelled as preparation blanks with the date, and initials of the person doing the preparation. Document on the prep worksheet or lab notebook that these were prepared and write the manufacturer's number from the can of tuna used. In addition, place three 5.0 g aliquots of the unblended tuna into separate 40-mL glass vials to provide background blank samples.

9. If metals are to be analyzed then transfer approximatly 50 grams of the coarse-ground sample to a stainless steel bowl containing liquid nitrogen. Place the frozen sample in a blender cup and blend the frozen tissue to a powder consistency. Transfer the blended sample to a clean 8-oz jar, marked for metals analysis, and store in a freezer until ready for analysis.

NOTE: The entire sample may be processed using the blender technique if so desired. This technique should be used when the desired homogeneity is not achieved for organics using the Hobart grinder. The blending technique is mandatory when metals are to be analyzed due to the small aliquot required for analysis.

10. Transfer any remaining fish sample (left from the preliminary Hobart grind) to a glass container and store in a freezer for later subsampling and analysis for pesticides and other semivolatile organics.

11. Initial and date the prep worksheet or lab notebook to indicate that the sample has gone through the homogenization steps.

En Chem, Inc.

Quality Assurance Document

SET No: 1

Standard Operating Procedure

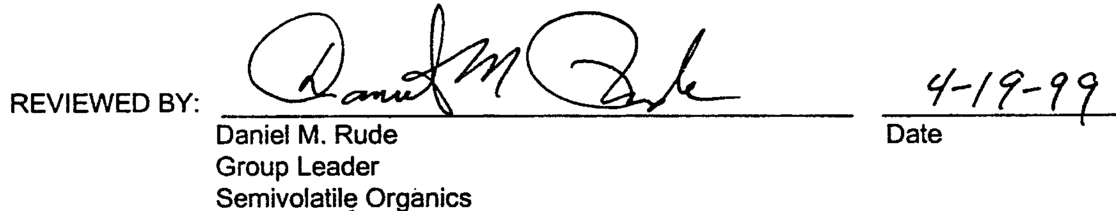
TITLE: Extraction of Biological Samples for Organochlorine Pesticides/PCBs

DEPARTMENT: Semivolatile Organic Extractions

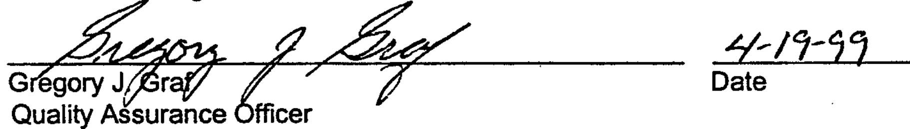
REFERENCES: Test Methods for Evaluating Solid Wastes
SW846 Method 3540C (Dec. 1996)

PROCEDURE SUMMARY:

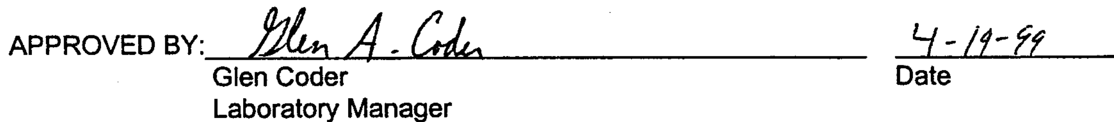
Approximately 20 grams of homogenized tissue sample is mixed with sodium sulfate and extracted with methylene chloride for a minimum of 16 hours in a soxhlet extractor. The methylene chloride extracts are exchanged to hexane and concentrated to a volume of 10 mL.



REVIEWED BY: Daniel M. Rude, Group Leader, Semivolatile Organics — Date: 4-19-99



Gregory J. Graf, Quality Assurance Officer — Date: 4-19-99



APPROVED BY: Glen Coder, Laboratory Manager — Date: 4-19-99

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QUALITY CONTROL:

Note: If sample extracts are to be analyzed by **both** Method 8081A and 8082, use PCB and pesticide spike solutions for the LCS and MS/MSD or extract separate spikes for the two methods.

- The samples may be kept frozen up to a year before being extracted.
- One method blank is extracted per 20 samples OR per extraction batch, whichever is more frequent. The method blank should be blank sodium sulfate or an analyte free biota matrix such as tuna fish for animal extractions or alfalfa for plant analyses.
- A laboratory control spike is extracted per 20 samples OR per extraction batch whichever is more frequent. Control spikes are usually prepared using analyte free tuna fish for animal analyses or alfalfa for plant analyses. These control spikes are fortified with a representative list of the analytes of interest.
- Surrogate standards must be added to all samples, laboratory control spikes, matrix spikes, and method blanks prior to extraction. Surrogates are used to monitor the efficiency of the method on each sample and possible matrix related affects.
- A matrix spike and a matrix spike duplicate must be performed for every 20 samples. Matrix spikes are used to indicate matrix effects on the analysis of the analytes of interest.

INTERFERENCES:

Method interferences may be caused by contaminants (primarily phthalate esters) in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Contact with common plastics or rubber products must be avoided.

MATERIALS AND APPARATUS:

Soxhlet Extractors	40 mm i.d. X 250 mm length holding cell with heaters and cold water condensers.
Concentrator tubes:	Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent).
Evaporation flask:s	Kuderna-Danish 300 mL (Reliance (Reliance G-9601-001 or equivalent).
Snyder columns:	Kuderna-Danish, three-ball macro (Kontes K-503000-0121 or equivalent).
Vials:	Glass, 12 mL capacity with Teflon-lined screw cap.
Funnel:	150-gram capacity.

Boiling chips:	Teflon or pre-rinsed silicon carbide.
Sodium Sulfate:	Preheated at 400^0 C for 4 hours in a crucible to remove contaminants.
Water bath :	Heated, with concentric ring cover, capable of temperature control (±5^0 C). The bath should be used in a hood.
Pipets:	2 mL short-stem.
Syringes:	250-1000 µL Gastight syringes (Hamilton 1000 series or equivalent).
Beakers:	250 mL
Erlenmeyer flasks	500 mL
Filter paper:	Whatman #41(or equivalent).
Balance:	Capable of weighing 300 g $\pm$ 0.01 g.
Spatulas	
Glass Wool	

REAGENTS:

Surrogate Spiking Solution:

The mixture contains the following components at:
20 µg/mL

DCB (decachlorobiphenyl)
TCMX (tetrachloro-m-xylene)

Spiking Solution: See appendix A.

Methylene chloride, acetone, and hexane pesticide grade.

EXTRACTION PROCEDURE OUTLINE:

Sample Extraction

1. Attach Soxhlet extractors (40 mm i.d. X 250 mm length) to 500-mL Erlenmeyer flasks with ground glass joints.

2. Add two plugs of glass wool to each extractor, one to cover the bottom to prevent sample from entering the solvent return arm and the other to cover the top of the sample.

3. Add 300 mL of glass-distilled methylene chloride to the Erlenmeyer flask, along with about five boiling chips. Attach the Erlenmeyer to the Soxhlet extractor.

4. Attach the extractors to the condensers in the fume hood.

5. Adjust the temperature so the extractors cycle at a rate of 12 to 15 cycles/hour.

6. Allow the extractors to rinse for 4 hours, then shut off the heaters and allow them to cool.

7. Remove the condensers and drain all the solvent remaining in the extractors into the Erlenmeyer flask.

8. Discard the solvent.

9. The extractors are now ready for the samples.

10. Weigh 20 g of sodium sulfate or the control matrix into a 250-mL beaker; this will represent the method blank.

11. Weigh 20 g of the control matrix into a second 250-mL beaker; this will be used for the control spike.

12. Weigh 20 g samples into separate 250-mL beakers. Record actual weight in the extraction log book.

13. Add 40 g of anhydrous sodium sulfate to each beaker and mix More sodium sulfate may be necessary (when a sufficient amount has been added, the sample will appear granular).

14. Place the beakers in a fume hood and let them dry, stirring occasionally.

15. Add 50 µL of surrogate spiking solution to each sample beaker and the method blank beaker.

16. Add 125 µL aliquot of the Pesticide Standard Mix A and 125 µL of Pesticide Standard Mix B to the LCS, MS, and MSD. NOTE: The Individual A and Individual B pesticide spiking solutions contain TMX and DCB. Do not fortify the LCS, MS, or MSD with 50 µL of surrogate spiking solution.

17. Remove the top glass wool plug from the Soxhlet extractors that have been pre-rinsed.

18. Transfer the entire sample to the extractor and place the glass wool plug on top.

    Note: The sample level in the extractor should not exceed the top of the solvent return arm; this will keep the entire sample immersed in solvent during the extraction process.

19. Add 150 mL of methylene chloride to the mixing beaker, swirl it and add the solvent to the respective Erlenmeyer flask along with about five boiling chips.

20. Attach the Soxhlet extractor to the Erlenmeyer flask.

21. Add 200 mL of glass-distilled methylene chloride to each Soxhlet extractor.

22. Attach the condensers and set the temperature so that the extractors cycle at a rate of 12 to 15 cycles per hour.

23. Let the extractors cycle for 16 hours.

24. After 16 hours, shut off the heating elements and allow the samples to cool.

25. Drain all solvent remaining in the extractor into the Erlenmeyer flask.

26. Rinse the extractor with about 50 mL of methylene chloride and drain it into the collection Erlenmeyer flask.

27. Assemble a K-D concentrator by attaching a 10-mL concentrator tube to a 500 mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D if equivalency is demonstrated for all pesticides listed in this method.

28. Pour the extracts into the K-D concentrators. Rinse the Erlenmeyer flasks with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

29. Add one or two clean boiling chips to the evaporative flasks and attach a three-ball Snyder column. Place the K-D apparatus on a hot water bath (80°C to 90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatuses and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 5 to 10 mL, remove the K-D apparatus. Allow it to drain and cool for at least 10 minutes.

30. Adjust the volume to 10 mL using methylene chloride.

Lipid Determination: One milliliter of the extract is used to determine the percent lipids using En Chem, Inc. method SVO-59. The remaining nine milliliters should go through the following cleanups necessary for the required analysis.

GPC Cleanup of Extracts: Sample extracts requiring pesticide and PCB analysis should have contaminants removed using gel permeation chromatography following En Chem Inc. Method SVO-26. This cleanup is not necessary for samples requiring only PCB analysis because the Florisil column cleanup procedure is validated specifically to remove contaminants from the PCBs.

Florisil Column Cleanup of Extracts for PCBs: Extracts requiring only PCB analysis should be cleaned using column chromatography with Florisil by En Chem Inc. Method SVO-57.

Silica Gel Separation of Pesticides and PCBs: Extracts which are to be analyzed for both pesticides and PCBs may have the PCBs separated from the majority of the pesticides using column chromatography with silica gel by En Chem Inc. Method SVO-58. Extracts may be screened prior to this cleanup to determine if PCBs will cause a problem with the identification of pesticides in the extract. If PCBs are not present , or present at levels which will not interfere with pesticide analysis, this cleanup is not necessary.

Appendix A

SPIKING SOLUTIONS

Historical data or requirements of specific projects may determine the analytes and concentrations added to the sample spikes. These concentrations are typically used:

Soln. 1. PCB Matrix Spike

10.0 µg/mL

One of either Aroclor 1016, 1242, 1248, 1254, or 1260.
(Aroclors 1016 and 1260 may be spiked together)

Soln. 2. Pesticide Matrix Spike (these may require more than one solution)

0.5 µg/mL

g-BHC
a-BHC
Endosulfan I
Heptachlor
Aldrin
b-BHC
d-BHC
a-Chlordane
g-Chlordane
Heptachlor epoxide

1.0 µg/mL

4,4'-DDD
4,4'-DDT
Dieldrin
Endrin
4,4'-DDE
Endosulfan II
Endosulfan sulfate
Endrin aldehyde
Endrin ketone

5.0 µg/mL

Methoxychlor

Soln. 3. 50.0 µg/mL

Toxaphene (used if toxaphene is a suspected analyte)

Additional compound which may be added as needed based on project needs:

2,4-DDT	2,4-DDD	2,4-DDE
Hexachlorobenzene	Pentachloroanisole	Oxychlordane
Trans-nonachlor	Cis-nonachlor	Mirex

En Chem, Inc.

Quality Assurance Document

SET No: 1

STANDARD OPERATING PROCEDURE

TITLE: The Determination of Lipids in Tissues, Fats and Plants.

DEPARTMENT: Semivolatile Organics

APPLICATION: This method is applicable to the determination of lipid in any matrix.

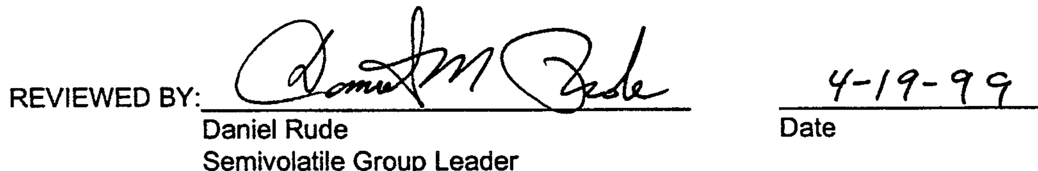
REFERENCES:

1. Randall, R.C., Lee, H., Ozretich, R.J., Lake, J.L., and Pruell, Pruell, R.J. "Evaluation of Selected Lipid Methods for Normalizing Pollutant Bioaccumulation". Environmental Toxicology and Chemistry, Vol 10, p. 1431-1436, (1991)

2. "Standard Methods for the Examination of Water and Wastewater." ALPH, AWWA, WPCF 18th ed., method 5520, (1992)

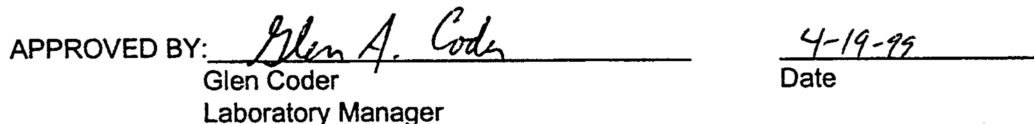
PROCEDURE SUMMARY:

An aliquot of a sample extract is placed into a pre-weighed aluminum weighing pan and the solvent is allowed to evaporate. The lipids are determined gravimetrically.



REVIEWED BY: Daniel Rude, Semivolatile Group Leader — Date: 4-19-99

REVIEWED BY: Gregory J. Graf, Quality Assurance Officer — Date: 4-19-99



APPROVED BY: Glen Coder, Laboratory Manager — Date: 4-19-99

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SAFETY PRECAUTIONS:

Observe all standard laboratory safety procedures as outlined in the Safety Training Manual.

INTERFERENCES:

Do not allow foreign material to fall into the weighing pans while they are drying or the results will be biased low. It is recommended to cover the weighing pans with a loose piece of aluminum foil while they are drying.

QUALITY ASSURANCE:

Calibrate the analytical balance prior to each day's weighing.

APPARATUS:

- Aluminum weighing pans
- One milliliter pipet or syringe
- Balance - Analytical, capable of accurately weighing to the nearest 0.01 g

PROCEDURE:

1. Label an aluminum pan for each sample to be analyzed.

2. Weigh the aluminum pan to the nearest 0.01 grams. Record this "pan weight" in a log book or have it electronically downloaded into a calculation sheet.
3. An extract is typically obtained by a soxhlet extraction of a sample followed by a concentration to a measured volume (refer to En Chem SOP SVO-60). Place one milliliter of sample extract into the pan. If sufficient volume of extract is not available, a lesser amount may be used. Record the volume.

4. Place the sample and pan onto a drying rack at room temperature or into a fume-hood for a minimum of 12 hours. The sample pans may need to be lightly covered with aluminum foil to avoid particulate matter from getting into the pans.

5. After the solvent has evaprated from the pan weigh the sample and pan again to obtain gross dry weight. Record this in the log book or have it recorded electronically.

CALCULATIONS:

1. The pan weight is subtracted from the gross dry weight yielding net dry weight.

2. The following calculation is used to calculate the percent lipid:

$$\% \text{ Lipid} = \frac{NDW \times V}{W \times A} \times 100$$

Where:

NDW = Net Dry Weight (g)
V = Total volume of Extract (mL)
W = Weight of Sample Extracted (g)
A = Aliquot volume of sample used for % lipid (mL)

REPORTING:

The percent lipid is reported as a percentage with three significant figures or two decimal places:

e.g. 23.6%
e.g. 0.34%

SET No: 1

Standard Operating Procedure
Analytical Method

TITLE: Analysis of Polychlorinated Biphenyls (PCB's) by Gas Chromatography

DEPARTMENT: Semivolatile Organics

APPLICATION: This method is used to determine the concentration of PCB's in water soil and biological samples. The following PCB Aroclors are routinely analyzed using this method:

Aroclor 1016	Aroclor 1232	Aroclor 1248	Aroclor 1260
Aroclor 1221	Aroclor 1242	Aroclor 1254	

REFERENCES: Test Methods for Evaluating Solid Waste, 3rd. Ed.
SW846 method 8000B, December 1996
SW846 method 8082, December 1996

PROCEDURE SUMMARY:

A volume of sample extract is injected into a gas chromatograph (GC) and compounds in the effluent are detected by an electron capture detector (ECD). Results are reported in parts per billion (µg/kg or µg/L). Soil and sediment sample results are corrected for moisture and reported on a dry weight basis. Biological results are reported based on wet weight or, "as is".

REVIEWED BY: Daniel M Rude — 1-19-2000
Daniel M. Rude — Date
Organics Group Leader

Gregory J. Graf — 1-19-2000
Gregory J. Graf — Date
Quality Assurance Officer

APPROVED BY: Glen A. Coder — 1-23-2000
Glen A. Coder — Date
Laboratory Manager

SAFETY PRECAUTIONS:

- The toxicity or carcinogenicity of chemicals used in this method have not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each analyst is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. Additional references to laboratory safety are available for the information of the analyst.

- PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. Primary standards of these toxic compounds should be prepared in a hood.

SAMPLE PREPARATION:

Prior to utilizing this analytical method, liquid samples are extracted using Method 3510C or 3520C. Soil and sediment samples are extracted using Method 3540C or 3550B. Biological samples are extracted using Method 3540C. A variety of cleanups may be performed as determined necessary. A column chromatography cleanup using Florisil (En Chem Method SVO-57) typically separates the Aroclors from most other typical environmental interference's in any of the matrices. Soil and sediment samples typically need to have sulfur removed using elemental mercury (En Chem Method SVO-27) or by gel permeation chromatography (En Chem Method SVO-26). A sulfuric acid cleanup may be used, as required, to further remove contaminants (En Chem Method SVO-28).

SAMPLE EXTRACT HANDLING AND STORAGE

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

APPARATUS AND MATERIALS:

Note: Equivalent apparatus and materials to those listed may be used.

Gas Chromatograph:	Hewlett Packard (HP) 5890 equipped with Electron Capture Detectors (ECD)
GC Autosampler:	HP7673A.
Data Processor:	TurboChrom IV.
Printer:	HP laserjet 4M/Plus.
Syringes:	10-1000 µL Gastight syringes (Hamilton series 1000.
Autosampler Vials:	2 mL with crimp top caps.
Detector:	ECD (HP).

GC Columns:

Column 1 - DB-17 Capillary column, 30 m x 0.32 mm I.D. (J&W Scientific).

Column 2 - DB-1701 Capillary column, 30 m x 0.32 mm I.D. (J&W Scientific).

Column 3 - DB-5 Capillary column, 30 m x 0.32 mm I.D. (J&W Scientific).

Column 4 - DB-608 Capillary column, 30 m x 0.53 mm I.D. (J&W Scientific).

GC Column Conditions: Carrier gas - Helium
Flow rate - 2.0 mL/min.
Make-up gas - Nitrogen
Flow rate - 65 mL/min.
Detector temp. -350° C
Injector temp. - 205° C
Splitless injection

GC Temperature Program:
Initial temp. - 110° C
Initial time - 0.5 min.
Rate (1) - 20° C/min.
Hold Time (1) - 0.0 min.
Rate (2) - 11° C/min.
Final temp. - 280° C
Final time - 10 min.

REAGENTS:

Solvents: Hexane, acetone, and isooctane (2,2,4-trimethylpentane) pesticide grade.

Stock Standards Solutions: Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf-life of standard solutions is 1 year from the date of preparation.

Calibration Standards: Prepare a five point curve for AR1016 and AR1260. These may be combined in the same solution (AR1660). Recommended concentrations are 0.1, 0.3, 0.5, 0.8, and 1.0 ug/mL. Prepare solutions of the remaining Aroclors at the mid-point level of the AR1660 curve. Shelf-life of the calibration solutions is 6 months from the date of preparation.

Surrogate Standards: Commercially prepared standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf-life of standard solutions is 6 months from the date of preparation.

INITIAL CALIBRATION:

Primary Column

The initial calibration includes analysis of five point calibration curve of a mixture of Aroclors 1016/1260 at concentrations of 0.1, 0.3, 0.5, 0.8, and 1.0 µg/mL. The Aroclor 1016/1260 calibration curve will also include TCX and DCB at concentrations of 0.01, 0.02, 0.05, 0.1, and 0.15 µg/mL. Inject a single point standard of Aroclors 1221, 1232, 1242, 1248, and 1254 at 0.5 µg/mL. Five or more peaks are selected for each Aroclor, 1016 and 1260. The calibration factor for each of the five peaks is calculated, as shown below, for each of the five levels. Calculate the %RSD for each Aroclor peak using all five calibration points. See Calibration Curve Criteria below.

Other calibration ranges may be substituted to meet expected concentrations of samples being analyzed. If only a select list of Aroclors are being analyzed for, then a five point calibration of only the Aroclors of interest may be substituted for the Aroclor 1016/1260 mixture.

The analyst will pick at least five (three for Aroclor-1221) of the largest peaks for each Aroclor for use in quantifying the samples. The peaks chosen for quantitation should have minimal co-elution with peaks of other Aroclors.

Confirmation Column

Confirmation is generally required using a second GC column of dissimilar stationary phase, or by GC/MS. When simultaneous analysis is performed for confirmation, the same initial and continuing calibration criteria apply.

Since Aroclors provide distinct multiple peak patterns which may be identified by an experienced analyst, and because the identification of an Aroclor is based primarily on this pattern recognition, the need for second column confirmation is not required for sites having a **single** Aroclor. In this case the analyst must document in the raw data the absence of major peaks representing any other Aroclor.

Calibration Curve Criteria: **All Initial calibration and calibration verification criteria apply to both analytical columns when applicable.**

1. Linear Calibration using Average Calibration Factors.

$$CF = \frac{\text{Peak Area}}{\text{Std. Concentration in ug/mL}}$$

The percent relative standard deviation (%RSD) of the five calibration factors for eachpeak, in each Aroclor, 1016 and 1260, must be less than or equal to 20%. If this is the case then linearity can be assumed, and the average calibration factor can be used in place of the calibration curve. If the %RSD is greater than 20%, a calibration curve must be used for quantitation.

2. Calibration alternatives

a. Linear Calibration.

A linear regression is used for a linear equation of the type, y=ax+b. The intercept should **not** be forced through the origin. The regression calculation will generate a correlation coefficient "r". In order to be used for quantitative purposes the r value must be greater than 0.99.

RETENTION TIME WINDOWS:

Retention time windows are generally not applicable to Aroclor analysis since pattern recognition is used to identify the types of Aroclors present. Retention time windows are calculated on each instrument when a new GC column is installed. Additional guidance is provided in method 8000.

1. Make at least three injections all analytes of interest over a 72 hour period.

2. Record the retention time for each selected peak for multi-component analytes, to three decimal places. Calculate the mean and standard deviation for each peak.

3. The width of the retention time window is defined as ± 3 standard deviations of the mean established . The minimum retention window will be ± 0.03 minutes.

4. Establish the center of the RT window for each analyte and surrogate using the absolute RT from the calibration verification standard at the beginning of the analytical shift. Optionally, the Initial Calibration RT windows may continue to be used as long as method criteria are met. For samples run during the same shift as an initial calibration, use the RT of the mid-point standard in the Initial calibration.

CALIBRATION VERIFICATION:

Analysis of Initial Calibration Verification Standard

May be required based on Project Requirements.

In order to consider the initial calibration acceptable, an Initial Calibration Verification Standard (ICV) must be analyzed within the same time clock as the calibration curve. The ICV standard must be

from a second source stock and meet the same criteria as the Continuing Calibration Verification standard before the initial calibration may be considered valid.

Continuing Calibration Verification Standard

All samples must be bracketed by acceptable calibration verifications.

A midpoint calibration check standard is injected following every ten sample injections for calibration verification. If the response factor (area/concentration) of the check standard deviates by more than 15% of the initial average response factor, the calibration is considered out of control and analysis must be stopped.

If the ending calibration verification standard exceeds the 15% criteria on the high side (i.e. an increase in sensitivity) samples which had no Aroclors detected do not need to be reanalyzed. If the continuing calibration standard criteria is exceeded on the low side (i.e. a drop in sensitivity), then the non-positive samples must be re-analyzed because the ability to meet the detection limit is in question. **Any samples injected prior to the failing calibration which do exhibit an Aroclor pattern must be reinjected under a valid calibration.**

Perform corrective action such as injection port or column maintenance. Prior to the analysis of any subsequent samples an acceptable calibration verification must be analyzed. In the event that this cannot be achieved a new initial calibration must be performed.

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA:

1. The percent difference (%D) is determined for all analytes. The %D must be within ± 15% of the calibration curve. (see below)

$$\%D = \frac{R_2 - R_1}{R_1} \times 100$$

where: R_1 = True value of standard.

R_2 = Calculated amount from succeeding analyses using the $\overline{CF}$.

The analyst should verify that the software is using appropriate values for calculations.

SAMPLE ANALYSIS

Once the Aroclor pattern has been identified, compare the responses of 3 to 10 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of

Aroclor is calculated using the individual calibration factor (single point) for each of the 3 to 10 characteristic peaks chosen for that specific Aroclor.

A concentration is determined using each of the characteristic peaks and then those 3 to 10 concentrations are averaged to determine the concentration of that Aroclor.

1. Calculations:

Aqueous samples

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(V_t)(D)}{(Cf)(V_i)}$$

Nonaqueous samples

$$\text{Concentration } (\mu g/kg) = \frac{(A_x)(V_t)(D)}{(Cf)(W)(S)}$$

where:

A_x = Area for selected peak.
V_t = Final volume of extract in mL (adjusting for GPC cleanup)
D = Dilution factor
Cf = calibration factor *
V_i = Initial sample volume (L)
W = Initial sample weight (kg)
S= % Solids/100

* Note: If Aroclor 1016 and/or 1260 is being quantified use the average calibration factor from the AR1660 curve. Use the single point calibration factor for other Aroclors. Surrogates are quantified based on the average calibration factors for TCMX and DCB analyzed with the AR1660 curve.

2. The method blank and LCS extracted along with the samples should be analyzed on the same instrument as the samples.

3. Surrogate recoveries must be evaluated using laboratory control limits (see appendix B). If **both** surrogate recoveries fail this criteria, re-extract the sample. One surrogate is allowed to be outside of the control limits. For instance, if an interfering peak obscures one surrogate, then that one surrogate may be excluded. In the case of a dilution, the analysts discretion should be used.

If the analyst determines that the interferences could be removed by sulfuric acid cleanup and/or sulfur removal, then the analyst will perform the necessary cleanups and re-analyze the samples. The blank will also undergo the same cleanups and be re-analyzed.

QUALITY CONTROL

1. The method blank must meet the surrogate limits (see appendix B). If the blank fails this criteria, all of the associated samples, matrix spikes and laboratory control spikes will be evaluated and a corrective action will be determined.

2. If the blank contains any analyte of interest above the reporting limit (see appendix A), all of the associated samples, matrix spikes, and laboratory control spikes **must** be re-extracted unless the sample concentration is greater than 20X the amount found in the blank or the analyte is not detected in an associated sample. Note: For Wisconsin projects this criteria will be "Above the LOD".

3. If the laboratory control spike does not meet the recovery criteria specified in Appendix B, the results of all QC performed with the samples will be evaluated by the analyst. Corrective actions include re-extraction of the samples or reanalysis of the extracts.

4. Sample matrix spike recoveries should fall within the Laboratory Control limits (see appendix B). If a matrix spike recovery fails this criteria, the recovery of the other spiked sample in the MS/MSD pair should be evaluated. If recovery failures are duplicated then the sample matrix is suspected as the problem and the data should be flagged and the failures discussed in the sample narrative. The LCS recoveries can be used to verify that the method was acceptable for the analysis in a clean matrix.

Note: The Aroclor(s) spiked and/or spike amounts may be adjusted when prior knowledge of the type or concentration of Aroclor(s) present in the sample matrix is known, or to comply with project workplans.

Appendix A
DETECTION LIMITS
for PCB's

Aroclor	En Chem[a] Detection Limit Water(µg/L)	En Chem Reporting Limit Water(µg/L)	En Chem[a] Detection Limit Soil (ug/kg)	En Chem[a] Detection Limit Biota(ug/kg)	En Chem Reporting Limit Soil & Biota(ug/kg)
AR1016	0.33	1.0	10	12	50
AR1221	0.33	1.0	10	12	50
AR1232	0.33	1.0	10	12	50
AR1242	0.33	1.0	10	12	50
AR1254	0.33	1.0	10	12	50
AR1260	0.33	1.0	10	12	50

[a] Method Detection Limit determination, USEPA 40CFR Pt.136, App.B, 1988. Method detection limits are updated periodically, the values currently in use may differ slightly from those published.

Appendix B

MS/MSD
QUALITY CONTROL LIMITS[a]
for PCB's

	Water % Rec.	Soil % Rec.	Biota % Rec.
AR1260	(58-124)	(63-129)	(63-129)

LCS
QUALITY CONTROL LIMITS[a]
for PCB's

	Water % Rec.	Soil % Rec.	Biota % Rec.
AR1260	(69-131)	(64-117)	(64-117)

[a]Limits derived from sample analyses, mean value ± 3SD. Control limits are updated periodically, the values currently in use may differ slightly from those shown above. Biota limits are based on soil recoveries and are advisory only.

Note: LCS and MS/MSD limits based on 1260 are being developed. These limits are based on AR1254 spiking, but should be applicable for use in the interim.

SURROGATE
LABORATORY CONTROL LIMITS[a]
for PCB's

	Water % Rec.	Soil % Rec.	Biota % Rec.
Surrogates			
Decachlorobiphenyl	(DL-148)	(60-155)	(60-155)
Tetrachloro-m-xylene	(52-134)	(41-148)	(41-148)

[a] Limits derived from sample analyses, mean value ± 3SD. Control limits are updated periodically, the values currently in use may differ slightly from those shown above. Biota limits are based on soil recoveries are advisory only.

file: SVO52r3

Attachment H-2

ANSP Protocols for Fish Preservation, Fixation, and Curation

ANSP Protocols for Preparation of Fish Samples for Contaminant Analysis

ANSP Protocols for Extraction and Cleanup of Fish Tissue for PCB and Pesticide Analysis

ANSP Protocols for Quantification of Individual Polychlorinated Biphenyl Congeners (PCBs), Chlorinated Pesticides, and Industrial Compounds by Capillary Column Gas Chromatography

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.

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

Analyte	Matrix	Sample container	Storage	
			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.

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

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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.

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:

$$(mass\ congener)_{sample} = (area\ congener)_{sample}\ X\ RRF_{std}\ X \left(\frac{mass\ istd}{area\ istd}\right)_{sample} \quad (2)$$

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
Rev. 1 (3/95)

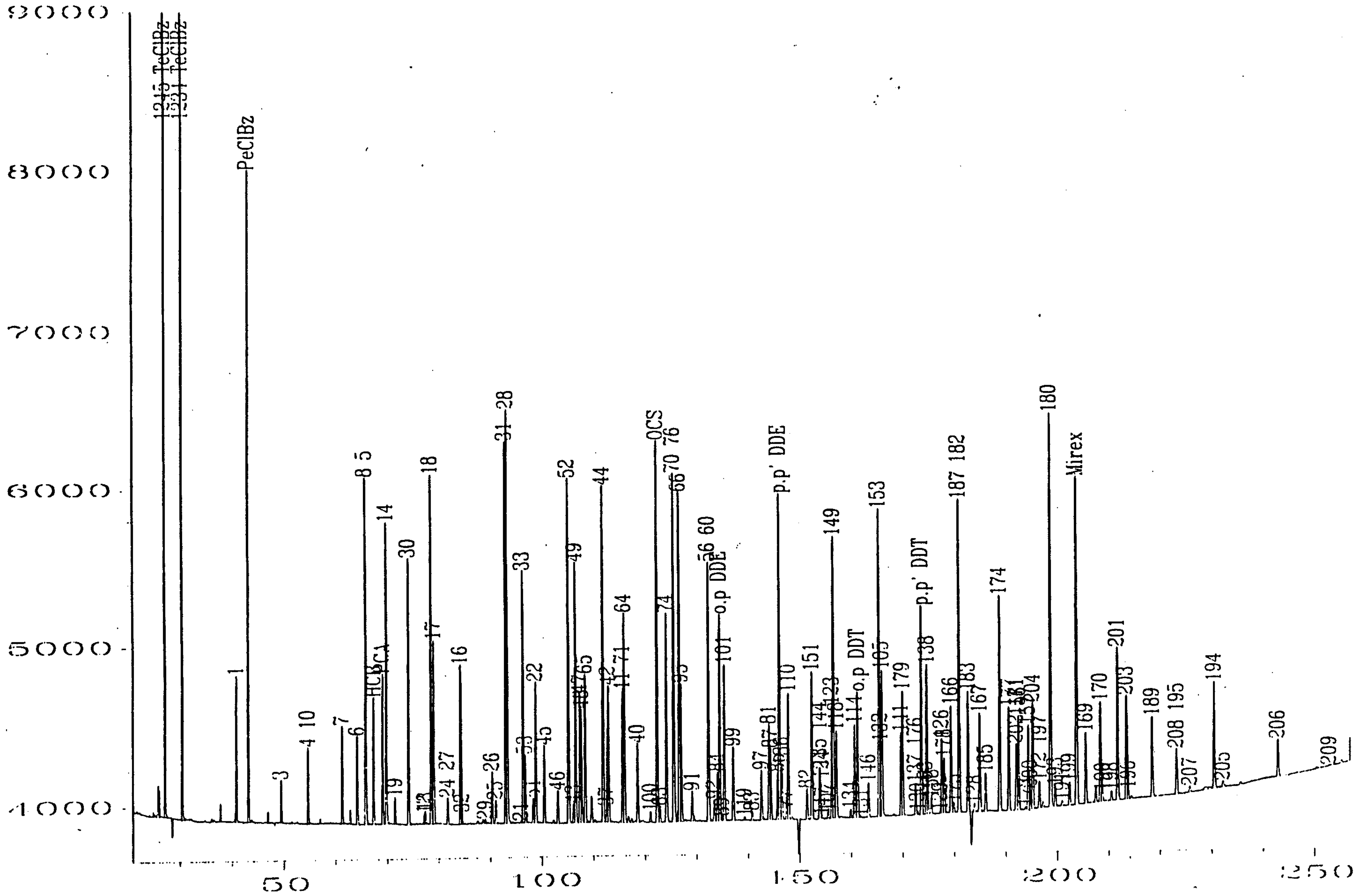
Relative Retention Time[1]	Amount (ng/mL)	Original Amount in Mullin's mix	Compound
0.8283	0.091		131
0.8369	1.6		146
0.8475	16.775		153
0.8487	2.9085		132
0.8503	*6.916*	1.916	105
0.8691	5.2		141
0.8706	*6*		*179*
0.8834	1.388		137+176 (SUM)
0.8855	0.25		130
0.8893	*21.396*		*p,p DDT*
0.8935	4.2967		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.



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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.

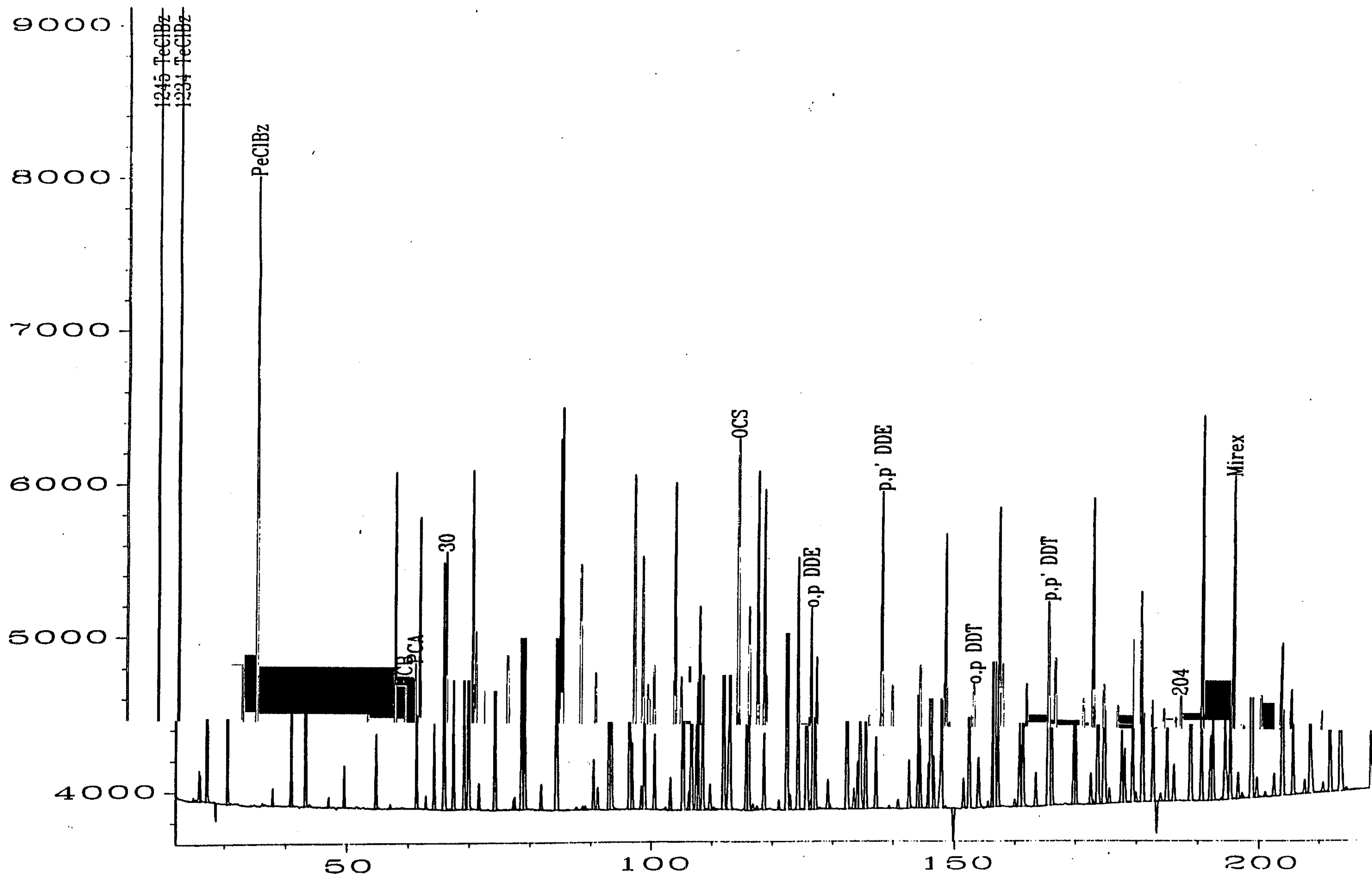
Procedure No. P-16-84
Rev.1 (3/95)

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



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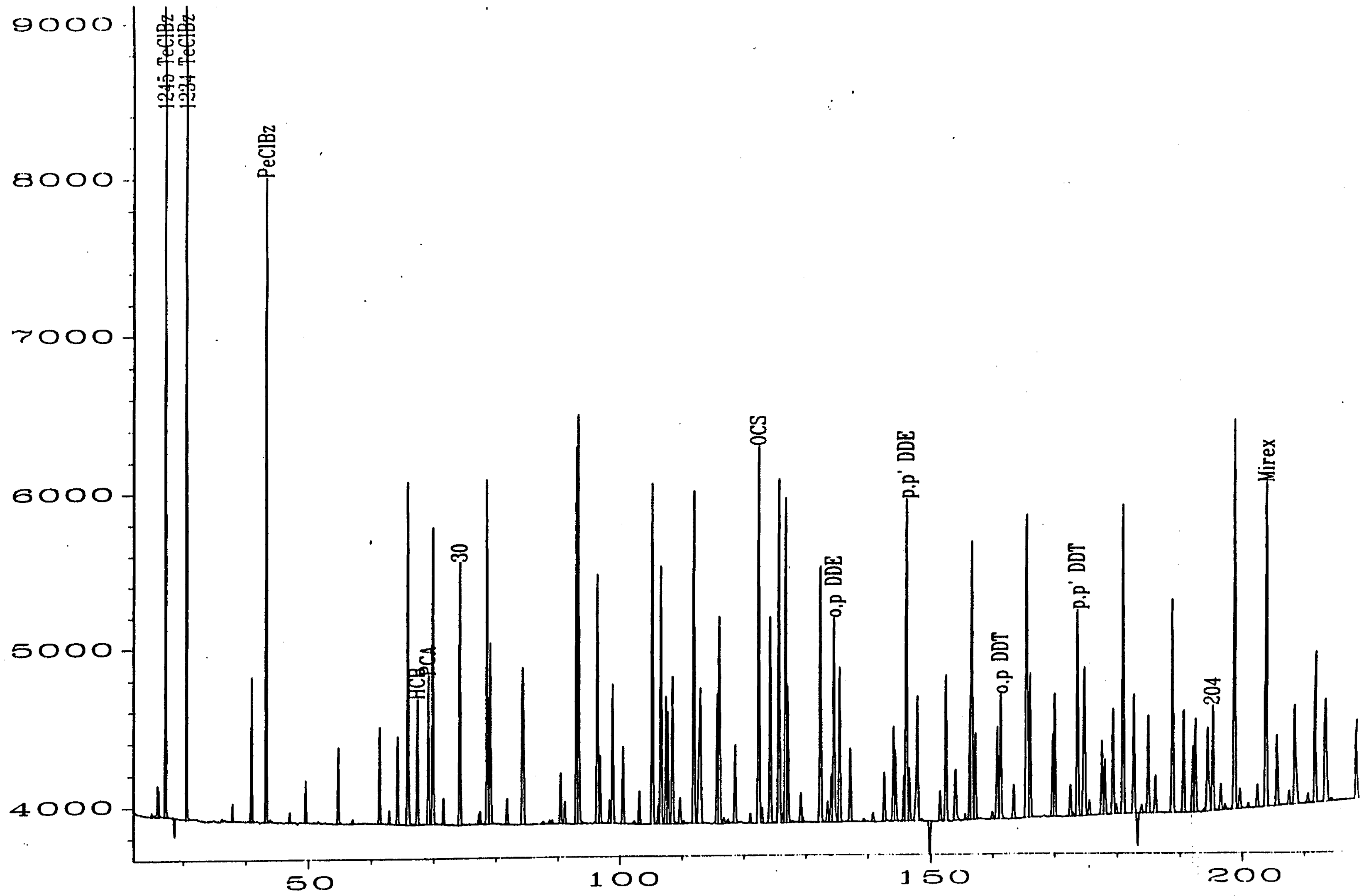

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Attachment H-3
Caged Bivalve Bioaccumulation Study Procedures

Attachment H-3

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.

Materials

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.

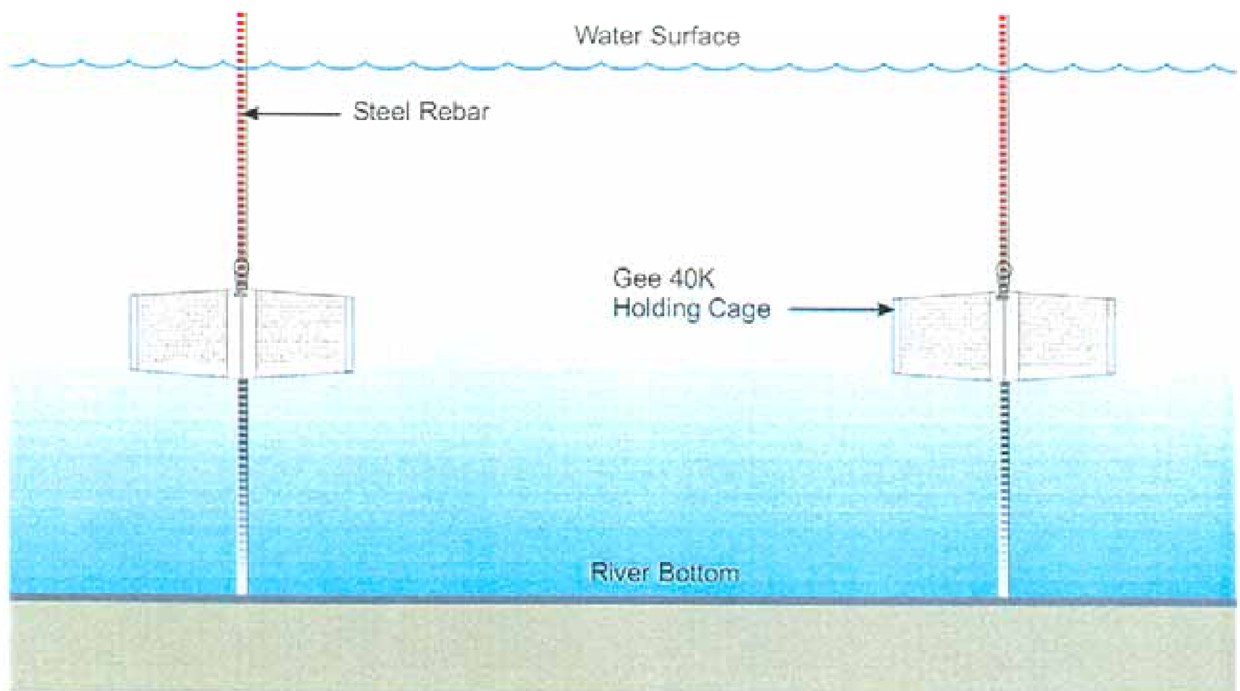
C. If samples are stored temporarily prior to shipment, they will be kept cold and placed in a secured storage area. Coolers will be sealed and custody seals affixed just prior to shipment.

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.



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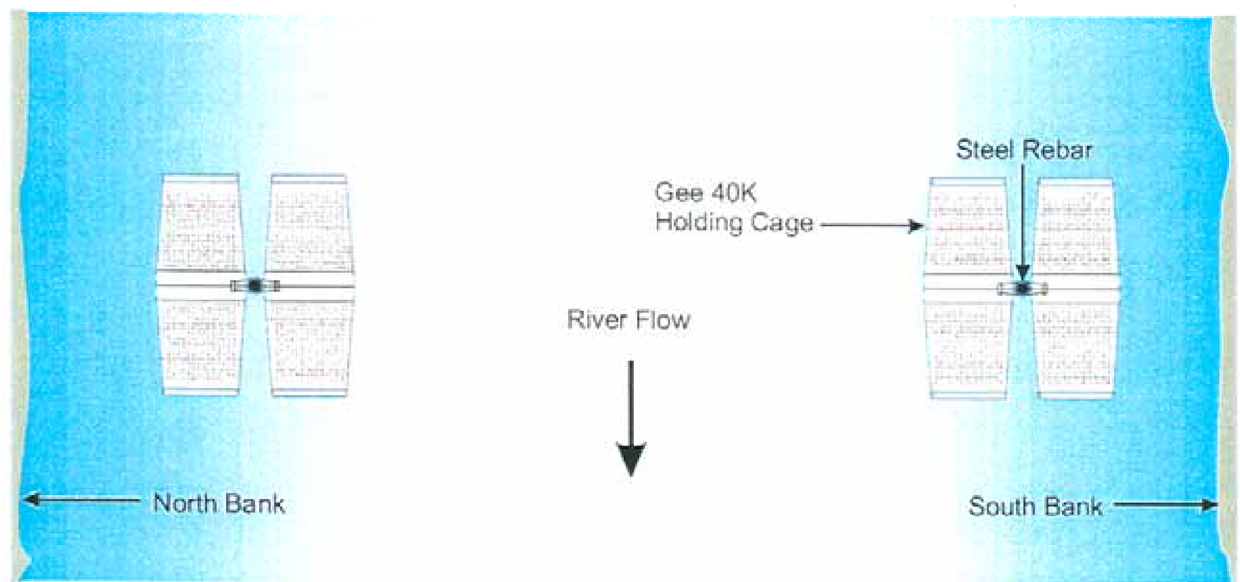

NOT-TO-SCALE

Plan View

GENERAL ELECTRIC COMPANY
PITTSFIELD, MASSACHUSETTS

CAGED BIVALVE IN-SITU EXPOSURE VESSEL SHALLOW WATER SET

BBL
BLASLAND, BOUCK & LEE, INC.
engineers & scientists

FIGURE
H-3A

01/02 SYR-D54-DJH
10111003/10111f01.CDR

Attachment H-4
Benthic Macroinvertebrate Sampling Procedures

Attachment H-4

Benthic Macroinvertebrate Sampling Procedures

I. Introduction

The following procedures describe the general methodologies that will be used in the field to sample the benthic macroinvertebrate community. 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

Equipment

The following collection equipment and materials will be available, as required, during benthic 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;
- Sediment dredge;
- Mesh or sieve screen (Standard U.S. No. 30);
- 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.

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).

Procedures

Benthic macroinvertebrate samples will generally be collected using a sediment dredge and approved sampling techniques. The collection methods to be used to during the benthic community and associated sediment sampling efforts 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, this data will be recorded once for each general area.

3. At each sample location, the opened dredge 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 dredge.

4. Retrieve the dredge into the boat and empty dredge contents into a sieve.

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 dredge sampling does not reoccur over previous sampled areas.

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) benthic macroinvertebrate 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. Affix airbill (if appropriate) with shipper's and consignee's addresses 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.

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:

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.

VII. 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.

Appendix I

Soil Gas Sampling Procedures

Appendix I

Soil Gas Sampling Procedures

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

The instructions presented in this document describe the procedures to collect soil gas samples for the analysis of volatile organic compounds (VOCs) by USEPA Method TO-14 (TO-14). The TO-14 method uses a 6-liter SUMMA® passivated stainless steel canister. An evacuated SUMMA® canister (<28 inches of mercury, Hg) will provide a recoverable whole gas sample of approximately 5.5 liters when allowed to fill to a vacuum of 2"Hg. The whole air sample is then analyzed for VOCs using a quadrapole or ion trap gas chromatograph/mass spectrometer (GS/MS) system to provide compound detection limits of 0.5 parts per billion volume (ppbv).

The following sections provide a list of the necessary equipment and detailed instructions for the installation of soil gas wells and the collection of a soil gas sample for VOCs analysis.

II. Well Installation

Soil vapor wells will be installed using a drill rig equipped with hollow-stem augers as specified in Appendix S. The augers will be advanced to the water table depth or 1 foot above the water table if the depth to water is known. A 1-inch inside diameter (ID) polyvinyl chloride (PVC) well casing with a 1-foot screen will be installed into the boring through the hollow-stem augers. The boring will be backfilled with layers of sand, cuttings, and bentonite while simultaneously withdrawing the augers to seal the well casing in the soil boring. The 1-inch ID well casing equipped with a PVC cap and stainless steel adapter will be used for the collection of a soil gas sample. The well will be developed prior to the collection of a soil gas sample using a battery powered sampling pump and a photoionization detector (PID). The equipment required for the installation of the well is presented below.

A. Equipment and Materials

- Drill rig equipped with hollow-stem augers.
- PVC tubing (1" ID).
- Air sampling pump.
- Rotometer or an electronic flow sensor.
- PID.
- Teflon® tubing.
- PVC caps (1" ID) with stainless steel adapter to fit Teflon® tubing.
- Commercially available clean sand or play sand.
- Bentonite.

B. Installation Procedures

Step 1 - Advance the hollow-stem augers to 1 foot above the water table (if the depth to the water table has been predetermined) or advance the augers until the water table is reached. After reaching the desired depth or the water table, install a 1-inch ID PVC well casing with 1 foot of screen and withdraw the augers.

Step 2 - If the boring contacted the water table, backfill the well casing to 1 foot above the water table with clean sand (this step is not required if the depth to groundwater was predetermined).

Step 3 - Place 1 foot of clean sand into the well casing so that the sand level is higher than the top of the screen.

Step 4 - Withdraw the augers and place 1 foot of bentonite on top of the clean sand layer.

Step 5 - Backfill the boring with the cuttings to 0.5-foot below ground surface.

Step 6 - Fill the remaining soil boring depth to the top of the ground surface with bentonite.

Step 7 - Place the 1-inch ID cap on the well casing and attach sufficient Teflon® tubing to reach the SUMMA® canister (2 to 3 feet).

Step 8 - Purge the soil gas well of at least one well volume (approximately 6 liters for a 10-feet deep well) using a battery powered air sampling pump. Connect a rotometer or electronic flow sensor between the pump and the Teflon® tubing to measure the pump flow rate and purge the well of the desired volume based on the duration of pumping.

Step 9 - Disconnect the battery powered sampling pump and attach the PID to the Teflon® tubing. Record the OVA reading in the field log book and on the sample chain-of-custody (COC) form. The laboratory will use this value to determine the proper dilution for sample analysis.

III. Soil Gas Sample Collection

After installation of the soil gas well, a grab sample will be collected using an evacuated 6-liter SUMMA® canister. An equipment list and the sampling procedures for the collection of a grab sample using a SUMMA® canister are provided below.

A. Equipment and Materials

- Six-liter stainless steel SUMMA® canisters with pressure gauges.
- In-line particulate filters.
- A 9/16" open-end wrench.

B. Sampling Procedures

Step 1 - Record the following information in the field log book (contact the local airport to obtain the information):

- Wind speed and direction;
- Ambient temperature;
- Barometric pressure; and
- Relative humidity.

Step 2 - Attach the particulate filter to the SUMMA® canister sampling train using a 9/16" open-end wrench.

Step 3 - Record the initial vacuum pressure in the SUMMA® canister by attaching the vacuum gauge to the SUMMA® canister with the 9/16" wrench (leave swage-lock cap on the vacuum gauge during this procedure), record in the field log book and on the sample COC form. If the initial vacuum does not register <28"Hg, then the SUMMA® canister **is not** appropriate for use and another canister should be used.

Step 4 - After purging the well with the portable sampling pump and measuring organic vapor levels with the PID, close the valve on the SUMMA® canister and remove the vacuum gauge from the SUMMA® canister. Attach the well tubing to the particulate filter fitting on the SUMMA® canister.

Step 5 - A soil gas sample is collected as a “grab sample” without using a variable- or fixed-flow controller. To collect the soil gas sample after connecting the well tubing to the SUMMA® canister, slowly open the SUMMA® canister valve. Stop collecting sample when the canister vacuum reaches approximately 2"Hg (leaving some vacuum in the canister provides a way to verify if the canister leaks before it reaches the laboratory). To determine when to stop filling, close the valve on the canister when the "hiss" of the filling canister is faint but still audible.

Step 6 - Disconnect the SUMMA® canister from the well cap and measure the vacuum as specified in Step 3. If the remaining vacuum is not greater than 1"Hg, reject the sample and use another SUMMA® canister to collect a sample for laboratory analysis.

Step 7 - Disconnect the SUMMA® canister from the well tubing, remove the filter from the SUMMA® canister, and package the canister 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.

Step 8 - Complete the appropriate forms and sample labels as specified in Appendix L.

IV. Soil Gas Analysis

Soil gas sample analysis will be performed using EPA TO-14 methodology. This method incorporates the use of a quadrapole or ion trap GC/MS with a capillary column to provide optimum detection limits. The GC/MS system requires a 1-liter gas sample (which can easily be recovered from a 6-liter canister) to provide a 0.5 ppbv detection limit. The 6-liter canister also provides several additional 1-liter samples in case subsequent reanalyses or dilutions are required. This system also offers the advantage of the GC/MS detector which provides a confirmation of the identity of detected compounds through the evaluation of their mass spectra. The soil gas samples for this investigation will be analyzed for a specific list of VOCs based on the results from the temporary well point groundwater investigation.

Appendix J

Air Monitoring Procedures

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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 (PM_{10}) 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/m^3.

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/m^3 (0.1$\mu g/m^3$) to 400 mg/m^3.

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/m^3.

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 PM_{10} concentrations at the on-site monitors will be compared with a notification level of 120 $\mu g/m^3$ – which represents 80% of the current 24-hour National Ambient Air Quality Standard (NAAQS) for PM_{10} (150 $\mu g/m^3$). 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 PM_{10} concentration at any on-site monitor exceeds the notification level of 120 $\mu g/m^3$, 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 m^3/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 m^3/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 m^3/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.

<u>Completeness</u> - 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.

<u>Precision</u> - 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%.

<u>Accuracy</u> - 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

   $Q_1 \ldots Q_n$ = flow readings from calibration table corrected to standard conditions (m^3/min)

2. Determine the average flow rate:

   $$QSTD = \frac{Q_1 + Q_2 \ldots Q_n}{N}$$

   where: Q_{STD} = average flow rate in standard conditions (m^3/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:

   $$V_a = \Sigma^{n}_{i-1}(T_i \times F_i)/1000 \text{ L/m}^3$$

   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.

$$CA = \frac{\mu g/PUF}{V_m/PUF}$$

where:

CA = concentration of PCBs in sample (µg/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 exeedance 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_2O	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 instrument

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 13.1 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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Attachment J-1
Laboratory SOP for Extraction Method

STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

NE151_04.DOC

REVISION NUMBER: 04

STANDARD OPERATING PROCEDURE FOR THE EXTRACTION AND PREPARATION OF POLYURETHANE FOAM AIR CARTRIDGES (PUFS) FOR EPA METHOD TO-4A -POLYCHLORINATED BIPHENYLS IN AIR CASSETTE MEDIA

AUGUST 23, 2006

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STANDARD OPERATING PROCEDURE

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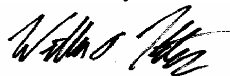
Northeast Analytical, Inc.
Issuing Section: Organic Extractions
SOP Name: NE151_04.DOC
Date Effective: 8/23/2006
Revision: 04

Reviewed by:



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NELAC Annual Review:
Date Last Reviewed: 8/23/06
Reviewed by: W. Kotas

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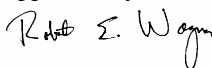

Robert E. Wagner
Laboratory Director

1.0 TITLE

Standard Operating Procedure for the extraction and cleanup of High Volume Polyurethane Foam (PUF) air cassette samples for Polychlorinated Biphenyl (PCB) analysis using the Soxhlet extraction technique (Modified SW-846 Method 3540C/EPA Method TO-4A for subsequent analysis by SW-846 Method 8082. Note: The Determinative Method (EPA Method 8082) requires secondary GC column analysis on dissimilar column for PUF samples.

2.0 PURPOSE

The purpose of this SOP is to provide to the chemist the procedures required to perform extractions of PCBs in PUF (air cassette) samples, using the soxhlet extraction technique and to perform the subsequent extract volume reduction and cleanup.

3.0 SCOPE

The following procedure is utilized by Northeast Analytical, Inc. for the extraction and cleanup of PCBs from PUF (air) samples using the soxhlet extraction method as per Method TO-4A for subsequent analysis by SW-846 Method 8082.

4.0 COMMENTS

4.1 All PUFs must be pre-cleaned using SOP NE0153 (PUF Preparation). All blanks and QC samples use pre-cleaned PUFs.

4.2 Interferences: 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.

5.0 SAFETY

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 and gloves must be worn when handling glassware and samples.

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.

6.0 REQUIREMENTS The chemist 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. The chemist must also be certified to perform the procedure by an approved instructor. The chemist should have completed an acceptable demonstration of precision and accuracy before performing this procedure without supervision.

7.0 EQUIPMENT

7.0.1 Water Cooled Condenser: Pyrex 45/50 #3840-MCO.

7.0.2 250ml Round Bottom Flask: Pyrex #4100.

7.0.3 Soxhlet Repetitive Flushing (reflux) Unit: 45/50 Pyrex #3740-M.

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

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

7.0.6 Boiling Chips: Hengar #5785 Alltech Associates, Inc. (or equivalent)

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

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

7.0.9 Diethyl Ether: Nanograde Mallinckrodt #3434-08

7.0.10 Turbo Vap Evaporator: Zymark #ZW640-3.

7.0.11 Turbo Vap Evaporator concentrator tubes: Zymark 250ml, 0.5ml 1ndpoint.

7.0.12 Beakers: Assorted Pyrex: 250ml, 600mL, and 1000mL, used for liquid containment and pipet storage.

7.0.13 Hexane 90%/10% Ether: 90% Hexane/10% Ether by volume solvent mixture prepared in the lab.

7.0.14 Vials: glass, 4 dram (with Polyseal sealed cap) (20 ml & 10 ml) capacity, for sample extracts.

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

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

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

7.0.18 Florisil: deactivated, solvent washed with 1:1 hexane/ether, baked at 130 C for 16 hours. Deactivated with D.I. water. EM Science #FX0282-1.

7.0.19 Replacement PUFs: 75mm, pre-cleaned and tested. CAT# P226131

7.0.20 Mercury: Triple distilled Mercury Refining Co, Albany, NY #328502. (or equivalent)

7.0.21 Sulfuric Acid: Na_2SO_4 (concentrated) Mallinkrodt #2468 #UN1830. (or equivalent)

7.0.22 Pipets: S/P Disposable Serological Borosilicate Pipets.

1. 1mL X 1/10 #P4650-11X
2. 5mL X 1/10 #P4650-15
3. 10mL X 1/10 #P4650-110

Kimble Pasteur Borosilicate glass pipet 9" #72050 (or equivalent)

7.0.23 Quartz Microfiber Filters(QMF): 10.16 cm Dia., 100 circles(Whatman Cat# 1851-101) or equivalent Baked at 450 degrees Celsius for 3 hours.

7.0.24 Surrogate Spike Solution: Laboratory prepared from primary stock solution Tetra-Chloro-meta-Xylene and Decachlorobiphenyl at 0.500 ug/mL.

7.0.25 Laboratory Control Spike Solution: Laboratory prepared from primary stock solution of PCB Aroclor at 0.500 ug/mL

8.0 PROCEDURES:

8.1 Sample Preparation

8.1.1 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 extraction logbook.

8.1.2 Before any steps are taken, the chemist should first review the sample job folder and fill in the appropriate spaces on the internal sample tracking form and initial.

8.1.3 Prior to extraction all surfaces and fume hoods used must be cleaned and wiped down with hexane. It is also advisable to remove any PCB solid or liquid waste containers from the fume hood

8.1.4 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.

8.1.5 Use extreme caution using Ether during this extraction. Ether and its vapors are extremely flammable and must be used in a fume hood.

8.2 Procedure: Sample Extraction

8.2.1 Rinse enough 250 mL round bottoms and soxhlets for each sample, blank, and QC sample. Place in fume hood and let dry.

8.2.2 After the glassware dries, add a few boiling chips to each round bottom and add approximately 200 mL of Hexane 10% Ether mixture to each round bottom. Label them with a sample ID.

8.2.3 Blank, QC PUFs, and baked QMF filters should be prepared prior to extraction using the PUF preparation SOP (NE153). Each sample must be handled using a clean pair of gloves. Use the pre-cleaned replacement PUFs and pre-baked QMF filters for the Blank and QC samples.

8.2.3.1 For each sample, use a pair of tweezers to pull the PUF out of its PUF tube and push it into the appropriate soxhlet. Try to handle as PUF as little as possible. Using pair of tweezers depress both sides of the PUF and push the PUF to the bottom of the soxhlet.

8.2.3.2 Using tweezers, fold the glass fiber filter that came with the sample and push it into the soxhlet. Use the tweezers topush 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.

8.2.3.3 Place the soxhlet onto the appropriate round bottom. Put clean gloves on and repeat with each sample.

8.2.4 Add Spike and Surrogate mixture to appropriate samples. See table below.

Table 8.4.2

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	0.500 ug/mL	0.500 mL
Aroclor 1221 Spike mix in hexane	0.500 ug/mL	0.500 mL
Aroclor 1232 Spike mix in hexane	0.500 ug/mL	0.500 mL
Aroclor 1242 Spike mix in hexane	0.500 ug/mL	0.500 mL
Aroclor 1248 Spike mix in hexane	0.500 ug/mL	0.500 mL
Aroclor 1254 Spike mix in hexane	0.500 ug/mL	0.500 mL
Aroclor 1260 Spike mix in hexane	0.500 ug/mL	0.500 mL

8.2.5 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. Turn on chiller to cool the condensers.

8.2.6 Place the round bottom flask with attached soxhlet extractor onto a heating mantle and attach condenser unit. Turn corresponding thermostats on to setting 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 three or more flushes per hour should be achieved.

8.2.7 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 pipet volumes of hexane. Disengage the soxhlet and condenser unit and rinse the joint off as well into the soxhlet.

8.2.8 Move all round bottom and soxhlet units to the fume hood. The diethyl-ether in the samples will continue to release vapors. Using 10 mL pipets push the PUF down to release solvent from the PUF and allow the unit to drain into the round bottom.

8.2.9 Rinse the connecting joint into the round bottom. Set the soxhlet aside at this time and leave it in the hood to evaporate the remaining diethyl -ether. Procure the same number of turbo tubes as there are samples. Rinse all the turbotubes with hexane and let dry. 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 pipet and hexane to rinse the last drop out of the mouth of the round bottom. Rinse the round bottom with several pipetfuls of hexane, swirl gently, and decant into same turbo tube. Repeat this step twice for same sample, then repeat all preceding steps for all other samples.

8.2.10 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.

8.3 Solvent Reduction: TurboVap Evaporator System

8.3.1 The TurboVap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The turbovap uses a heated water bath and positive pressure nitrogen flow/vortex action. The 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.

8.3.2 Turn the unit on (switch is located on the back side 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.

8.3.3 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. Make sure to wipe down all surfaces with hexane before concentration samples.

8.3.4 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.

8.3.5 The process for solvent (hexane/ether) reduction takes approximately 20-30 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.

8.3.6 Concentrate the solvent to approximately 1.0 mL. Remove the samples from the turbovap and place in the rack. The remaining solvent will consist largely of hexane since the ether component is fractionally removed at a faster rate than hexane; however, a solvent exchange with hexane should be completed 3x to ensure the ether has been entirely removed. 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 1.0 mL point is achieved. Samples which stop reducing should be removed as soon as possible.

8.3.7 Quantitatively transfer the sample extract with a disposable transfer pipette into an appropriate volumetric flask (5mL for PUF extracts). Rinse Turbotube with 2 pasteur pipets of hexane, then transfer the rinse to the volumetric. After the sample has been transferred, rinse the disposable transfer pipet with 0.5mL 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 4 dram vial.

8.3.8 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.

8.4 Sample Extract Cleanup

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.

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 the extraction logbook.

Sample extract cleanups are performed on set volume extracts. The final concentrated extract volume is 5 mL for PUF samples.

8.4.1 Sulfuric Acid Wash

8.4.1.1 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds which are co-extracted with the PCB residues.

8.4.1.2 Chill the sample to approximately 0^0C. Add 2.0 mL concentrated H_2S0_4 and shake for 30 seconds by hand, centrifuge for approximately 1 minute on a setting of #4, transfer the hexane upper layer to 4 dram vial.

8.4.1.3 Repeat 8.4.2 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.

8.4.2 Elemental Sulfur Clean-up

8.4.2.1 Elemental sulfur is soluble in the extract solvents used for sediment and soil 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.

8.4.2.2 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.

8.4.3 Removal of Sulfur Using Mercury

Note: Mercury is a highly toxic metal. All operations involving mercury should be performed within a hood. Prior to using mercury, the chemist should become acquainted with proper handling and emergency spill/clean-up procedures associated with this metal and must have reviewed the material safety data sheet MSDS.

8.4.3.1 Add 1-3 drops of mercury to the sample extracts, cap, and place on the wrist shaker for 30 mins. 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.

8.4.3.2 Remove the sample extracts from the wrist shaker and place in the centrifuge for 2 minutes on speed setting on #4.

8.4.3.3 Transfer the sample extract to a clean 4 dram vial.

8.4.3.4 The precipitated sulfur can be removed from the extract by performing a sulfuric acid clean-up or a Florisil slurry.

8.5 Florisil Adsorption (Slurry)

8.5.1 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.

8.5.2 Add approximately 0.5 grams of tested and approved deactivated florisil to each vial containing

the sample extract. **SEE EXTRACTION SUPERVISOR FOR THE APPROPRIATE DEACTIVATION CONCENTRATION TO BE USED.**

8.5.3 Vigorously shake the vial for approximately 1 min by hand or on the wrist shaker.

8.5.4 Place the vial(s) into the centrifuge for 2 minutes on setting #4.

8.5.5 Transfer the extract to a clean 4 dram vial.

8.6 Extract Screening and Dilution:

8.6.1 PCB extracts are ordinarily 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.

8.6.2 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. For example, a 1 to 10 dilution is performed by adding 1.0 mL of the extract to 9.0 mL hexane. The vial containing the diluted extract is labeled denoting the equivalent extract volume after the dilution; e.g. a 5mL extract diluted 1 to 10 is labeled "250X", an undiluted 25mL extract is labeled "25X".
When high dilutions are prepared, secondary (serial) dilutions of the initial diluent are prepared; e.g. a 100 fold dilution is prepared by a 1 to 10 dilution of the initial extract,then a 1 to 10 dilution of the resulting diluent.

8.6.3 Perform the dilution using an appropriate class A disposable volumetric pipet to transfer the extract and a calibrated volumetric autodispensette to dispense 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.

8.6.4 Transfer 1 mL of the extract to a labeled 1.5 mL GC autosampler vial. Record the screening dilution and Set Volume in the Log Book, enter data into the LIMS. Create a Access Report Sheet prepared in LIMS. Submit the Access Report Sheet, a photocopy of the logbook, and completed internal chain of custody tracking form with the sample extracts to the GC analyst.

9.0 Quality Assurance/Quality Control

9.1 Verification PUF sample:

Averification (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

9.1.1 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).

9.1.2 Submit extract for analysis by GC-ECD (EPA Method 8082) as described in 8.6.4.

9.1.3 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.

9.2 Laboratory Method Blank

9.2.1 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.

9.3 Laboratory Control Spike/ Laboratory Control Spike Duplicate Sample

9.3.1 A laboratory control spike(LCS)/ laboratory control spike duplicate (LCSD) sample is prepared by spiking a pre-cleaned PUF cassette and filter with an 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 8.2.4 for spike information. The percent recovery must meet project specified or laboratory established limits. The default Recovery Limit is 60-140 %.

9.3.2 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.

9.3.3 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.

9.4 Field Spike Sample

9.4.1 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 9.3.

9.5 Surrogate Spike

9.5.1 Every site sample and QC sample is spiked with the TCMX/DCBP surrogate solution described in table 8.4.2. 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.

9.6 Field Blank Sample

9.6.1 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.

10.0 Sample Collection, Preservation and Holding Times

10.1 Samples are collected as per EPA method TO-4A and the client's Field Sampling and Analysis Plan. Northeast Analytical does not provide field sample collection services for air monitoring projects. Samples should be stored at < 4 degrees Celsius until shipping to laboratory.

10.2 Field samples are shipped to the laboratory in a cooler chilled with Ice (<4 Degrees Celsius).

10.3 Upon receipt samples are stored in laboratory under refrigeration at < 4 degrees Celsius until extraction.

10.4 Samples must be extracted within 7 days of collection and analysis must be performed within 40 days of extraction.

11.0 References

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

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

3. Guide to Environmental Analytical Methods, Genium Publishing Corporation, Schenectady, NY 12304. 1997

12.0 Attachments

A. Method Outline Summary

ATTACHMENT A: METHOD OUTLINE

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 16 HOURS
6. BREAKDOWN SOXHLET EXTRACTOR APPARATUS
7. TRANSFER SOLVENT TO TURBO TUBE
8. SOLVENT REDUCTION, USING THE ZYMARK TURBOVAP EVAPORATION SYSTEM
9. TRANSFER AND SET VOLUME
10. EXTRACT CLEANUP (ACID, MERCURY OR TBA, FLORISIL)
11. EXTRACT DILUTION
12. GC SCREENING/ ANALYSIS

Attachment J-2
Laboratory SOP for Test Method

STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

NE148_05.DOC
REVISION NUMBER: 05

Standard Operating Procedure for the Determination of Polychlorinated Biphenyl (PCB) Aroclors by US-EPA SW-846 Method 8082

AUGUST 24, 2006

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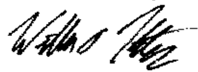
Northeast Analytical, Inc.
Issuing Section: GC Organics
SOP Name:NE148_05.DOC
Date Effective:24-August-2006
Revision: 05

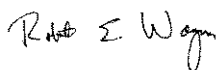
Reviewed by



William A. Kotas
QA Officer

NELAC Annual Review:
Date last reviewed: 24-August-2006
Reviewed by: W. Kotas

Approved by:



Robert E. Wagner
Laboratory Director

1.0 Title: EPA SW 846 Method 8082- Polychlorinated Biphenyl (PCB) Aroclors by Capillary Column GC

Standard operating procedure for the analysis of Polychlorinated Biphenyls by Gas Chromatography with Electron Capture Detection and Total Aroclor Quantification.

2.0 Purpose

The purpose of this SOP is to provide a detailed written document for measurement of Polychlorinated Biphenyls (PCBs) according to SW-846 Method 8082 specifications.

3.0 Scope

3.1 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, and tissue samples, air cassette samples including polyurethane foam (PUF) and associated filters for EPA Methods TO-4A and TO-10.

3.2 The following analytes can be determined by this method:

Analyte	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

3.3 In general, samples are extracted, or in the case of oils and waste solvents diluted, with a pesticide grade solvent. The extracts are further processed by concentrating or diluting, depending on the PCB concentration, and carried through a series of clean-up techniques. The sample is then analyzed by injecting onto a gas chromatographic system and detected by an electron capture detector.

3.4 This SOP provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCBs by gas chromatography. Each matrix requires different sample handling or special preparation procedures before analysis can be performed. Each sample will be covered separately in the extraction standard operating procedures.

4.0 Comments

4.1 One of the major sources of interference in the analysis of PCBs is that organochlorine pesticides are co-extracted from the samples. Several of the commonly found pesticides and associated degradation products (DDT, DDE, DDD) overlap the PCB profile envelope and co-elute with several PCB GC peaks and therefore cannot be accurately measured. The analyst must be careful in chromatographic pattern review and flag peaks that are suspected of being contaminated so that they are not included in the total PCB values generated.

4.2 Laboratory contamination can occur by 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 respond on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification.

5.0 Safety

5.1 Safety glasses and disposable gloves must be worn when handling samples and extracts.

5.2 All manipulations of sample extracts should be conducted inside a chemical fume hood. The analyst should minimize manipulation of sample extracts outside of a fume hood.

5.3 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 of handling applicable solvents and chemicals used to process

samples. The analyst should refer to the laboratory chemical hygiene plan for further safety information.

5.4 Samples remaining after analysis should be either returned to the customer for disposal or disposed of through the laboratory's disposal plan. Refer to the sample custodian for

assistance in this matter and also standard operating procedure NEO54, disposal of laboratory waste.

6.0 Requirements

6.1 Extensive knowledge of this standard operating procedure and EPA SW-846 Method 8082 is required.

6.2 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.

7.0 Equipment

7.1 Instrumentation

7.1.1 Gas chromatograph: Varian Model 3400, equipped with Model 1077 split/splitless injector, temperature programmable oven, electron capture detector, Model 8100 autosampler.

7.1.2 Dual Column Gas Chromatograph: Varian CP-3800, Dual Injector System split/splitless, Varian CP-8400 autosampler, Dual Electron Capture Detectors. Column 1- 30 Meter 0.25 uM I.D. Phenomenex ZB-1 Column, Column2 -30 Meter 0.25 uM I.D. Phenomenex ZB-5 Column

7.1.3 Chromatograph Data System: A data system for measuring peak height and peak area. A Millennium_32 computer network based workstation (Waters Associates), will be employed to capture detector response and digitally store the chromatographic information. This system will allow for chromatographic review of data from the gas chromatograph, electronic peak integration for precise calculations, database structuring of the analytical information, and archival capabilities.

7.2 Glassware and Accessories:

7.2.1 25 mL volummetric flasks, Class A, acid washed, (Baxter Cat. No. F4635-25)

7.2.2 5 mL volummetric flasks, Class A, acid washed (Baxter Cat. No. F4635-5)

7.2.3 10 mL volummetric flasks, Class A, acid washed (Baxter Cat. No. F 4635-10)

7.2.4 50 mL volummetric flasks, Class A, acid washed (Baxter Cat. No. F4635-50)

7.2.5 100 mL volummetric flasks, Class A, acid washed (Baxter Cat. No. F4635-100)

7.2.6 4 dram vials for sample extract storage
(Kimble Opticlear, part no. 60910, code no. 60910-4)

7.2.7 8 dram vials for sample extract storage (Kimble Opticlear, part no. 60910, code no. 60910-8)

7.2.8 Pasteur pipettes (Kimble, part no. 72050)

7.2.9 250 mL beakers, glass (Baxter Cat. No. B2650-250)

7.2.10 100 mL beakers, glass (Baxter Cat. No. B2650-100)

7.2.11 Disposable 10 mL pipettes (Baxter P4650-110)

7.2.12 Disposable 5 mL pipettes (Baxter P4650-15)

7.2.13 Disposable 1.0 mL pipette (Baxter P4650-11X)

7.3 Chemicals:

7.3.1 Hexane, Burdick and Jackson, (Cat.No. 216-4)

7.3.2 Acetone, Burdick and Jackson, (Cat.No.010-4)

7.3.3 Toluene, Baker, (Cat.No. 9336-03)

7.3.4 Methylene Chloride, Burdick and Jackson, (Cat. No. 300-4)

7.4 Analytical Standard Solutions:

7.4.1 Aroclor Stock Standard Solutions

7.4.1.1 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.

7.4.1.2 Stock standards are prepared from individual Aroclor formulations by weighing an exact amount of the neat material to the nearest 0.1 mg, 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. For DCBP, dissolve neat formulation in 10 mL of toluene and then transfer to a 100 mL volumetric flask bringing to volume with hexane.

7.4.1.3 The stock standards are transferred into Boston bottles and stored in a refrigerator at 0-6°C, protected from light.

7.4.1.4 Stock PCB standards must be replaced after one year, or sooner if comparison with certified check standards indicate a problem. See 8.5.3 for limits.

7.4.1.5 For quality control and general labeling requirements refer to standard operating procedure NE050, Preparation of Standards.

7.4.2 Calibration Standards:

7.4.2.1 Calibration standards are prepared at five concentration levels using a prepared working standard. See Attachment A, Table 2 for the preparation and exact concentrations of the working standards. The following five standards make up the initial calibration curve standard set: 20 ng/mL, 100 ng/mL, 250 ng/mL, 500 ng/mL, 1000 ng/mL.

7.4.2.2 The two surrogates TCMX and DCBP are included in the A1254 calibration standards. The stock standard for TCMX is prepared by diluting 1 mL of TCMX solution (ULTRA, cat. #IST-440, at 2000 ug/mL) into a 100 mL volumetric flask resulting in a solution of TCMX at 20 PPM.

7.4.2.3 To prepare the working surrogate standard, pipet 5.0 mL of the 100ppm DCBP stock standard and 2.5 mL of the 20ppm TCMX stock standard into a 100 mL volumetric flask and bring to volume with hexane.
The final concentration of this solution will be 5.0ppm of DCBP and 0.5ppm of TCMX.

7.4.2.4 Refer to Attachment A, Table 4 for instructions on
preparation of the calibration standards containing A1254 and the surrogates. Refer to Attachment A, Table 3 for instructions on preparing the remaining calibration standards.

7.4.2.5 Transfer all calibration standards to 8 dram 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. See 8.5.3 for limits.

7.4.3 Continuing Calibration Standards:

7.4.3.1 Continuing calibration check 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.

8.0 Procedure

8.1 Gas Chromatographic Operating Conditions

8.1.1 Establish the gas chromatograph (GC) operating parameters as follows:

Autosampler parameters: Multi-vial mode, ECD Attenuation and range are 1.

Refer to Attachment C for all other GC operating procedures.

Note: GC helium gas flow is optimized after instrument maintenance by adjusting nitrogen flow to elute a PCB calibration standard to a known retention time.

8.2 Data Acquisition:

8.2.1 Chromatographic information will be collected and processed utilizing a computer based data acquisition workstation (Waters Associates, Millennium_32 computer network based workstation) The GC workstation acquires the millivolt detector signal, performs an analog to digital conversion and stores the digital chromatogram on the computer network's disk. The chromatography software performs all data reduction including, long term data storage on magnetic media, chromatographic peak integration, all calculations, report generation, chromatogram plots, and calibration functions.

8.3 Initial GC Calibration:

8.3.1 GC calibration will be performed by the external calibration procedure. Prior to running samples the system must be calibrated and system performance must be verified.

8.3.2 Establish the gas chromatographic operating parameters outlined in Section 8.1 and prepare the calibration standards at the five concentrations outlined in Section 7.4.2.

8.3.3 Inject each calibration standard using the GC autosampler and the parameters outlined in section 8.1, which are those used for actual samples.

8.3.4 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 group number for calibration purpose.

Aroclor	Group Number
A1221	1
A1232	2
A1016	3
A1242	4
A1248	5
A1254	6
A1260	7

8.3.5 Attachment D is an example of chromatograms of standards of each Aroclor for a DB5-MS column with peaks selected for calibration labeled.

8.3.6 For the initial calibration curve to be considered valid, the percent relative standard deviation must be less than 20% over the working range. The calibration curve is used for quantification in every case and is not replaced with the average calibration factor. See attachment E for an example of response factors and the calculation of the percent relative standard deviation.

8.4 Retention Time Windows:

8.4.1 The GC system should be checked by the analyst to make sure it is functioning properly before establishing retention time windows. Make three injections of each Aroclor at a midlevel concentration throughout a minimum 72-hour time period.

8.4.2 For the 5 peaks selected for calibration of each Aroclor, calculate the standard deviation resulting from the variation in the three retention times for that peak.

8.4.3 The retention time window is defined as plus or minus three times the standard deviation of the three retention time determinations.

8.4.4 If the standard deviation of the selected peak is zero, the standard deviation of the peak eluting after it is used. If it is the last eluting peak that has zero for the standard deviation, then substitute the standard deviation of the peak eluting before the last peak.

8.4.5 Retention time windows established in section 8.4.3 to 8.4.4 may not be practical when samples containing matrix interferences are injected onto the GC column. The default R.T. Window of +/- 0.07 minutes is employed when the established windows are too narrow. Besides using retention time windows to assign peaks for quantification, the analyst should rely on their experience in pattern recognition of multi-response chromatographic response exhibited by PCB Aroclors.

8.4.6 Attachment F provides examples of calculated retention time windows generated by the above outlined procedures.

8.5 Gas Chromatographic Analysis:

8.5.1 Prior to conducting any analyses on samples, calibrate the system as specified in Section 8.3

8.5.2 To start an analytical sequence, the 500 ppb calibration standard is injected and analyzed for the seven Aroclors that the system is calibrated for, if more than 24-hours has elapsed since the last valid continuing calibration check standard. If less than 24-hours has elapsed since the last valid continuing calibration check standard, select one 500 ppb continuing calibration check standard. Selection of the continuing calibration check standard should be based on anticipated Aroclor contamination that the samples may exhibit. Selection of the continuing calibration check standard should also be alternated among the seven Aroclors.

8.5.3 The calculated value for the continuing calibration check standard must be $\pm$15% for it to be valid and analysis to proceed. If this 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.

8.5.4 The daily retention time windows must be established. Use the retention time for the selected 5 peaks of the continuing calibration check standard as the midpoint of the window for that day. If all seven Aroclors were analyzed as the initial continuing calibration check standard, then establish retention time windows using the retention time plus or minus the windows established in Section 8.4. If only one Aroclor was analyzed as the continuing calibration check standard (i.e., less than 24-hours had elapsed), use the retention time from this standard as the midpoint plus or minus the windows established in Section 8.4. to establish the daily retention time windows. For the remaining six Aroclors, go back to the previous time all seven Aroclors were analyzed as the initial calibration check standards and use those retention times plus or minus the windows established in Section 8.4 to develop daily retention time windows.

8.5.6 All succeeding continuing calibration check standards analyzed during an analysis sequence must also have a percent difference of 15% or less when compared to the initial calibration generated from the 5 point calibration curve.

8.5.7 All succeeding standards in an analysis sequence should exhibit retention times that fall within the daily retention time window established by the first continuing calibration check standard(s) of that analytical sequence. If the retention times are outside the established windows instrument maintenance must be performed and recalibration may be required.

8.5.8.1 The following is 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 every tenth injection in the

analytical sequence. All analytical sequences must end with a continuing calibration check standard regardless of the number of samples analyzed.

ANALYTICAL SEQUENCE

INJECTION	MATERIAL INJECTED
1	Hexane Blank
2-36	Initial Calibration Standards
37-43	Continuing Calibration Check Standard
44-52	Samples analyses, including method blanks, matrix spikes, matrix duplicates, matrix spike duplicates, and QC reference check standard. A maximum of 9 samples between continuing calibration check standards.
53	Continuing calibration check standard
54 and higher	repeat inject. 44-53 sequence

8.6 Quality Control:

8.6.1 This section outlines the necessary quality control samples that need to be instituted at the time of sample extraction. The data from these quality control samples is maintained to document the quality of the data generated.

8.6.2 Each analyst must demonstrate competence in accuracy and precision on quality control samples before beginning analysis on samples. This demonstration must be on-going and be repeated if there is any modification to the method.

8.6.3 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 organic-free reagent water blank is processed. For soil, sediment, and solid waste samples, a laboratory sodium sulfate blank is processed. For fish and other biota samples, a sodium sulfate blank is processed. For oil samples, a PCB-free blank control oil is processed. For Air Cassette samples a pre-cleaned PUF and PUF filter are extracted for the method blank.

8.6.4 The method blank must exhibit PCB levels less than the matrix defined practical quantification limit (PQL). If the method blank exhibits PCB contamination above the reportable PQL, 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.

8.6.5 A matrix spike is to be analyzed at a rate of 1 matrix spike per every 20 samples. Also a matrix spike duplicate or duplicate sample is to be analyzed at a rate of 1 per every 20 samples. Duplicate samples may be appropriate in place of matrix spike duplicates, for soil and waste samples, where detectable amounts of organics are present.

8.6.6 Analyze one unspiked and one spiked sample. Calculate the percent recovery based on Aroclor concentration of both samples as follows:
A = concentration of spiked sample
B = concentration of unspiked sample (background)
T = known true value of the spike
Percent Recovery (p) = 100 (A-B) %/T

Compare the percent recovery calculated with the lab established limits or the default limits of 70-130% if lab limits are not available. If the concentrations of the matrix spikes are *greater* than five times the calculated sample amount then the quality control limits should be applied. If the concentrations of the matrix spikes are *less* than five times the sample than there are no established limits applicable. If the percent recovery falls outside the acceptance range for the given Aroclor used as the spiking analyte, then the matrix spike recovery failed the acceptance criteria. Inform quality control manager and document matrix spike recoveries.

A relative percent difference (RPD) must also be calculated on the matrix spike set recoveries. This is calculated as follows:

A = % recovery of matrix spike sample
B = % recovery of matrix spike duplicate sample

RPD = [(A-B)/{(A+B)/2}] x 100
where (A-B) *is taken as an absolute value*

If the concentrations of the matrix spike set are *greater* Than five times the calculated PQL then an RPD of twenty percent or less is acceptable. If the concentrations of the matrix spike set are *less* than five times the PQL than there are no established limits applicable to the RPD.

8.6.7 A QC reference check standard (laboratory control spike sample) is also prepared and analyzed. Spike one liter of laboratory organic free water, extract, and analyze. Calculate the percent recovery for the Aroclor spike and compare to the lab established limits or the default limits of 70-130% if lab limits are not available. If the percent recovery for the QC reference check standard (laboratory control spike sample) is out of criteria, the analysis is out of the control for

that analyte and the problem should be immediately corrected. The entire batch of samples will need to be re-extracted and re-run.

8.6.8 Method accuracy, based on matrix spike data, is maintained by the laboratory as part of the QC program. For each sample matrix, upper and lower warning and control limits for method performance are established.

8.6.9 Surrogate compounds are added to each sample, matrix spike, matrix spike duplicates, duplicate, method blank, and QC reference check standard (laboratory control spike sample) at time of extraction. Surrogate compounds chosen for this method are tetra-chloro-meta-xylene (TCMX) and decachlorobiphenyl (DCB). The following are typical surrogate amounts added to normal encountered matrices. These amounts may be adjusted by the analyst, if PCB background levels are high and surrogates are being diluted out of analysis range.

Water: 1.0 mL of 0.05ppm TCMX/0.5ppm DCB
Soil & Sediment: 0.5 mL of 0.5ppm TCMX/5.0ppm DCB
Oil: 0.5 mL of 0.5ppm TCMX/5.0ppm DCB
Wipes: 0.5 mL of 0.5ppm TCMX/5.0ppm DCB
PUF cassettes: 0.5 mL of 0.5ppm TCMX/5.0ppm DCB

8.6.10 Surrogate percent recovery data for each matrix is tabulated as part of the on-going laboratory QC program. The standard deviation is calculated once enough surrogate data is available for each matrix, typically based on 25 to 30 samples. Upper and lower warning limits ($p \pm 2SD$) and upper and lower control limits ($p \pm 3SD$) are established.

8.6.11 Only one surrogate analyte needs to meet established control limits for the analysis to be valid. The data is compared to the lab established limits or the default limits of 70-130% if lab limits are not available. If percent surrogate recovery is not within limits for either surrogate, the following steps are required:

8.6.11.1 Review calculations that were used to generate surrogate percent recovery values to make certain there are no errors.

8.6.11.2 Check by GC analysis surrogate solutions used during sample extraction steps to ensure that no problems exist with spiking solutions.

8.6.11.3 Re-analyze the extracts that did not meet control limits, either at the previously analyzed dilution or at a more dilute level to assess if the sample matrix interfered with surrogate measurement.

8.6.11.4 If the above steps do not give satisfactory results, re-extract and re-analyze the sample. Report this data if surrogate recovery is within limits. If surrogate percent recovery is out of limits for the re-extracted samples, low or high surrogate recovery is due to matrix affects and the data can be reported as estimated and the data user is notified in the form of a case narrative.

8.6.12 PCB Aroclor Qualitative Identification and Secondary GC Column Confirmation:

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.

8.6.12.1 Dual Column/Confirmatory Column Analysis by GC:

Inject samples under same operating conditions and analytical run QA/QC parameters as described in section 8.5. on a secondary GC column of dissimilar phase (e.g DB-1 and DB-5). Note: If using dual injection dual GC column system samples are injected simultaneously onto both columns. Analyze and report concentration results.

8.6.12.2 Dual Column/Confirmatory column analysis data reporting

Report concentration results to client based on project specific protocols.

Possible Protocols:

i) Report highest concentration of the two column analysis with note that analysis was confirmed (C- qualifier).

ii) Report all results from lab-designated primary GC column with note that analysis was confirmed (C -qualifier).

iii) Report both concentration results (as per US-EPA CLP program requirement)

8.6.12.3 Dual Column/Confirmatory column RPD % requirement

Calculate the relative percent difference (RPD%) of the 2 GC column results. For EPA Method 8082 protocol the RPD should be less than 40 %. If the RPD exceeds 40 % and both concentration results are greater than the practical quantitation limit inspect chromatograms for possible co-eluting interference. Re-inject samples if injection problem is indicated. Report data with flag (P-qualifier) and case narrative describing the RPD excursion.

9.0 References

9.1 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, December 1996.

9.2 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants", July 1988.

9.3 Standard Methods for the Examination of Water and Waste Water, 18th Edition 1992, American Public Health Association, American Water Works Association, Water Pollution Control Federation.

9.4 New York State Department of Health, "Environmental Laboratory Approval Program Certification Manual", Wadsworth Center for laboratories and Research, 1988, updated 1998.

9.5 "Guide to Environmental Analytical Methods", fourth edition, Genium Publishing Corporation, 1998.

10.0 Attachments

10.1 Attachment A: PCB Standards Preparation Tables

10.2 Attachment B: PCB Continuing Calibration Tables

10.3 Attachment C: GC Operating Procedures

10.4 Attachment D: Chromatograms of PCB standards.

10.5 Attachment E: Response Factor Calculation.

10.6 Attachment F: Retention Time Windows

11.0 Glossary

Accuracy: Accuracy means the nearness of a result or the mean (±) of a set of results to the true value. Accuracy is assessed by analysis of reference samples and percent recoveries.

Analytical Batch: The basic unit for analytical quality control is the analytical batch. The analytical batch is defined as samples which are analyzed together whereas the sample method sequence, the reagent lots, and manipulations are common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition (e.g. ground water, sludge, ash, etc.).

Aroclor: Polychlorinated biphenyls (PCBs) were commercially produced for a variety of uses

including, transformers, capacitors, inks, paints, dedusting agents, and pesticides to list several. Monsanto Corporation was a major producer and sold PCBs under the trade name Aroclor.

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 so 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 in 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.

Calibration Check Standard: Standard used to determine the state of calibration of an instrument between periodic recalibration. A calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards.

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.)

Laboratory Control Spike: A blank which has been spiked with the analyte(s) from an independent source in order to monitor the execution of the analytical method is called a check sample. The level of the spike shall be at the regulatory action level when applicable. Otherwise, the spike shall be at 5 times the estimate of the quantification limit. The matrix used shall be phase matched with the samples and well characterized: for example, reagent grade water is appropriate for an aqueous sample.

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.

Environmental Sample: An environmental sample or field sample is a representative sample of any material (aqueous, nonaqueous, or multimedia) collected from any source for which determination of composition or contamination as requested or required. Environmental samples are normally classified as follows:

Drinking Water--delivered (treated or untreated) water designated as potable water;
Water/Wastewater--raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluent;
Sludge--municipal sludges and industrial sludges;
Waste--aqueous and nonaqueous liquid wastes, chemical solids, contaminated soils, and industrial liquid and solid wastes.

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.

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.

Matrix: The predominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

Matrix Spike: Aliquot of a 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.

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.

Method Blank: An analytical control consisting of all reagents, internal standards and surrogate standards, which is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

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. The thrust of the law is to have those who make, distribute, and use hazardous materials responsible for effective communication. See the Hazard Communication Rule, 29 CFR, Part 1910, 1200, as amended, Sec. g. See Schedule I, Sec. 12, of the Canadian Hazardous Products Act.

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.

Precision: Precision is 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.

Quality Control: Set of measures within a sample analysis methodology to assure that the

process is in control.

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.

Stock Solution: Standard solution which can be diluted to derive other standards.

Surrogate: Surrogates are 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 samples) and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Surrogate Standard: A pure compound added to a sample in the laboratory just before processing sc that the overall efficiency of
a method can be determined.

ATTACHMENT A

Table 1
PCB Stock Standard Preparation Table

PCB Formulation	Supplier	Catalog #	Std. weight(mg)	Conc.(PPM)
A1016	GE Archive	NA	93.2	932.0
A1221	GE Archive	NA	106.8	1068.0
A1232	GE Archive	NA	103.3	1033.0
A1242	GE Archive	NA	99.0	990.0
A1248	GE Archive	NA	101.9	1019.0
A1254	GE Archive	NA	99.6	996.0
A1260	GE Archive	NA	99.2	992.0
DCBP	Chem Service	F2170	10	100.0

Unless otherwise noted hexane is the solution used to make all dilutions.

Table 2
PCB Working Standard Preparation Table

PCB Stock Standards	Init. Volume(mL)	Final Volume(mL)	Conc.(PPM)
A1016	1.0	100	9.32
A1221	1.0	100	10.68
A1232	1.0	100	10.33
A1242	1.0	100	9.90
A1248	1.0	100	10.19
A1254	1.0	100	9.96
A1260	1.0	100	9.92

Table 3
PCB Calibration Standard Preparation Table

Init. Volume(mL)	Final Volume(mL)	Final Concentration (PPM)					
		A1016	A1221	A1232	A1242	A1248	A1260
5.0	50	0.932	1.068	1.033	0.990	1.019	0.992
2.5	50	0.466	0.534	0.5165	0.495	0.5095	0.496
1.25	50	0.233	0.267	0.25825	0.2475	0.2548	0.248
1.00	50	0.1864	0.2136	0.2066	0.198	0.2038	0.1984
0.500	50	0.0932	0.1068	0.1033	0.0990	0.1019	0.0992
5.0*	50	0.01864	0.02136	0.02066	0.0198	0.02038	0.01984

*This initial volume is of the nominal 0.200 ppm standard. All others are from the nominal 10 ppm standard.

Table 4
PCB A1254 Calibration Standard Preparation Table

Init. Volume(mL) A1254	Init. Volume(mL) Surrogate	Final Volume(mL)	Final Concentration(PPM)		
			A1254	TCMX	DCBP
5.0	0	50	0.996	0	0
10.0	4.0	100	0.996	0.020	0.200
25.0*	0	50	0.498	0.010	0.100
1.25	0.800	50	0.249	0.008	0.080
0.500	0.500	50	0.0996	0.005	0.050
0.100**	0.200	50	0.01992	0.002	0.020

*This initial volume is of the A1254 0.996 ppm solution with surrogates.
**This initial volume is of the A1254 9.96 ppm solution without surrogates.
All others are from the A1254 9.96ppm working standard.

ATTACHMENT B

Table 1
PCB Continuing Calibration Stock Standards

PCB	Supplier	Catalog #	Conc. (ug/mL)
A1016	Chem Service	F107AS	1000
A1221	Chem Service	F108AS	1000
A1232	Chem Service	F113AS	1000
A1242	Chem Service	F109AS	1000
A1248	Chem Service	F110AS	1000
A1254	Chem Service	F111AS	1000
A1260	Chem Service	F112BS	1000

Table 2
PCB Continuing Calibration Working Standards

PCB	Initial Volume(mL)	Final Volume(mL)	Concentration(PPM)
A1016	1.0	100	10
A1221	1.0	100	10
A1232	1.0	100	10
A1242	1.0	100	10
A1248	1.0	100	10
A1254	1.0	100	10
A1260	1.0	100	10

Table 3
PCB Continuing Calibration Standards

PCB	Initial Volume(mL)	Final Volume(mL)	Concentration (PPM)
A1016	2.5	50	0.500
A1221	2.5	50	0.500
A1232	2.5	50	0.500
A1242	2.5	50	0.500
A1248	2.5	50	0.500
A1254	2.5	50	0.500
A1260	2.5	50	0.500

ATTACHMENT C

Gas Chromatograph Operating Procedures

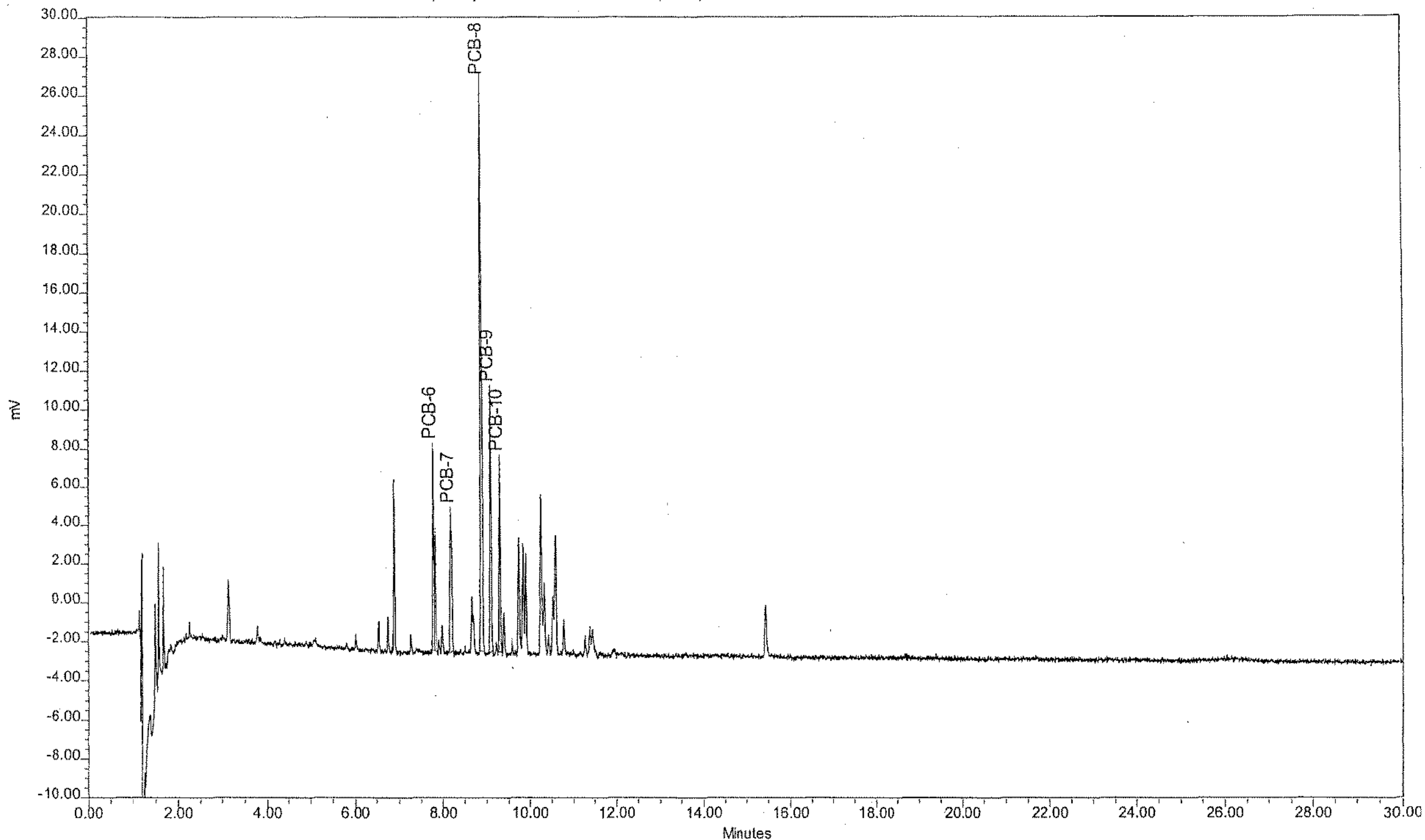
Column Type	Capillary	Capillary
Column ID	DB5-MS	DB-1
Vendor	J&W	J&W
Part Number	122-5532	122-1032
Column Length(m)	30	30
ID(mm)	0.25	0.25
Film Thick.(um)	0.25	0.25
1)Initial Col. Temp. (^{o}C)	140	140
1)Col. Hold Time (min.)	1.0	1.0
1)Col. Temp. Rate (^{o}C/min.)	10	10
1)Final Col. Temp. (^{o}C)	200	200
1)Col. Hold Time (min.)	NA	NA
2)Col. Temp. Rate (^{o}C/min.)	5	5
2)Final Col. Temp. (^{o}C)	245	245
2)Col. Hold Time (min.)	14.50	14.50
GC Col. gas flow rate (mL/min.)	17-24	17-24
ECD autozero	Yes	Yes
Detector Temp.(^{o}C)	300	300
Init. Injector Temp. (^{o}C)	300	300
A/S Vial Needle Depth	85	85
A/S Solvent Select	3	3
A/S Upper Air Gap	Yes	Yes
A/S Lower Air Gap	Yes	Yes
A/S Viscosity Factor	4	1
A/S Hot Needle Time (min.)	0.05	0.05

Autosampler(A/S) Model Number	8100	8100
A/S Injection Volume (uL)	1.3	1.3
A/S Injection Time (min.)	0.01	0.01
A/S Injection Rate (uL/sec.)	Fast 4.0	Fast 4.0
A/S Solvent Inj. plug size (uL)	0.2	0.2

ATTACHMENT D
DB5-MS Chromatograms

Chromatogram Report, PCB by SW846 Method 8082

Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com

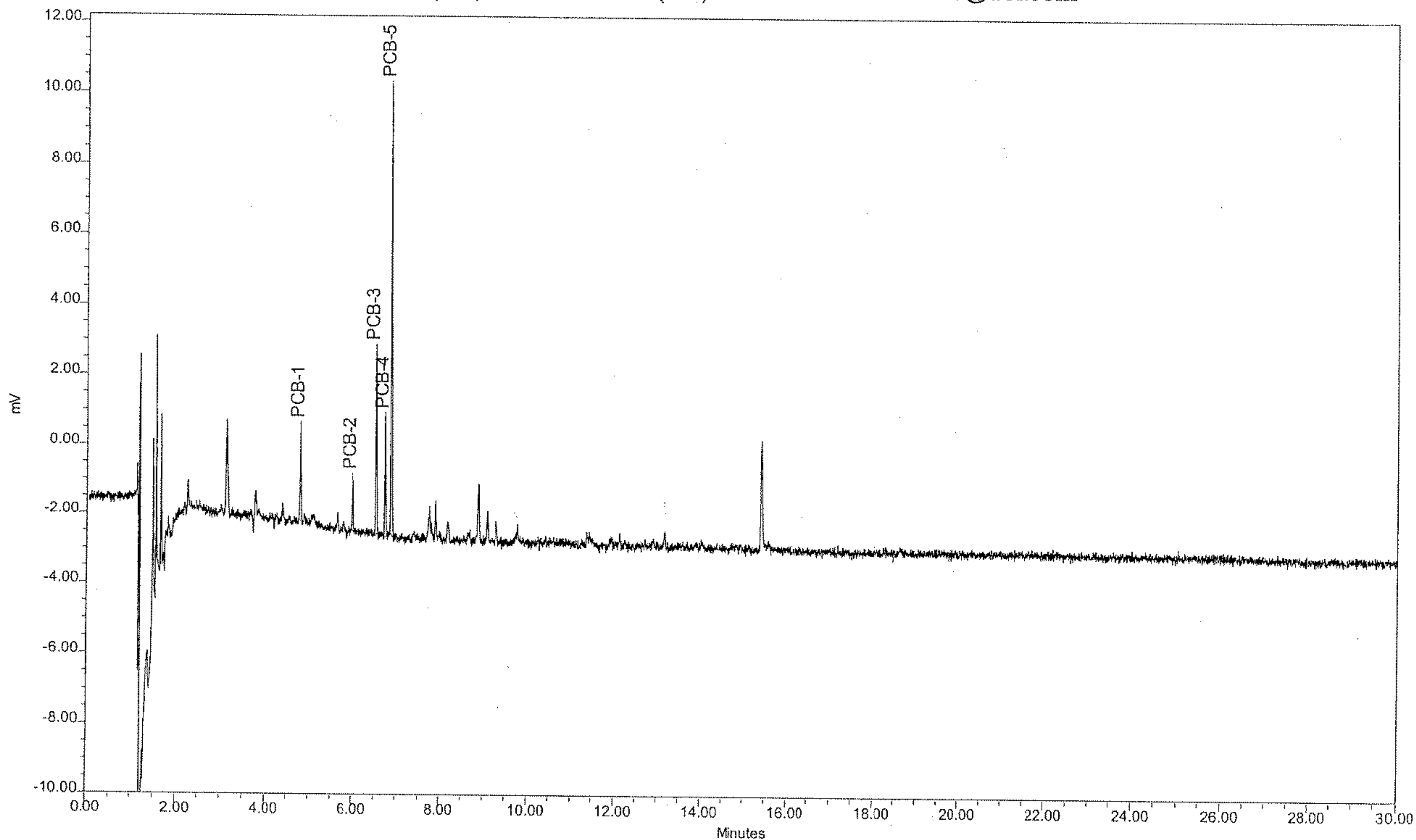


Sample Name:	CS160818	Sample Amount:	1
Sample ID:	A1016 500 PPB	Dilution:	1
Date Acquired:	08/18/1999 10:21:00	Processing Method:	GC5_8082_081399

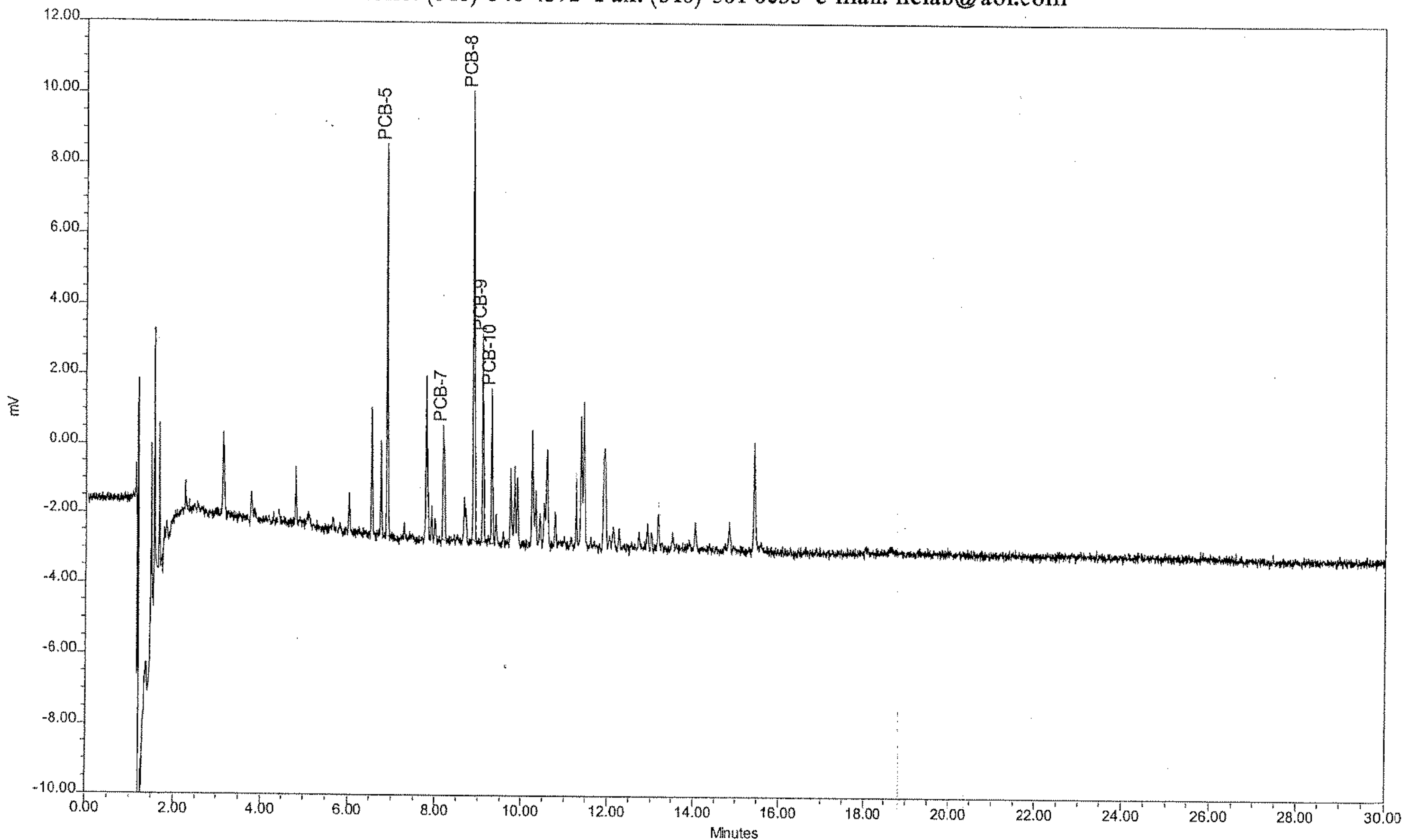
Chromatogram Report, PCB by SW846 Method 8082

Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308

Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



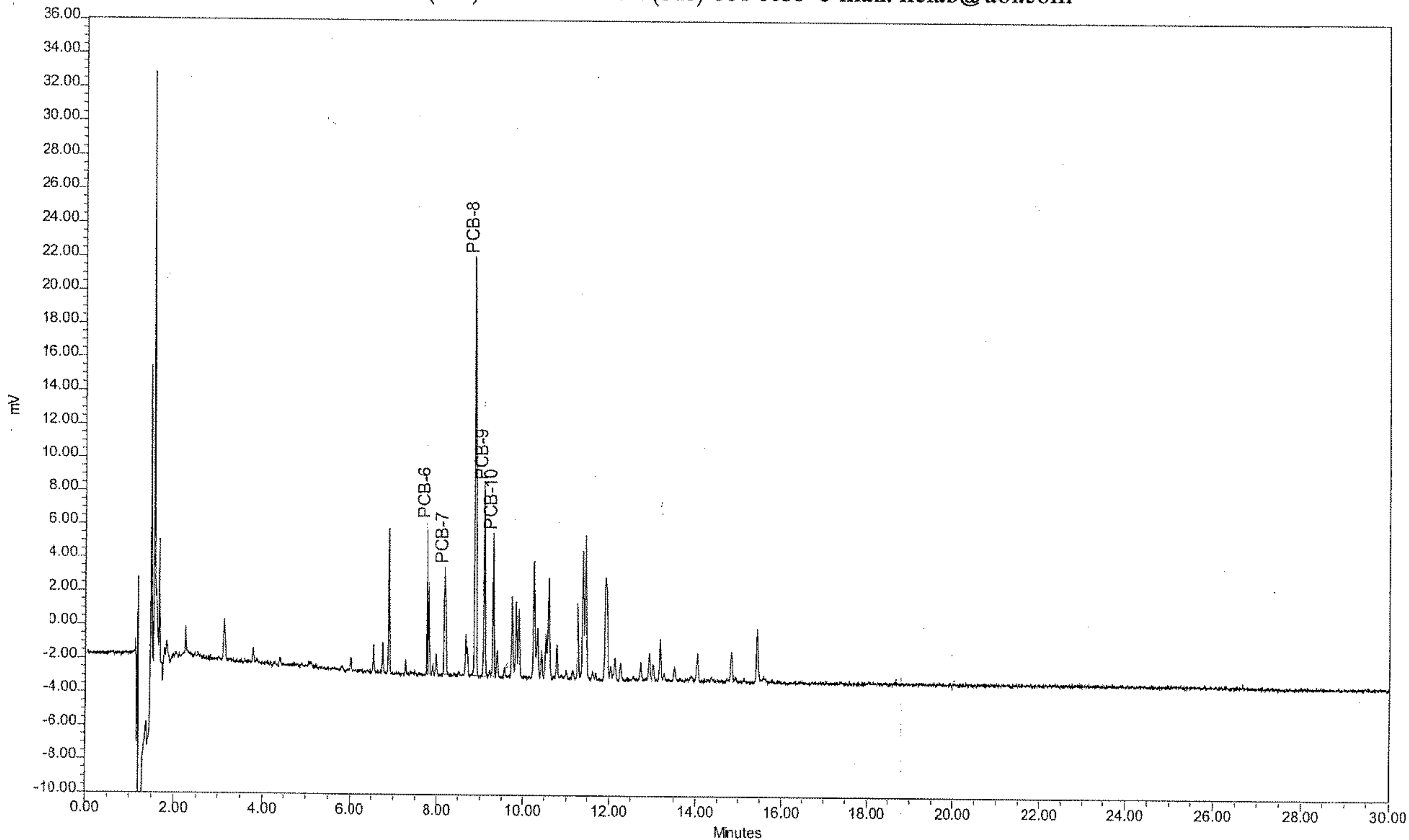
Sample Name:	CS210818	Sample Amount:	1
Sample ID:	A1221 500 PPB	Dilution:	1
Date Acquired:	08/18/1999 10:56:10	Processing Method:	GC5_8082_081399



Sample Name:	CS320818	Sample Amount:	1
Sample ID:	A1232 500 PPB	Dilution:	1
Date Acquired:	08/18/1999 11:31:22	Processing Method:	GC5_8082_081399

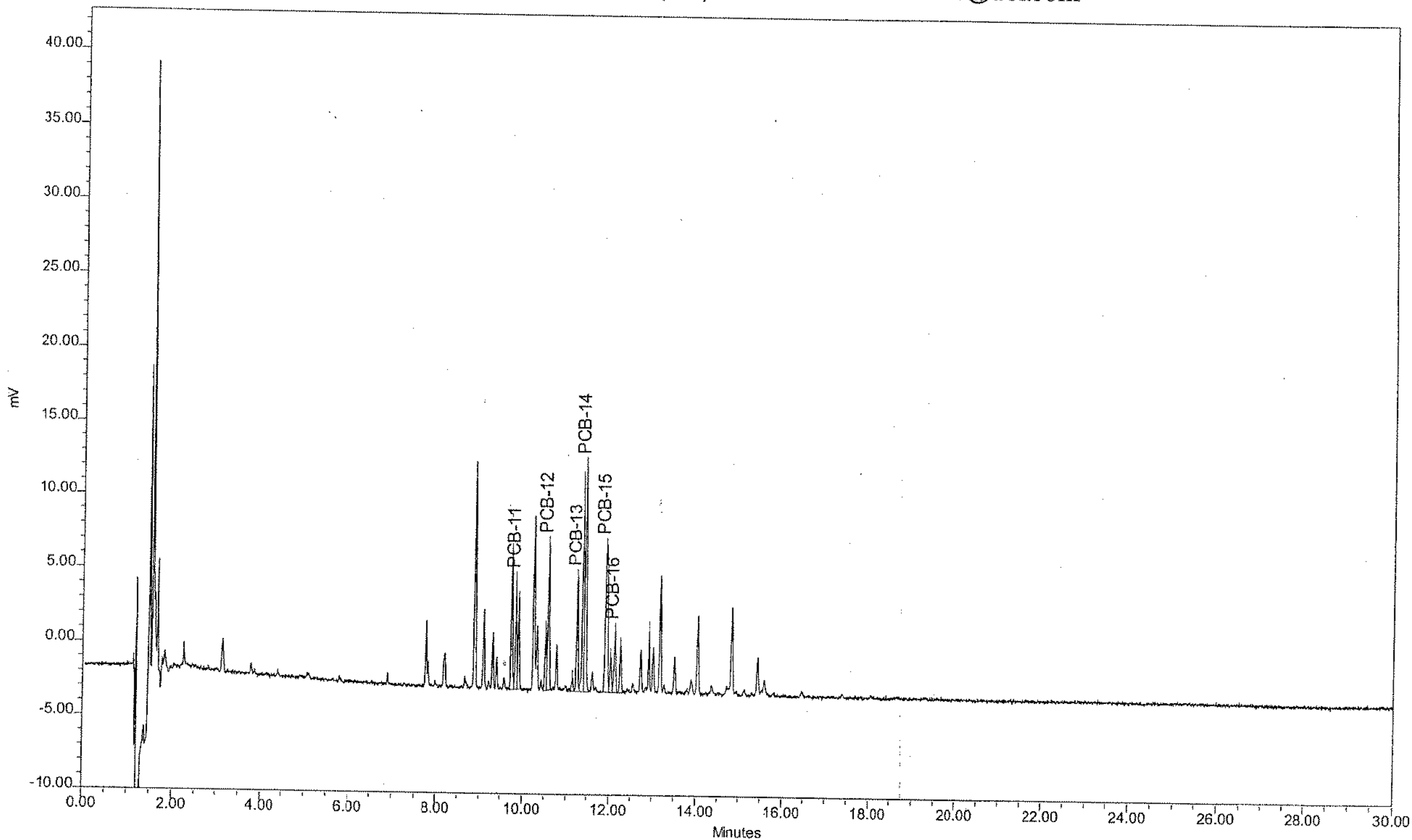
Chromatogram Report, PCB by SW846 Method 8082

Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS420818	Sample Amount:	1
Sample ID:	A1242 500 PPB	Dilution:	1
Date Acquired:	08/18/1999 12:06:31	Processing Method:	GC5_8082_081399

Chromatogram Report, PCB by SW846 Method 8082
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com

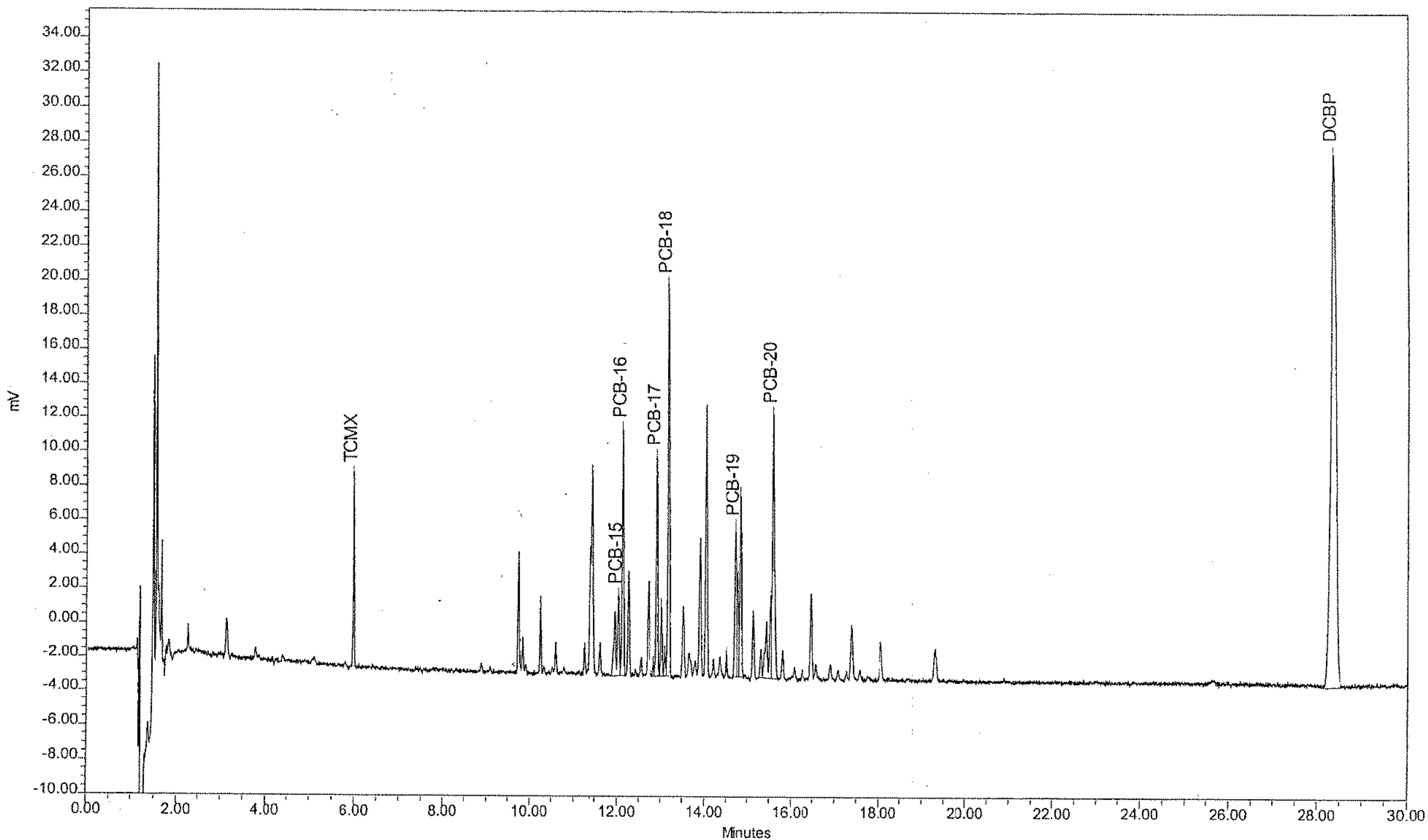


Sample Name: CS480818
Sample ID: A1248 500 PPB
Date Acquired: 08/18/1999 12:41:43

Sample Amount: 1
Dilution: 1
Processing Method: GC5_8082_081399

Chromatogram Report, PCB by SW846 Method 8082

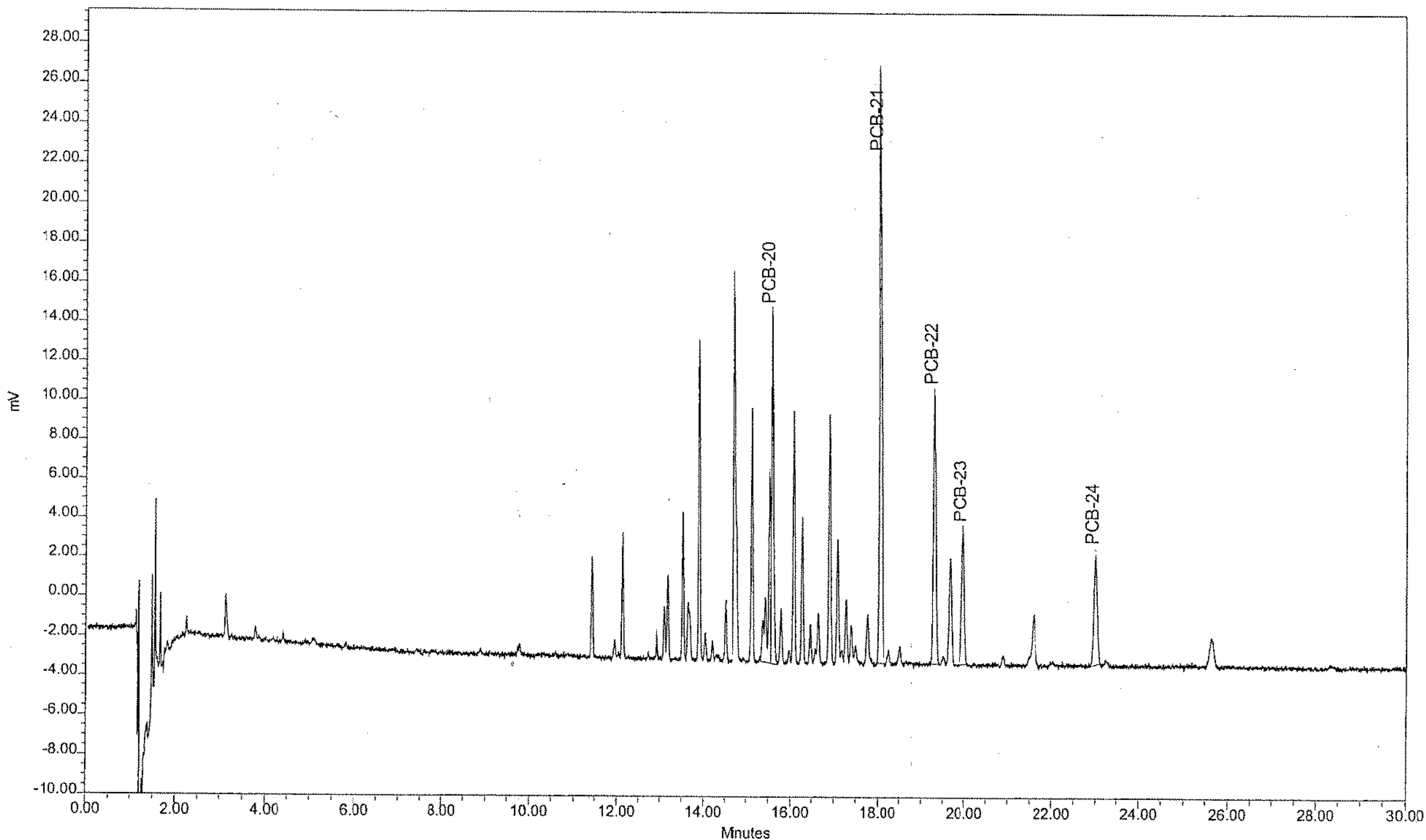
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS540818	Sample Amount:	1
Sample ID:	A1254 500 PPB	Dilution:	1
Date Acquired:	08/18/1999 1:16:54	Processing Method:	GC5_8082_081399

Chromatogram Report, PCB by SW846 Method 8082

Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com

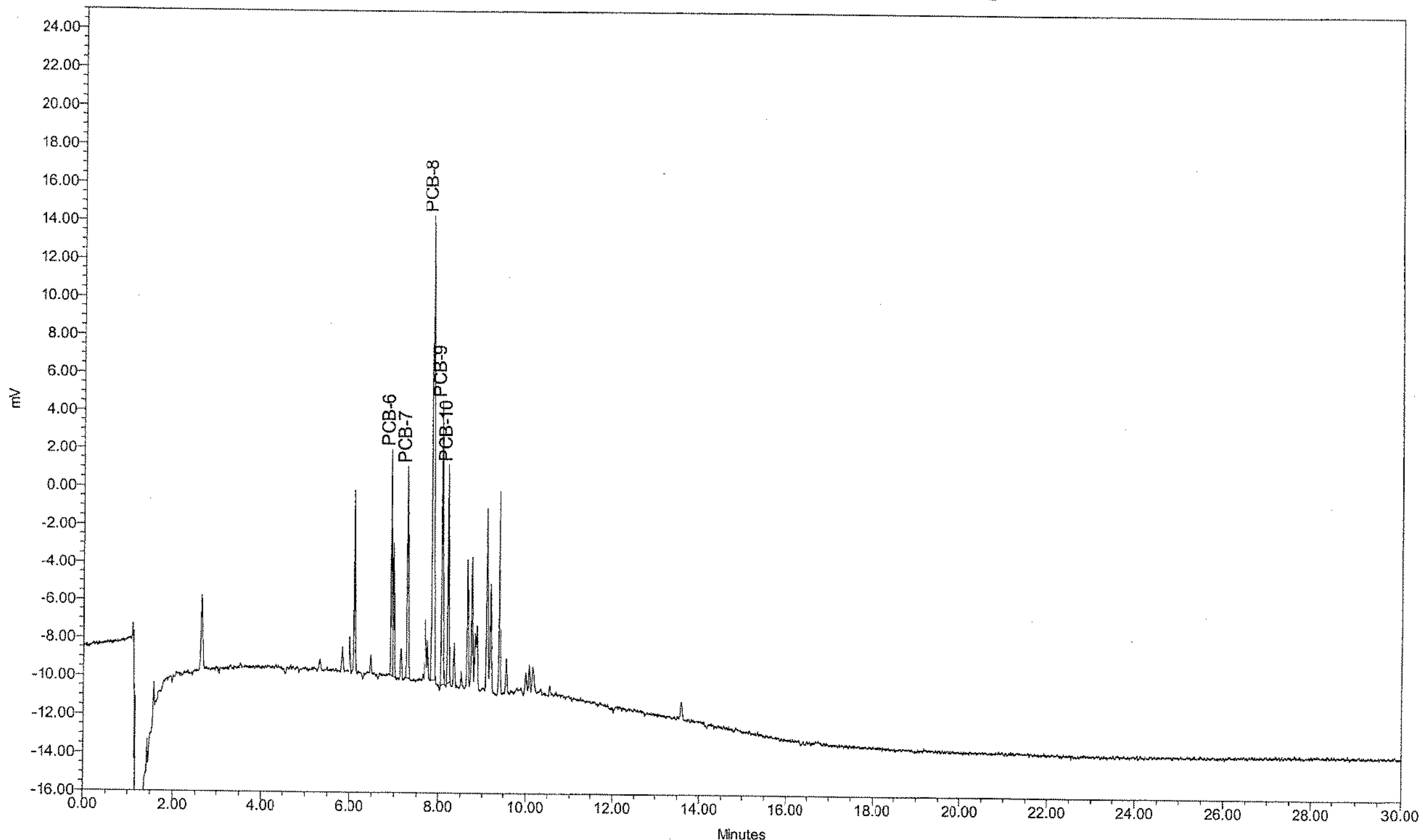


Sample Name:	CS600818	Sample Amount:	1
Sample ID:	A1260 500 PPB	Dilution:	1
Date Acquired:	08/18/1999 1:52:08	Processing Method:	GC5_8082_081399

ATTACHMENT D
DB-1 Chromatograms

Chromatogram Report, PCB by SW846 Method 8082

Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com

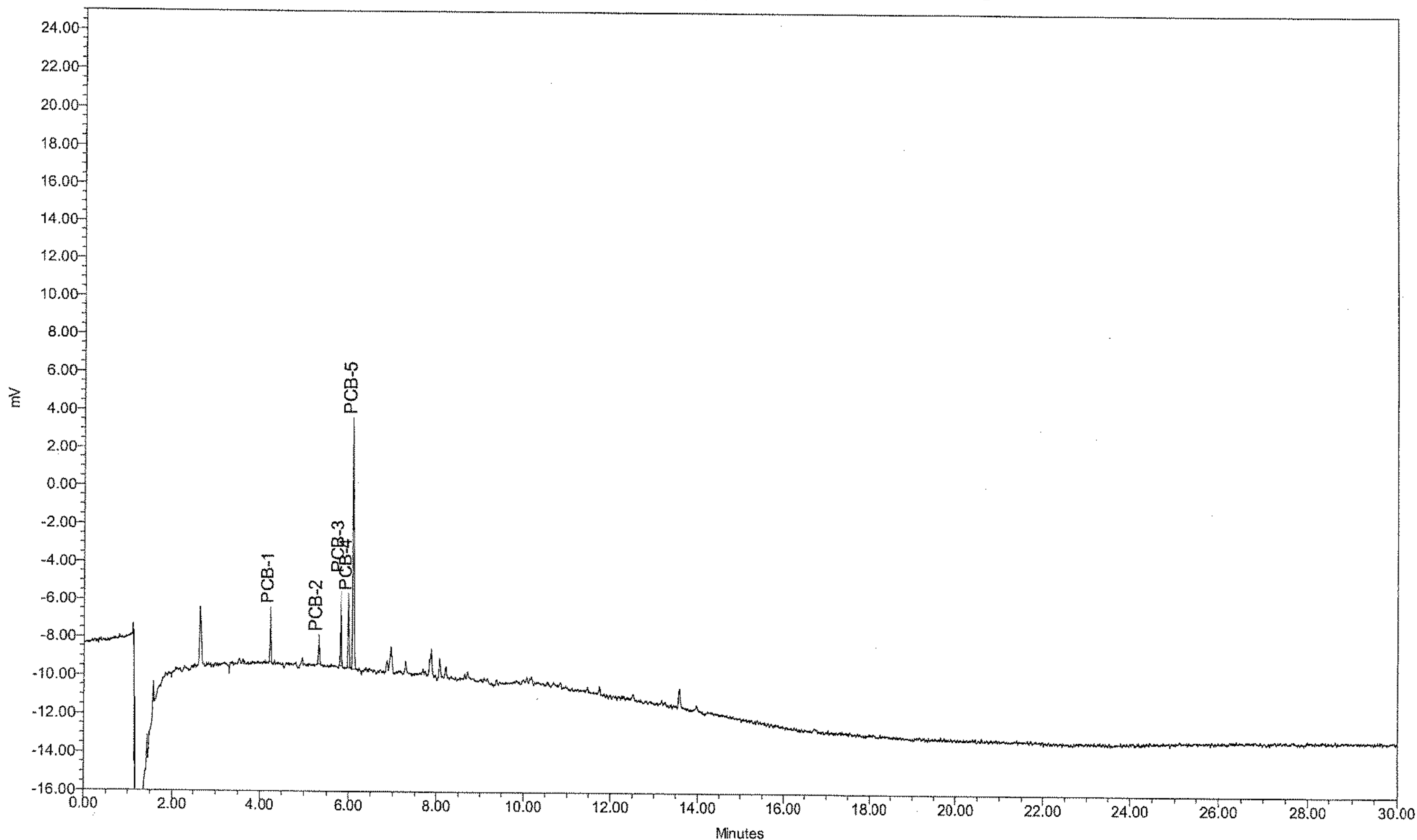


Sample Name:	CS160725	Sample Amount:	1
Sample ID:	A1016 500 PPB	Dilution:	1
Date Acquired:	07/26/1999 09:32:16	Processing Method:	GC7_8082_060899

Chromatogram Report, PCB by SW846 Method 8082

Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308

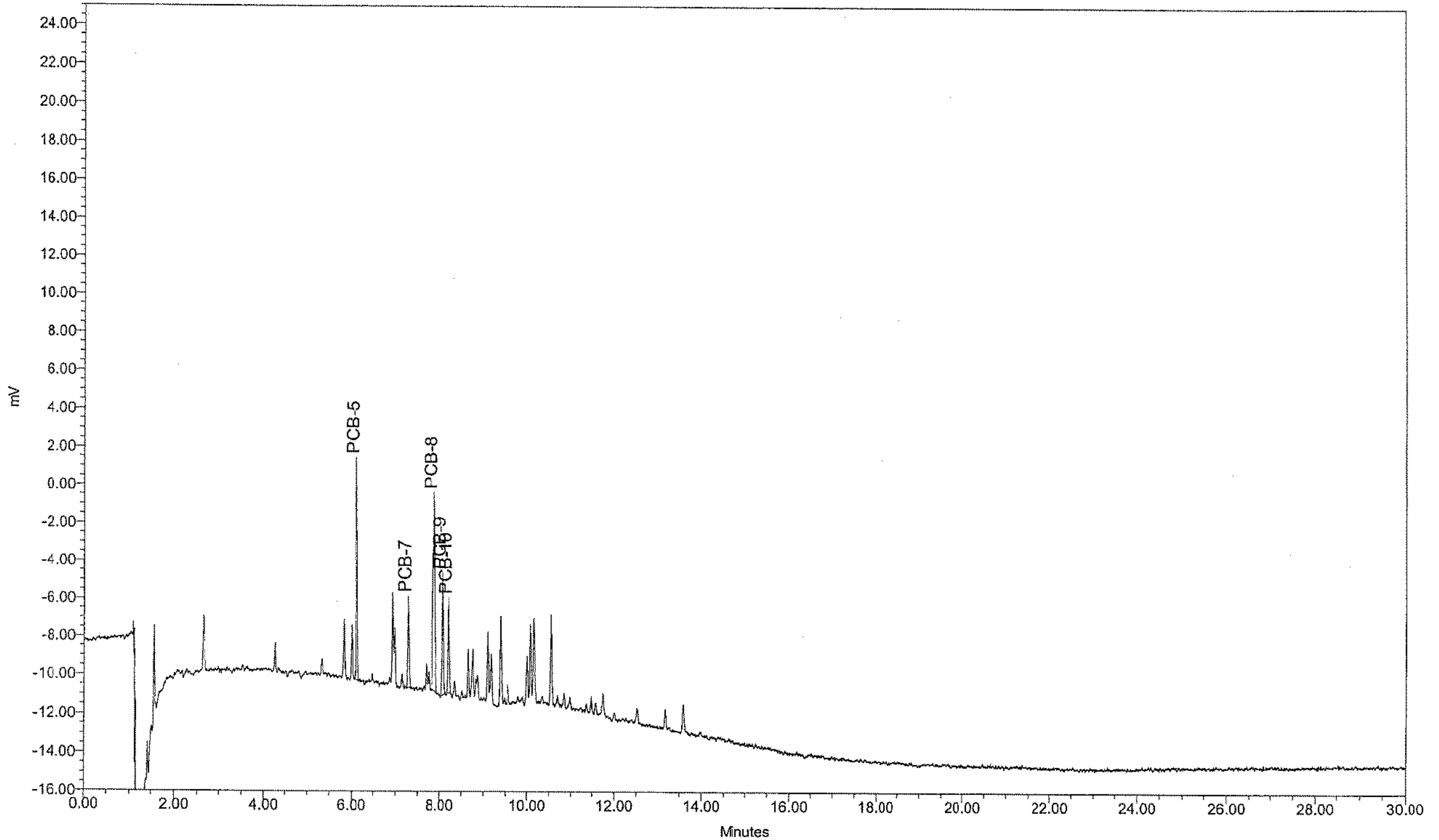
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS210725	Sample Amount:	1
Sample ID:	A1221 500 PPB	Dilution:	1
Date Acquired:	07/26/1999 10:08:26	Processing Method:	GC7_8082_060899

Chromatogram Report, PCB by SW846 Method 8082

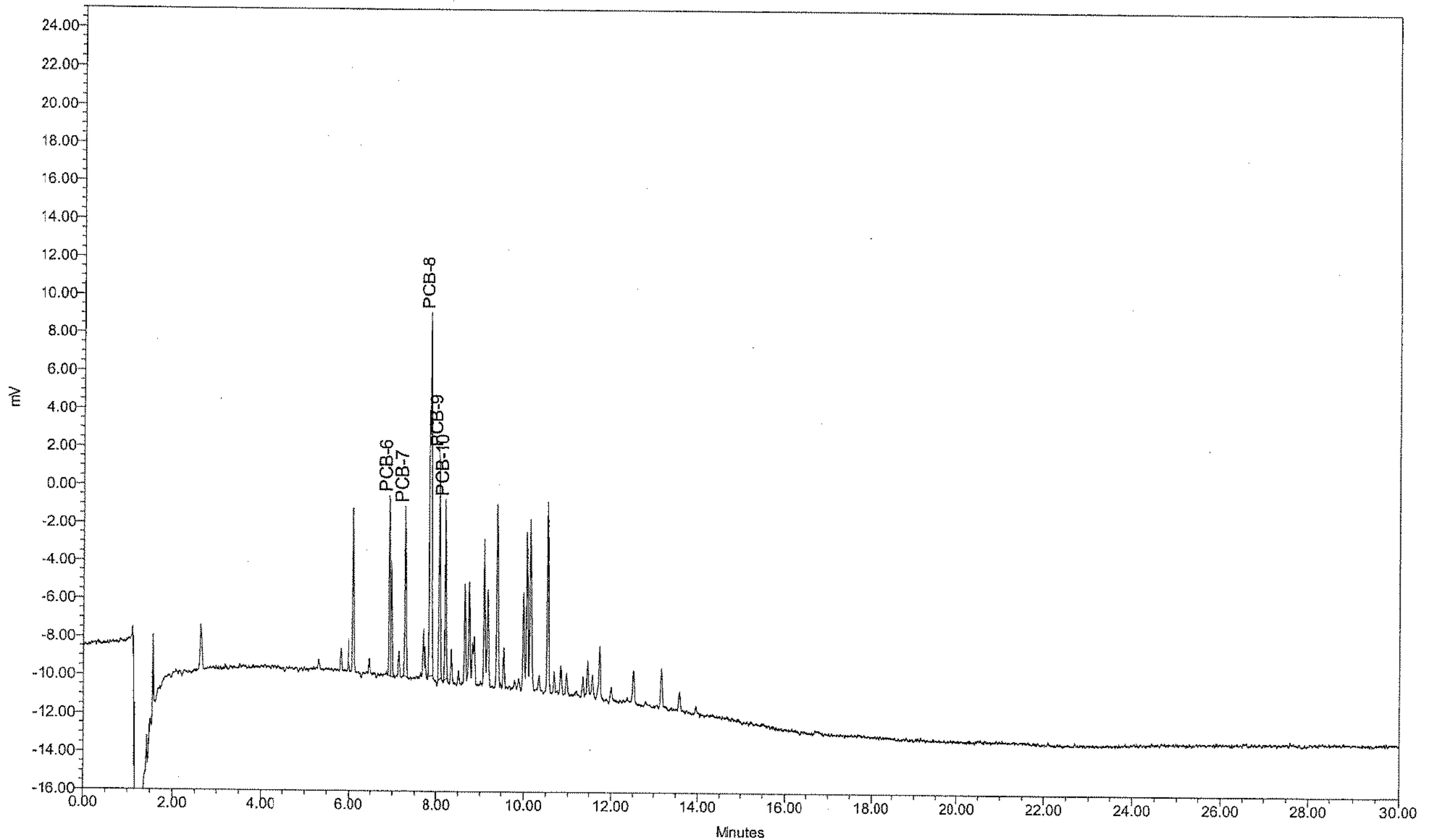
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS320725	Sample Amount:	1
Sample ID:	A1232 500 PPB	Dilution:	1
Date Acquired:	07/26/1999 11:09:59	Processing Method:	GC7_8082_060899

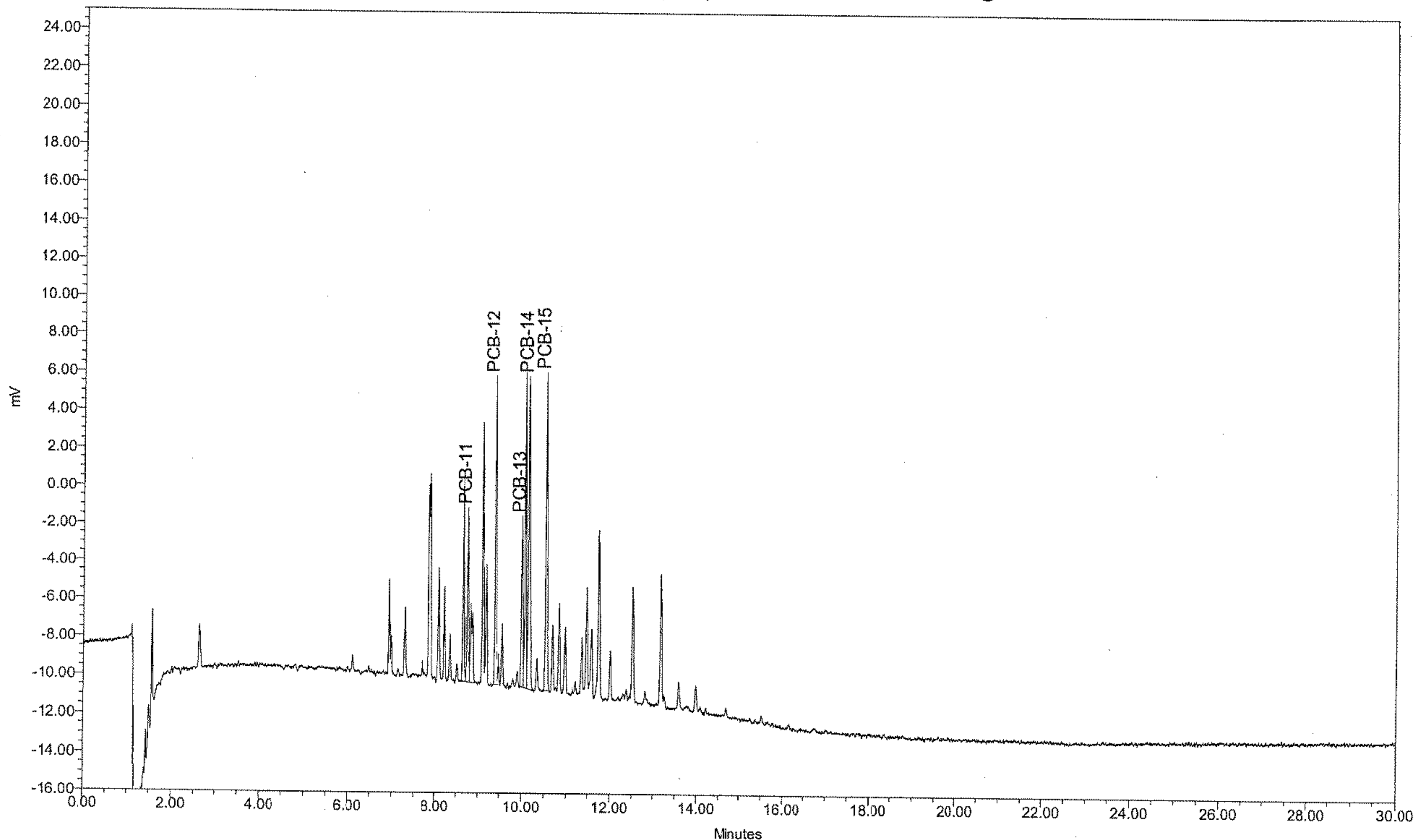
Chromatogram Report, PCB by SW846 Method 8082

Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS420725	Sample Amount:	1
Sample ID:	A1242 500 PPB	Dilution:	1
Date Acquired:	07/26/1999 11:46:07	Processing Method:	GC7_8082_060899

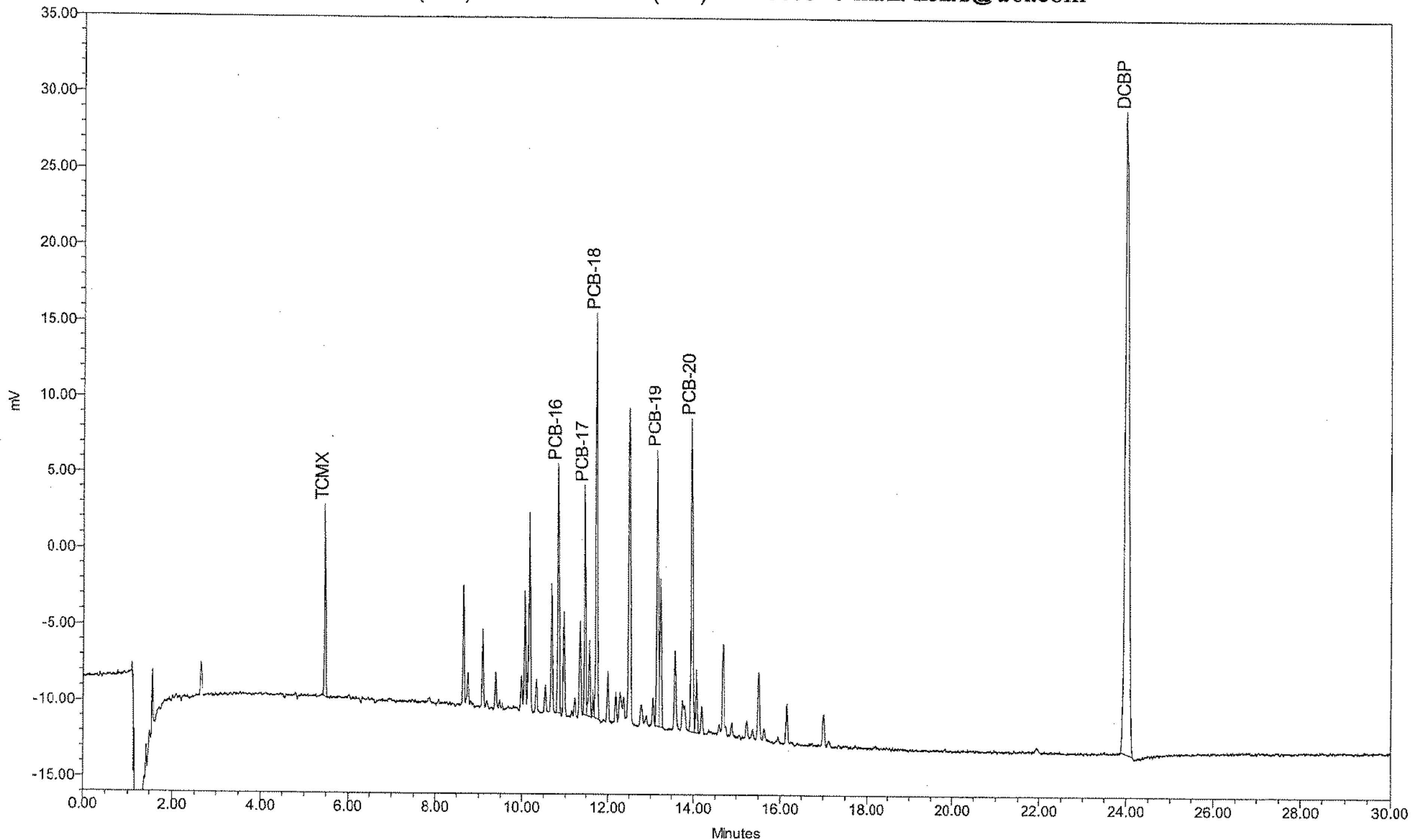
Chromatogram Report, PCB by SW846 Method 8082
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS480725	Sample Amount:	1
Sample ID:	A1248 500 PPB	Dilution:	1
Date Acquired:	07/26/1999 12:22:14	Processing Method:	GC7_8082_060899

Chromatogram Report, PCB by SW846 Method 8082

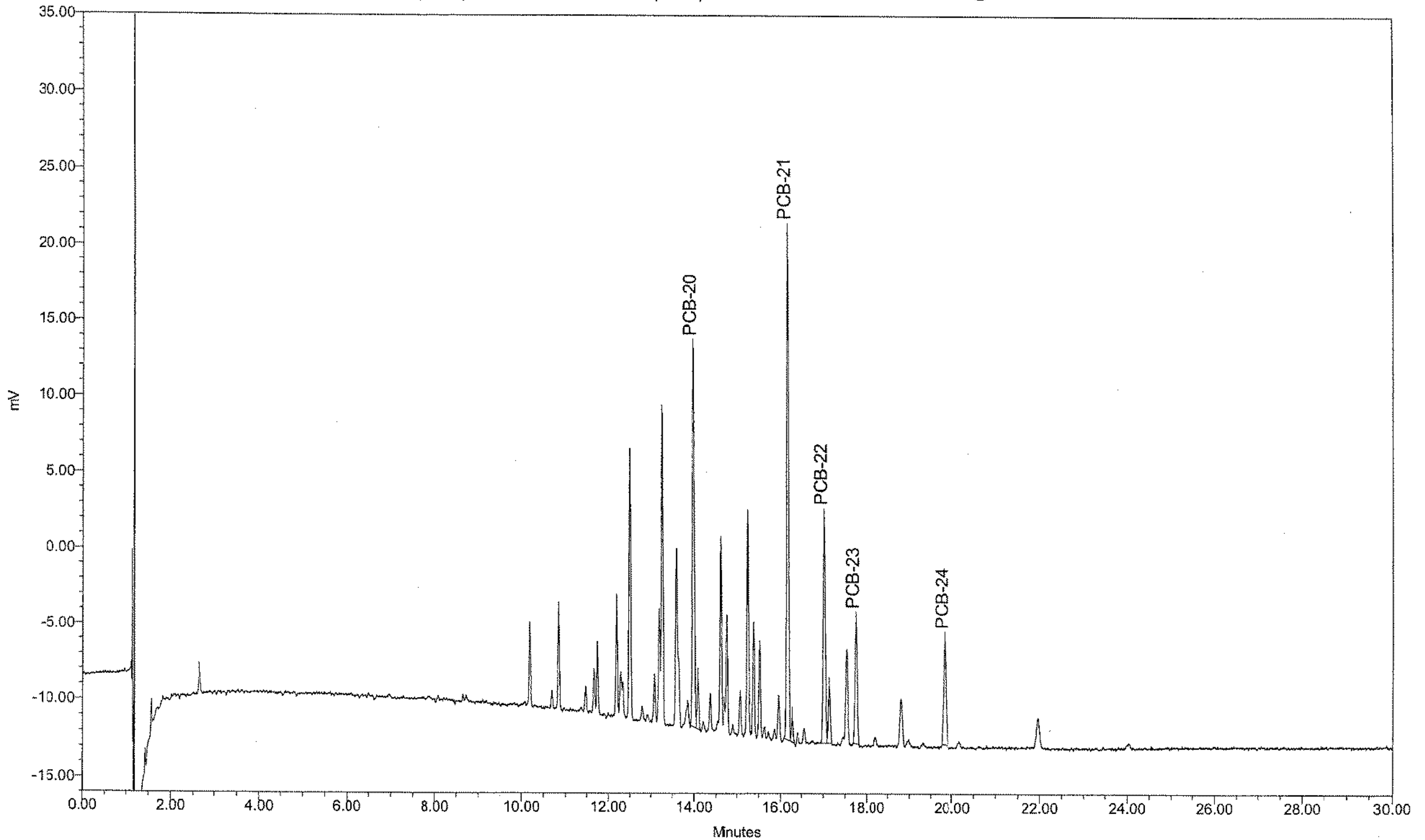
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name: CS540725
Sample ID: A1254 500 PPB
Date Acquired: 07/26/1999 12:58:21

Sample Amount: 1
Dilution: 1
Processing Method: GC7_8082_060899

Chromatogram Report, PCB by SW846 Method 8082
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com



Sample Name:	CS600725	Sample Amount:	1
Sample ID:	A1260 500 PPB	Dilution:	1
Date Acquired:	07/26/1999 13:34:27	Processing Method:	GC7_8082_060899

ATTACHMENT E

Calibration Report: PCB by SW-846 Method 8082

Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com

System Name:	Instrument 05	Sample Set Date:	08/13/1999 3:35:46 PM
Sample Set Name:	GC5_8082CC_081399	Date Calibrated:	08/16/1999 2:13:22 PM
User Name:	KristenV	Processing Method:	GC5_8082_081399
Report Method Name:	8082 CalCurve Summary by RF 01		

Calibration Component Summary Table
Component Summary For RF

	Sample Name	A1254-20	A1254-16/20	A1254	A1248
1	081254A	289.400	233.718	392.161	
2	081254B	307.665	239.346	418.651	
3	081254C	309.076	237.264	412.930	
4	081254D	299.203	229.949	402.504	
5	081254E	305.003	233.686	410.308	
Mean		302.069	234.793	407.311	
Std. Dev.		8.027	3.629	10.269	
% RSD		2.7	1.5	2.5	

Calibration Component Summary Table
Component Summary For RF

	Sample Name	TCMX	PCB-15	PCB-16	PCB-17	PCB-18	DCBP
1	081254A	1743.600					2464.880
2	081254B	1905.680					2607.388
3	081254C	1838.488					2539.036
4	081254D	1852.060					2498.834
5	081254E	1873.950					2528.173
Mean		1842.756					2527.662
Std. Dev.		60.970					53.053
% RSD		3.3					2.1

Calibration Report: PCB by SW-846 Method 8082

Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055 e-mail: nelab@aol.com

System Name: Instrument 07
Sample Set Name: GC7_8082_Calibration_042
User Name: System
Report Method Name: 8082 CalCurve Summary by RF 01
Sample Set Date: 04/22/1999 14:27:01
Date Calibrated: 05/14/1999 11:19:00
Processing Method: GC7_8082_051499

Calibration Component Summary Table
Component Summary For RF

	Sample Name	A1254-16/20	A1254-20	A1260	A1260-23	A1254
1	042354A	299.563	361.997			455.381
2	042354B	273.188	342.796			450.960
3	042354C	275.873	344.896			454.235
4	042354D	272.145	342.847			452.535
5	042354E	273.574	344.877			454.616
Mean		278.868	347.483			453.545
Std. Dev.		11.648	8.179			1.782
% RSD		4.2	2.4			0.4

Calibration Component Summary Table
Component Summary For RF

	Sample Name	TCMX	PCB-17	PCB-18	PCB-19	PCB-20	DCBP
1	042354A	1954.700					2445.172
2	042354B	1969.361					2455.106
3	042354C	1991.331					2494.547
4	042354D	1933.980					2433.796
5	042354E	2000.251					2446.710
Mean		1969.925					2455.066
Std. Dev.		26.936					23.339
% RSD		1.4					1.0

ATTACHMENT F

Northeast Analytical Inc

07/06/99

Retention Time Window Study
for Aroclors (PCB) by GC/ECD
EPA Method 8082

Instrument: GC 5
Column: DB5-MS 30Meter

Analyte	PEAK	Standard 1 500 PPB R.T. Min CS__0426	Standard 2 500 PPB R.T. Min CS_0503	Standard 3 500 PPB R.T. Min CS_0511	STD. DEV Min	%RSD	Window +/- Min.
Aroclor 1016	1	7.798	7.797	7.798	0.0006	0.01	0.002
	2	8.216	8.215	8.217	0.0010	0.01	0.003
	3	8.918	8.916	8.919	0.0015	0.02	0.005
	4	9.122	9.120	9.123	0.0015	0.02	0.005
	5	9.325	9.323	9.326	0.0015	0.02	0.005
Aroclor 1221	1	4.838	4.841	4.844	0.0030	0.06	0.009
	2	6.039	6.043	6.044	0.0026	0.04	0.008
	3	6.569	6.572	6.574	0.0025	0.04	0.008
	4	6.776	6.779	6.781	0.0025	0.04	0.008
	5	6.916	6.920	6.922	0.0031	0.04	0.009
Aroclor 1232	1	6.917	6.920	6.922	0.0025	0.04	0.008
	2	8.216	8.222	8.220	0.0031	0.04	0.009
	3	8.919	8.923	8.924	0.0026	0.03	0.008
	4	9.123	9.127	9.129	0.0031	0.03	0.009
	5	9.326	9.329	9.332	0.0030	0.03	0.009
Aroclor 1242	1	7.800	7.803	7.803	0.0017	0.02	0.005
	2	8.216	8.221	8.220	0.0026	0.03	0.008
	3	8.920	8.924	8.925	0.0026	0.03	0.008
	4	9.124	9.128	9.128	0.0023	0.03	0.007
	5	9.327	9.330	9.331	0.0021	0.02	0.006
Aroclor 1248	1	9.869	9.872	9.875	0.0030	0.03	0.009
	2	10.616	10.620	10.624	0.0040	0.04	0.012
	3	11.291	11.295	11.296	0.0026	0.02	0.008
	4	11.481	11.484	11.486	0.0025	0.02	0.008
	5	11.950	11.964	11.958	0.0070	0.06	0.021
Arolcor 1254	1	12.160	12.162	12.162	0.0012	0.01	0.003
	2	12.945	12.947	12.948	0.0015	0.01	0.005
	3	13.211	13.214	13.215	0.0021	0.02	0.006
	4	14.752	14.753	14.754	0.0010	0.01	0.003
	5	15.614	15.616	15.616	0.0012	0.01	0.003
Arolcor 1260	1	15.613	15.615	15.616	0.0015	0.01	0.005
	2	18.079	18.083	18.084	0.0026	0.01	0.008
	3	19.336	19.342	19.344	0.0042	0.02	0.012
	4	20.003	20.009	20.013	0.0050	0.03	0.015
	5	23.038	23.040	23.047	0.0047	0.02	0.014
TCMX (SURROGATE)	Surr.	6.02	6.021	6.023	0.0015	0.03	0.005
DCB (SURROGATE)	Surr.	28.389	28.40	28.41	0.0093	0.03	0.028

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Northeast Analytical Inc 04/04/02

Retention Time Window Study
for Aroclors (PCB) by GC/ECD
EPA Method 8082 (Short Method)

Instrument: GC 7
Column: DB1-30Meter

Analyte	PEAK	Standard 1 50 PPB R.T. Min	Standard 2 50 PPB R.T. Min	Standard 3 50 PPB R.T. Min	STD. DEV Min	%RSD	Window +/- Min.
Aroclor 1016		C__0401B	CS_0403	CS_0404			
	6	10.431	10.434	10.430	0.0021	0.020	0.006
	7	10.777	10.780	10.775	0.0025	0.023	0.008
	8	11.321	11.325	11.320	0.0026	0.023	0.008
	9	11.498	11.502	11.496	0.0031	0.027	0.009
	10	11.616	11.619	11.613	0.0030	0.026	0.009
Aroclor 1221		C__0401B	C__0403A	CS__0404			
	1	7.705	7.707	7.706	0.0010	0.013	0.003
	2	8.833	8.837	8.835	0.0020	0.023	0.006
	3	9.334	9.335	9.333	0.0010	0.011	0.003
	4	9.508	9.512	9.510	0.0020	0.021	0.006
	5	9.619	9.621	9.619	0.0012	0.012	0.003
Aroclor 1232		CS__0401	CS__0403	CS__0404			
	5	9.622	9.619	9.622	0.0017	0.018	0.005
	7	10.779	10.776	10.779	0.0017	0.016	0.005
	8	11.325	11.321	11.323	0.0020	0.018	0.006
	9	11.501	11.498	11.499	0.0015	0.013	0.005
	10	11.619	11.615	11.617	0.0020	0.017	0.006
Aroclor 1242		CS__0401	CS__0403	CS__0404			
	6	10.431	10.432	10.430	0.0010	0.010	0.003
	7	10.777	10.778	10.774	0.0021	0.019	0.006
	8	11.322	11.322	11.320	0.0012	0.010	0.003
	9	11.498	11.498	11.496	0.0012	0.010	0.003
	10	11.616	11.617	11.614	0.0015	0.013	0.005
Aroclor 1248		CS__0401	CS__0403	CS__0404			
	11	12.074	12.071	12.070	0.0021	0.017	0.006
	12	12.582	12.579	12.578	0.0021	0.017	0.006
	13	13.052	13.048	13.047	0.0026	0.020	0.008
	14	13.168	13.165	13.163	0.0025	0.019	0.008
	15	13.454	13.451	13.450	0.0021	0.015	0.006
Arolcor 1254		CS__0401	CS__0403	CS__0404			
	16	13.655	13.651	13.651	0.0023	0.017	0.007
	17	14.099	14.097	14.098	0.0010	0.007	0.003
	18	14.291	14.289	14.288	0.0015	0.011	0.005
	19	15.383	15.382	15.381	0.0010	0.007	0.003
	20	16.042	16.041	16.039	0.0015	0.010	0.005
Arolcor 1260		CS__0401	CS__0403	CS__0404			
	20	16.045	16.045	16.041	0.0023	0.014	0.007
	21	18.212	18.210	18.207	0.0025	0.014	0.008
	22	19.183	19.182	19.179	0.0021	0.011	0.006
	23	20.016	20.016	20.007	0.0052	0.026	0.016
	24	22.425	22.420	22.410	0.0076	0.034	0.023
TCMX (SURROGATE)	Surr.	8.988	8.986	8.987	0.0010	0.011	0.003
DCB (SURROGATE)	Surr.	27.277	27.273	27.270	0.0035	0.013	0.011

Appendix K

Radioisotope Analysis of Cesium-137 and Beryllium-7 in Sediments

Appendix K

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 Blasland, Bouck & Lee 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, Packaging, and Shipping Procedures

Appendix L

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) turn around 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 Patrick Foos at BBL by facsimile at (315) 449-2347.

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

ARCADIS
Infrastructure, environment, facilities

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:

20730826 CofC AR Form 01.12.2007

Distribution: **WHITE – Laboratory returns with results** **YELLOW – Lab copy** **PINK – Retained by BBL**

Attachment L-2
Sample Label

Project #	Date
Sample I.D.	

Sample Type	Collection Mode	Time
☐ Soil/Sediment ☐ Water	☐ Composite ☐ Grab	

Analysis

Sampler(s)	Preservative

Attachment L-3
Custody Seal Label

CUSTODY SEAL

SEALED BY

____

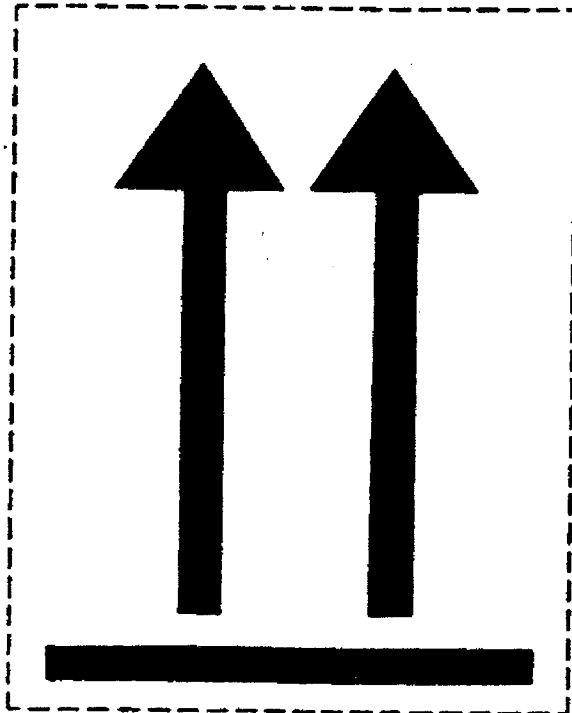
DATE __________ **TIME** __________

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

Standard Operating Procedures for Shipment of Department of Transportation Hazardous Materials

Appendix M

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

Appendix N

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, #1
Sunnyvale, CA 94086

December 1998

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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.

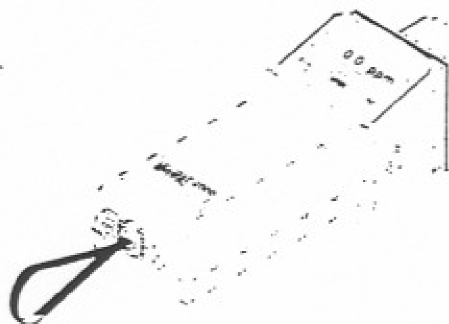


Figure 2-1 MiniRAE 2000

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:

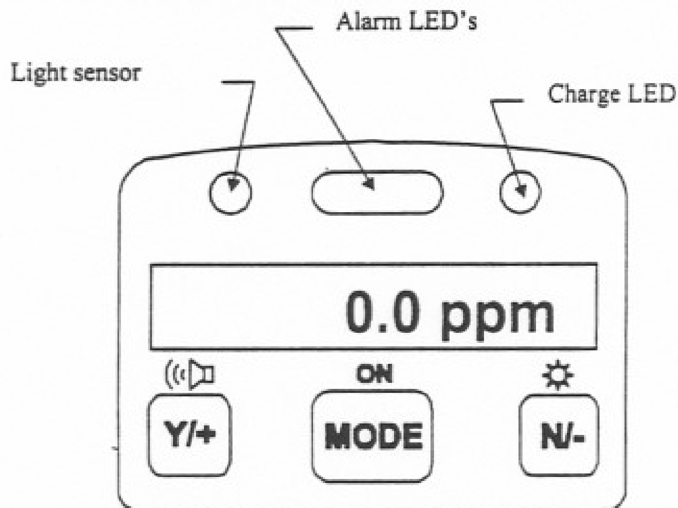


Figure 2.2 LCD display and Keypad

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

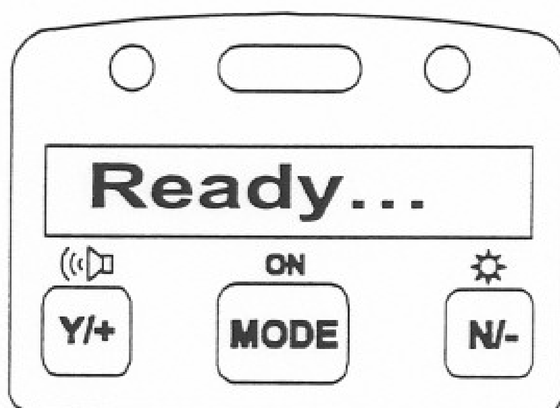
is completely drained or is disconnected from the monitor for more than 30 minutes, the real time clock will be lost. In this case, the user needs to enter the real time clock information again, as described in Section 4, or send the PC clock during configuration through the PC communication.

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.

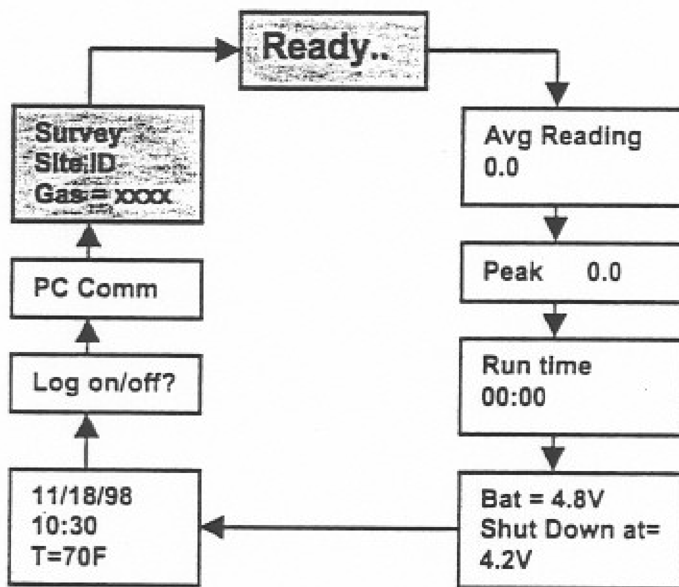


Press the **[MODE]** key to cycle through the idle operation menu. The PID sensor and pump are turned off during this idle operation.
Main operation menu displays include:

- **"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.

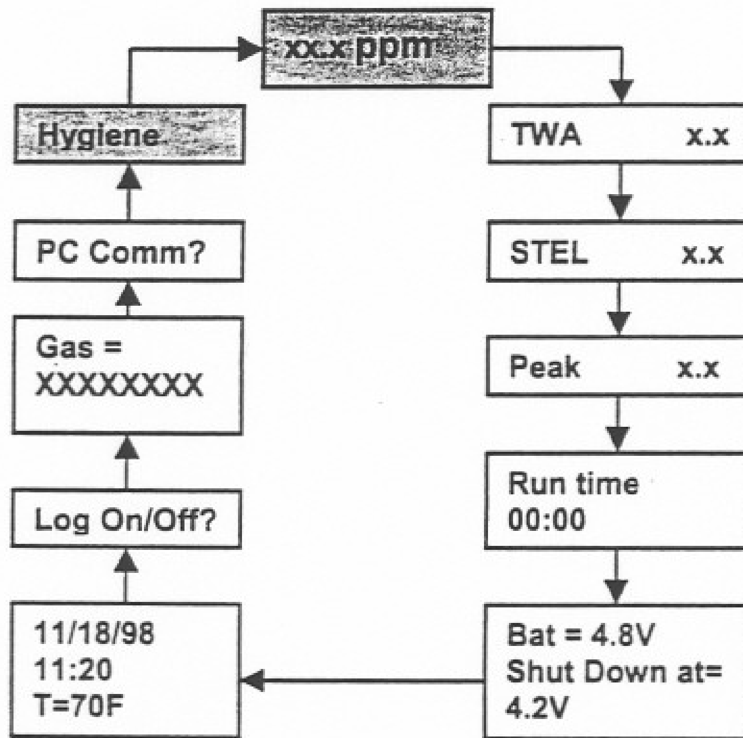
2.4.2 Hygiene Mode

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

Appendix O

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 June 3, 1998 USEPA Region I draft guidance document entitled *Draft Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductivity, oxidation/reduction potential [ORP], and turbidity)*, which is included as Attachment O-1. The EPA draft guidance document was prepared primarily to address calibration of multi-probe water quality monitoring instruments, but is also applicable to the calibration of most single-parameter monitoring instruments.

- *Temperature:* Perform annual accuracy check according to procedures outlined in 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 8. 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 8.

- *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 7. 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. Allow approximately 1 minute for readings to stabilize 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
U.S. Environmental Protection Agency Region 1 Draft Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/ specific conductance, oxidation/reduction potential [ORP], and turbidity)

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION 1

DRAFT CALIBRATION OF FIELD INSTRUMENTS
(temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity)

I. SCOPE & 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 ground water and surface water. Water quality parameters include temperature, pH, dissolved oxygen, conductivity/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 utilize multiple probes (temperature, pH, dissolved oxygen, conductivity/specific conductance, and/or oxidation/reduction potential [ORP]) and the probe readings for pH, dissolved oxygen, and specific conductance are automatically corrected for temperature. Communications to the instrument (programming and displaying the measurement values) are performed using a display/logger or a computer. Information sent to the instrument is entered through the keypad on the display/logger or computer. It is desirable that the display/logger or computer have data storage capabilities. If the instrument does not have a keypad, follow the manufacturer's instructions for entering information into the instrument.

For ground water 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 because turbidity cannot be measured in a flow-through-cell. 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.

II. GENERAL

All monitoring instruments must be calibrated before they are used to measure environmental samples. Part of the calibration is performed prior to the field event. For instrument probes that rely on the temperature sensor (pH, dissolved oxygen, conductivity/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.

Most instruments will require at least two standards to bracket the expected measurement range, that is, one standard less than the expected value and one higher. Calibration must be performed at the beginning of each sampling day prior to sample collection. To determine if the instruments have remained in calibration during transport to each sampling location, use one of the previously used standards as a check standard at the sampling site. If the check measurement does not agree with the initial calibration or to within the specifications of the instrument, then the instrument must be re-calibrated. When an environmental sample measurement falls outside the calibration range, the instrument must be re-calibrated to bracket the new range before continuing measurements.

This SOP requires that the manufacturer's instruction manual (including the instrument specifications) accompany the instrument into the field.

III. CALIBRATION PROCEDURES

Prior to calibration, all instrument probes must be cleaned according to the manufacturer's instructions. Failure to perform this step (proper maintenance) can lead to erratic measurements.

Program the multi-probe instrument so that the following parameters to be measured will be displayed: temperature, pH, percent dissolved oxygen, mg/l dissolved oxygen, conductivity, 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 additional information).

While calibrating or measuring, make sure there are no air bubbles lodged between the probe and the probe guard.

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.

Verification Procedure

1. Allow a container filled with water to come to room 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 and 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 (usually ±0.15°C). If the measurements do not agree, the instrument may not be working properly and the manufacturer needs to be consulted.

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. For ground water, the pH will usually be close to seven. 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. For those instruments that will not accept three standards, the instrument will need to be re-calibrated if the water sample's pH is outside the initial calibration range described by the two standards.

Calibration Procedure

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 distilled water, blot dry with soft tissue.

4. Select monitoring/run mode. Immerse probe into the initial standard (e.g., pH 7).

5. Stir the standard 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. Select monitoring/run mode. The readings should remain within manufacturer's specifications; if they change, re-calibrate. If readings continue to change after re-calibration, consult manufacturer.

6. Remove probe from the initial standard, rinse with distilled water, and blot dry.

7. Immerse probe into the second standard (e.g., pH 4). Repeat step 5.

8. Remove probe from the second standard, rinse with distilled water, and blot dry. 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 9) and repeat step 5.

10. Remove probe from the third standard, rinse with distilled water, and blot dry.

11. Select monitoring/run 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. Place pH probe in its storage container.

DISSOLVED OXYGEN

Dissolved oxygen (DO) content in water is measured using a membrane electrode. The DO probe's membrane and electrolyte solution should be replaced prior to the sampling period. Failure to perform this step may lead to erratic measurements.

draft

Calibration Procedure

1. Gently dry the temperature sensor according to manufacturer's instructions.

2. Place a wet sponge or a wet paper towel on the bottom of the DO calibration container.

3. Place the DO probe into the container without the probe coming in contact with the wet sponge or paper towel. The probe must fit tightly into the container to prevent the escape of moisture evaporating from the sponge or towel.

4. 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 monitoring/run mode. Check temperature readings. Readings must stabilize before continuing to the next step.

5. Select calibration mode; then select "DO %".

6. 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].

7. The instrument should indicate that the calibration is in progress. The instrument will take approximately one minute to calibrate. After calibration, the instrument should display percent saturated DO.

8. Select monitoring/run mode. 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 to the accuracy of the instrument (usually $\pm$ 0.2 mg/L), repeat calibration. If this does not work, change the membrane and electrolyte solution.

9. Remove the probe from the container and place it into a 0.0 mg/L DO standard (see note). The standard must be filled to the top of its container and the DO probe must fit tightly into the standard's container (no head space). Check temperature readings. They must stabilize before continuing.

10. Wait until the "mg/l DO" readings have stabilized. The instrument should read 0.0 mg/L or to the accuracy of the instrument (usually ± 0.2 mg/L). If the instrument cannot reach these values, it will be necessary to clean the probe, and change the membrane and electrolyte solution. If this does not work, prepare a new 0.0 mg/L DO standard. If these measures do not work, contact manufacturer.

Note: To prepare a zero mg/L DO standard 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 to water. The standard container must be completely filled (no head space). This solution is prepared prior to the sampling event. If some of the solution is lost during instrument calibration, add more water to the container so that the standard is stored with no head space.

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, but below the specific conductance of the environmental samples. A second standard which is above the environmental sample specific conductance is used to check the linearity of the instrument in the range of measurements.

Calibration Procedure

1. Allow the calibration standard to equilibrate to the ambient temperature.

2. Remove probe from its storage container, rinse the probe with a small amount of the conductivity/specific conductance standard (discard the rinsate), and place the probe into the conductivity/specific conductance standard.

3. Select monitoring/run mode. Wait until the probe temperature has stabilized.

4. Look up the conductivity value at this temperature from the conductivity versus temperature correction table usually found on the standard bottle or on the standard instruction sheet. You may need to interpolate the conductivity value between temperatures. Select calibration mode, then conductivity. Enter the temperature corrected conductivity value into the instrument.

5. Select monitoring/run 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.

6. Read the specific conductance on the instrument and compare the value to the specific conductance value on the standard. The instrument value should agree with the standard within the manufacturer's specifications. If not, re-calibrate. If the re-calibration does not correct the problem, the probe may need to be cleaned or serviced by the instrument manufacturer.

7. Remove probe from the standard, rinse the probe with a small amount of the second conductivity/specific conductance standard (discard the rinsate), and place the probe into the second conductivity/specific conductance 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, but above the first standard and repeat the verification. If values still do not compare, try cleaning the probe or consult the manufacturer.

8. When monitoring ground water or surface water, use the specific conductance readings.

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) to equilibrate to ambient temperature.

2. Remove the probe from its storage container, and place it into the standard.

3. Select monitoring/run mode.

4. While stirring the standard, wait for the probe temperature to stabilize, then read the temperature.

5. 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.

6. Select monitoring/run mode. The readings should remain unchanged within manufacturer's specifications. If they change, re-calibrate. If readings continue to change after re-calibration, consult manufacturer.

7. If the instrument instruction manual states that the instrument is factory calibrated, then verify the factory calibration against the standard. If they do not agree within the specifications of the instrument, the instrument will need to be re-calibrated by the manufacturer.

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.

Some instruments will only accept one standard. For these instruments, the 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 according to the EPA analytical Method 180.1.

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, free from fingerprints and dust. If the cuvette is scratched or dirty, discard or clean the cuvette respectively.

4. Zero the instrument by using either a zero or 0.02 NTU standard. A zero standard (approximately 0 NTU) can be prepared by passing distilled water through a 0.45 micron pore size membrane filter.

5. Using a standard in the range of 5 - 20 NTUs, calibrate according to manufacturer's instructions or verify calibration if instrument will not accept a second standard. If verifying, the instrument should read 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.

7. Using a standard between 20 and 100 NTUs, calibrate according to manufacturer's instructions or verify calibration if instrument does not accept a third standard. If verifying, the instrument should read standard value to within the specifications of the instrument. If the instrument has range of scales, check each range that will be used with the proper standard for that scale.

IV. DATA MANAGEMENT AND RECORDS MANAGEMENT

All calibration records must be documented in the project's log book. At a minimum, include the instrument manufacturer, model number, instrument identification number, standards used to calibrate the instruments (including source), calibration date, and the instrument readings.

References

Standard Methods for the Examination of Water and Wastewater, 19th edition, 1995.

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.

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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.

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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.

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Appendix P

In-situ Hydraulic Conductivity Test Procedures

Appendix P

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 Log

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	△

BLASLAND & BOUCK ENGINEERS, P.C.

Appendix Q

Water Level/Oil Thickness Measurement Procedures

Appendix Q

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

WATER LEVERL/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

Passive Oil Recovery Procedures

Appendix R

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 (Attachment R-1).

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

Appendix S

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 aboveground well completion is illustrated in Attachment S-1.

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-2.

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-4). 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-4). 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.

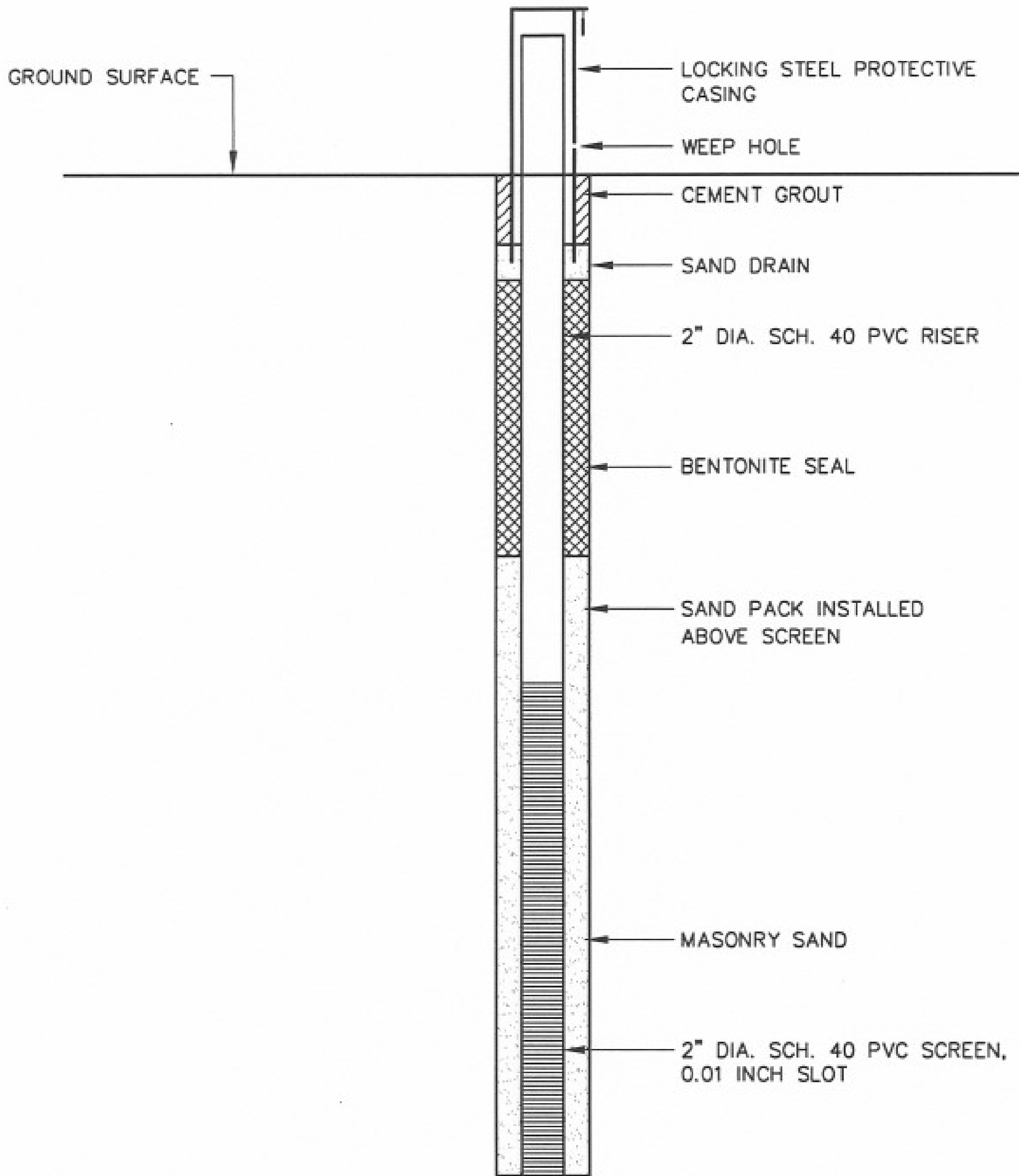
Step 13 - Secure lid on well.

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-1
Stick-Up Monitoring Well Detail



(DRAWING NOT TO SCALE)

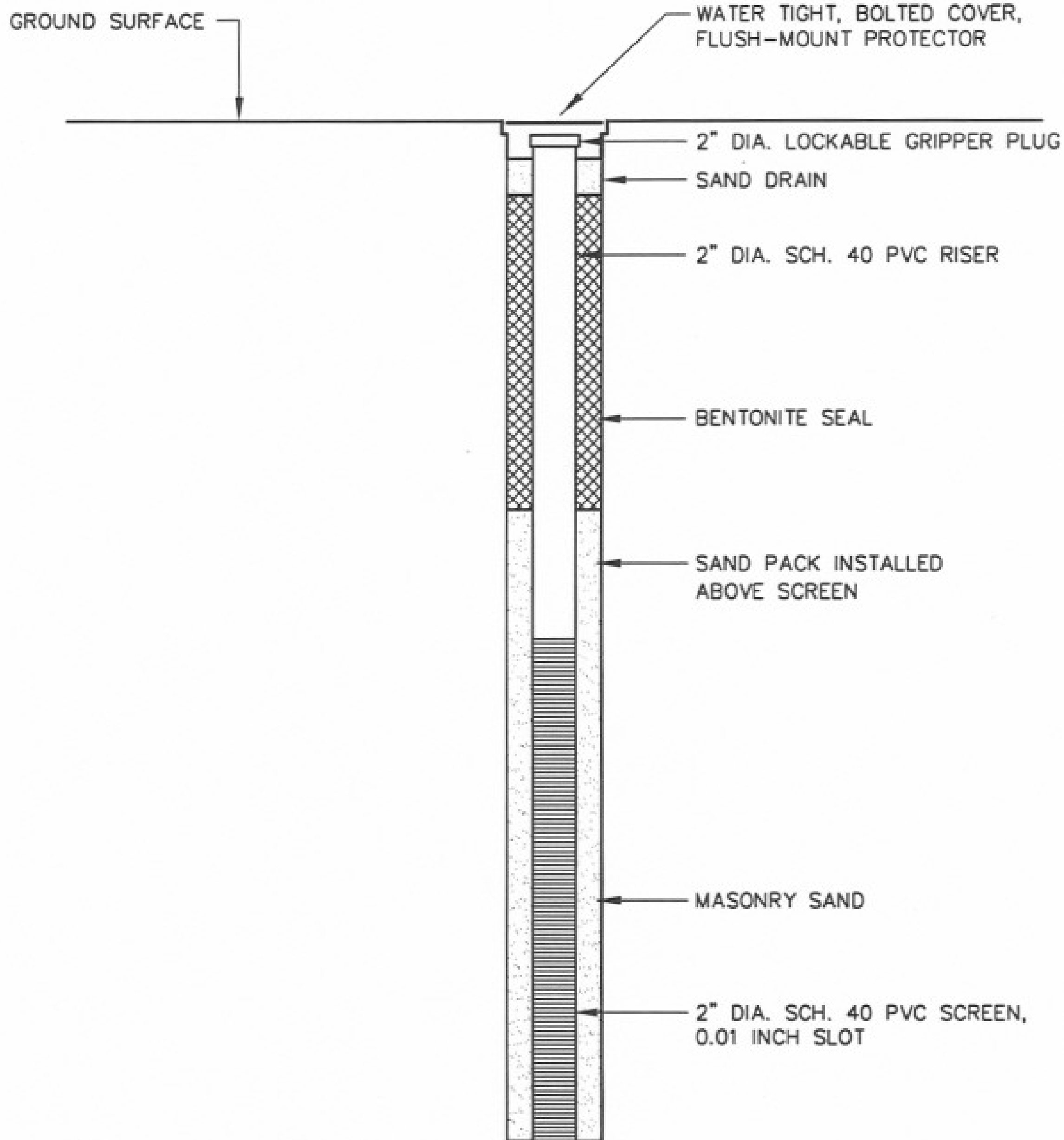
STICK-UP MONITORING WELL DETAIL

BBL BLASLAND, BOUCK & LEE, INC. *engineers & scientists*

FIGURE **S-1**

MVA.PCP
9/8/00 SYR-54-RCB
20139002/20139L02.DWG

Attachment S-2
Flush-Mount Monitoring Well Detail



(DRAWING NOT TO SCALE)

FLUSH MOUNT MONITORING WELL DETAIL

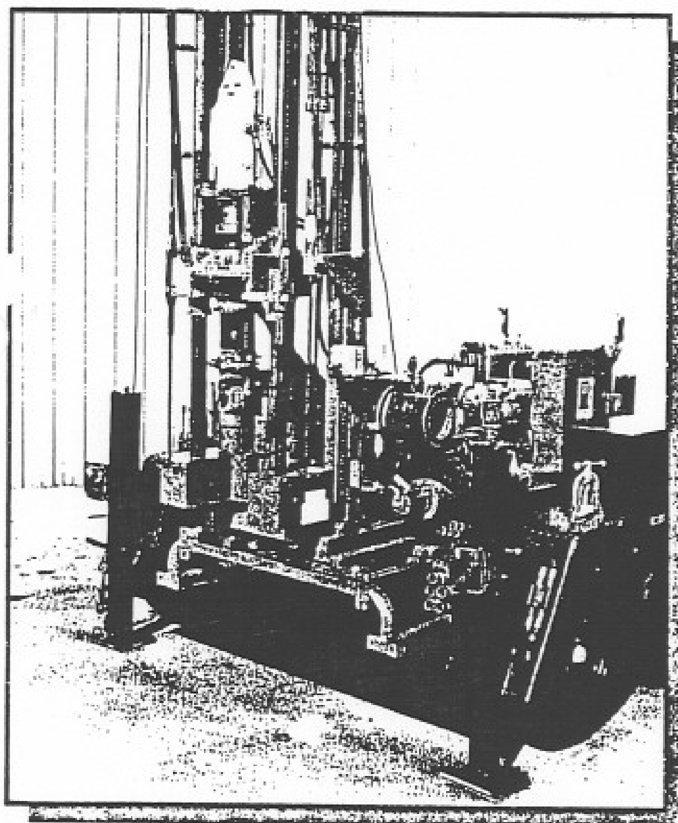
BBL BLASLAND, BOUCK & LEE, INC. engineers & scientists

FIGURE S-2

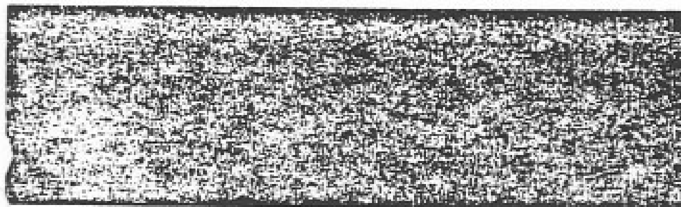
MVA.PCP
9/8/00 SYR-54-RCB
20139001/20139L01.DWG

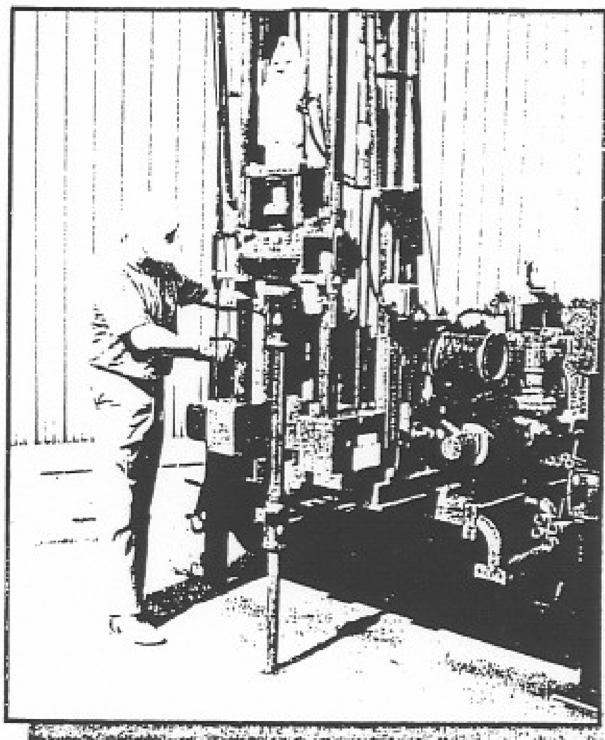
Attachment S-3
Direct-Push Drilling Method
Vendor Specifications

Cuttings free exploration with the ESP System eliminates disposal problems

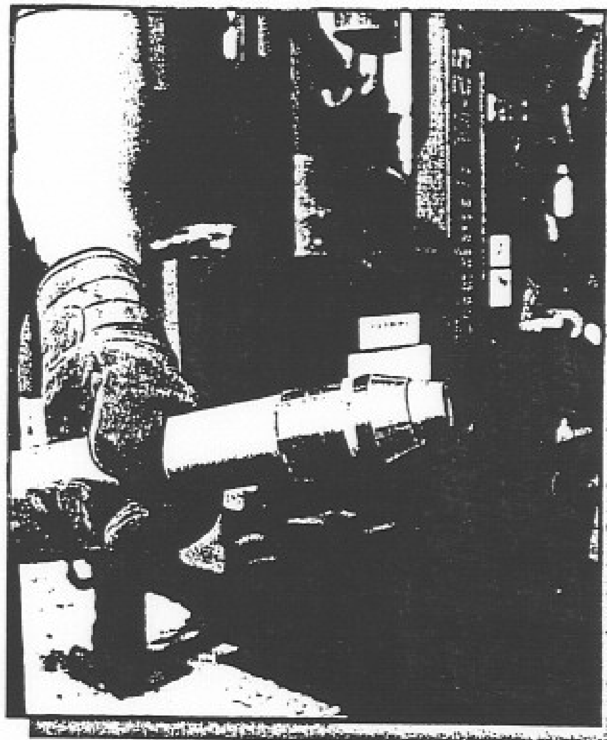


▲The ESP System allows you to utilize your drill for maximum equipment efficiency and adapts to most open spindle drill rigs.





▲Hydraulically operated hammer fits to your drill rig system. This hammer delivers 140 foot pounds/1680 inch pounds of energy with each blow. The hammer operates at 1200 blows per minute providing the force needed to penetrate very stiff/medium dense soil formations.



▲Tooling is designed to take the abuse offered by Mother Nature. The double rod system follows standard operating procedures common to the drilling industry.

86 ELM STREET· HOPKINTON, MA 01748 (508) 435-5543 (508) 435-5512 Fax

Attachment S-4
Well Development Field Log

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

Appendix T

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 Form

MAGNETOMETER SURVEY

CREW____________________ WEATHER_______________ NOTES_____________

DATE/TIME	STATION	READING

V:\GE_PITTSFIELD_GENERAL\REPORTS AND PRESENTATIONS\FSP-QAPP 2007\VOL II\164711324APPXT-ATTAT-1.DOC

Appendix U

Seismic Refraction Survey Procedures

Appendix U

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 a 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

Appendix V

Ground Penetrating Radar (GPR) 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

Standard Operating Procedures for Equipment Cleaning

Appendix W

Equipment Cleaning

I. Introduction

The equipment cleaning procedures described herein include pre-field, in the field, and post-field cleaning of sampling equipment which will be conducted in the field, or at GE's Building 12Y Equipment Decontamination Area (EDA), as appropriate. Sampling equipment consists of soil sampling equipment, well construction materials, groundwater, sediment, and surface water collection devices, water testing instruments, down-hole geophysical instruments, and other activity specific sampling equipment. 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 as specified in the applicable work plan.

II. Equipment and Materials

A designated area will be established to conduct cleaning of sampling equipment in the field prior to and between sample collection. Equipment cleaning areas will be set up within or adjacent to the specific work area. Equipment to be cleaned in the field may include split-spoons, bailers, well pumps, spatulas, etc.

A. Field Cleaning Materials

The following materials, as required, will be available during field cleaning procedures:

- health and safety equipment, as required in the *Health and Safety Plan* (HASP);
- distilled/deionized water;
- non-phosphate soap;
- tap water;
- appropriate cleaning solvent (e.g., hexane, acetone);
- nitric acid;
- 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 Sampling Equivalent when Analyzing for Organic Constituents

Step 1 - Non-phosphate detergent and water wash to removal all visible particulate matter and any residual oils or grease.

Step 2 - Tap water rinse to remove the detergent solution.

Step 3 - Solvent rinse with hexane (unless volatiles are being sampled, in which case methanol should be used).

Step 4 - 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 Sampling Equipment when Analyzing for Inorganic Constituents

Step1 - Non-phosphate detergent and water wash to removal all visible particulate matter and any residual oils or grease.

Step 2 - Tap water rinse to remove the detergent solution.

Step 3 - Nitric acid rinse.

Step 4 - Non-phosphate detergent and water wash to removal all visible particulate matter and any residual oils or grease.

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 and tap water rinse, followed by a flush of potable water through the pump. This flushing will be accomplished by the use of an appropriate container filled with potable 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. Disconnect the pump from 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, 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.

III. Soil Boring Installation

A. Decontamination of Heavy Equipment

Drilling rigs and other larger equipment utilized during sampling episodes may be cleaned at GE's Building 12Y EDA. Items such as drill rigs, well casing, and auger flights all present potential sources of contamination to environmental samples. 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 in Section IV. It will also be cleaned between drilling locations. Those areas that are in close proximity to materials being sampled, such as auger flights, drill rods, and drill bits, will be targeted for cleaning.

IV. Subsurface Soil Sampling Procedures

This procedure applies to heavy equipment, such as drill rigs, well casings, auger flights, etc., that are cleaned in the Building 12Y EDA.

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. 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:

- shop vacuum;
- 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.

C. Set Up

Step 1 - Put on protective clothing.

Step 2 - Provide proper signs and barricades for the cleaning area to control access.

Step 3 - Place the item to be cleaned on a cart inside one of the EDA washing chambers.

D. Cleaning Procedures

Step 1 - Pre-clean the entire piece of equipment to remove all loose dust, dirt, scale, etc. using a shop vacuum designed for solid material, supplemented by scraping, chipping, 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 cloth soaked in the cleaning solution. Control the application so that little or none of the cleaning solution puddles or runs down to the floor. 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 clean and dry. For larger items it may be appropriate to clean the equipment in sections.

Step 3 - Rinse the equipment with water in the EDA high pressure (1,500 psi) washing chamber. Water is treated and re-used.

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 re-use conditions of the GE PCB Cleaning Protocol.

Step 5 - Completely cover the equipment with polyethylene sheeting and secure the sheeting.

Step 6 - Use the shop vacuum to remove all loose material from the plastic sheeting on the floor.

Step 7 - Update the equipment tag and log.

Step 8 - Before leaving the area where a piece of equipment has been cleaned, a final check should be conducted 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, boots and gloves must be left behind. At the end of the day all PPE must be cleaned and stored on-site. No contaminated clothing 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, are to be collected and stored in DOT-approved drums. All drums shall be properly marked, labeled, stored, and disposed in accordance with existing site waste management procedures.

Appendix X

Building Material Sampling Procedures

Appendix X

Building Material Sampling Procedures

X-1 Wood Floor Sampling Procedures

X-2 Sheetrock Sampling Procedures

X-3 PCB Wipe Sampling Procedures

X-4 Oil Reservoir Sampling Procedures

X-5 Concrete Floor Sampling Procedures

X-6 Wood Column Sampling Procedures

X-7 Brick Wall Sampling Procedures

X-8 Wood Beam and Joist Sampling Procedures

X-9 Roof Deck Sampling Procedures

Appendix X-1

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

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

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

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

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. Decontaminate drill bit in accordance with the protocols presented in Appendix W.
3. Put on clean latex/nitrile protective gloves for each new sample collection.
4. Place paper template with center hole cut out over determined sample location.
5. Using a coring machine bit, drill the full depth of the concrete floor. Concrete debris should be uplifted and deposited on paper template.
6. Place the concrete sample into the sample container.
7. Close, cap, and seal sample container.
8. Remove and dispose of protective gloves in appropriate container.
9. Repeat procedure at nearby locations until sufficient volume has been obtained to complete the sample requirement.
10. Complete chain-of-custody forms, prepare shipping containers, and send to laboratory for analysis.

Appendix X-6

Wood Column Sampling Procedures

Proper personal safety equipment shall be worn in accordance with the Site Health and Safety Plan.

Make sure wood component is sound and in good condition before executing any sampling activities.

Drill bit should be less than 2 inches in diameter.

1. Documentation shall be in accordance with Appendix L.
2. Decontaminate drill bit and spatula in accordance with the protocols presented in Appendix W.
3. Put on clean latex/nitrile protective gloves for each new sample collection.
4. Place clean collection plate below drilling location on both sides of column to catch wood debris sample.
5. Drill hole through entire width of column.
6. Remove and tap drill bit with wood tool to remove remaining wood debris and collect on plate.
7. Using the decontaminated spatula, collect the wood sample debris and deposit in sample container.
8. Close, cap, and seal sample container.
9. Remove and dispose of gloves in appropriate container.
10. Complete chain-of-custody forms, prepare shipping containers, and send to laboratory for analysis.
11. 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

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. Decontaminate drill bits and spatula in accordance with the protocols presented in Appendix W.
3. Put on clean latex/nitrile protective gloves for each new sample collection.
4. Place clean collection plate against wall below drilling location to catch brick debris sample.
5. Using a Hilti hammer drill equipped with a pulverizing bit, drill 3-1/2 inches into brick wall. Brick debris should fall on collection plate. Remove drill bit and inspect for grouting. Sample is to be obtained from inner wythe (i.e., course) of brick wall only. Redrill in ¼-inch increments to the depth of the first brick course or until grouting is observed.
6. Remove and tap drill bit to remove remaining brick debris and collect on plate.
7. Using the decontaminated spatula, collect the brick sample debris and deposit in sample container.
8. Close, cap, and seal sample container.
9. Remove and dispose of protective gloves in appropriate container.
10. Repeat procedure for total number of samples required.
11. Complete chain-of-custody forms, prepare shipping containers, and send to laboratory for analysis.

Appendix X-8

Wood Beam and Joist Sampling Procedures

Proper personal safety equipment shall be worn in accordance with the Site Health and Safety Plan.

Make sure wood component is sound and in good condition before executing any sampling activities.

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.
2. Decontaminate drill bit and spatula in accordance with the protocols presented in Appendix W.
3. Put on clean latex/nitrile protective gloves for each new sample collection.
4. Place sample location at mid-height of beam and at a distance of approximately 1/6 of total beam length from the end.
5. Place clean collection plate below drilling location to catch wood debris sample.
6. Drill 1-inch diameter hole through entire width of beam.
7. Remove and tap drill bit with wood tool to remove remaining wood debris and collect on plate.
8. Using the decontaminated spatula, collect the wood sample debris and deposit in sample container.
9. Close, cap, and seal sample container.
10. Remove and dispose of protective gloves in appropriate container.
11. Repeat procedure for total number of samples required.
12. Complete chain-of-custody forms, prepare shipping containers, and send to laboratory for analysis.

Appendix X-9

Roof Deck Sampling Procedures

Proper personal safety equipment shall be worn in accordance with the Site Health and Safety Plan.

Make sure roof component is sound and in good condition before executing any sampling activities.

Coordinate with GE project manager and/or subcontractor to repair roof following execution of sampling procedure.

1. Documentation shall be in accordance with Appendix L.
2. Decontaminate saw in accordance with the protocols presented in Appendix W.
3. Use new clean razor blade (on Carpenter's knife) to cut roof membrane and expose 4-inch by 4-inch area of wood decking.
4. Put on clean latex/nitrile protective gloves for each new sample collection.
5. Using saw, cut 4-inch by 4-inch area from roof decking and place in sample container.
6. Close, cap, and seal sample container.
7. Remove and dispose of protective gloves in appropriate container.
8. Repeat procedure for total number of samples required.
9. Complete chain-of-custody forms, prepare shipping containers, and send to laboratory for analysis.

Appendix Y

Selection of Drilling Method

Appendix Y

Selection of Drilling Method

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

Appendix Z

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 Concret/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 for Using Passive-Diffusion Bags

Appendix AA

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 principal 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 biphenols, 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 sampler, constructed as described below;
- Water level probe;

- Downhole temperature, pH, specific conductivity, ORP, and/or turbidity meters (optional);
- Appropriate water sample containers;
- Appropriate blanks (trip blank supplied by the laboratory).

III. Procedures

Passive-Diffusion Bag Sampler Construction

Figure AA-1 illustrates a passive-diffusion bag sampler design. Passive-diffusion bag samplers will be constructed in a clean, laboratory or office environment according to the following procedures.

Step 1 - Cut a roll of 3-mil to 4-m polyethylene, flat (2-inch wide) tubing into 18-inch-long sections. Close one end of each cut section using an appropriate heat sealer to create an elongate bag. Fill the bag with approximately 250 milliliters (ml) of laboratory-grade analyte-free water. Heat-seal the other end of the bag with minimal headspace.

Step 2 - Construct a similar, 12-inch-long weight bag filled with washed gravel (standard monitoring well sand-pack material) and analyte-free water.

Step 3 - Cut a roll of polyethylene mesh sleeve into 3-foot-long sections. Place the passive-diffusion sample bag and weight bag in a polyethylene mesh sleeve. Weave nylon pull-ties into the mesh sleeve to secure the weight bag and the diffusion bag and close the ends of the mesh sleeve.

Step 4 - Attach a braided nylon rope to the upper nylon pull-tie and the top of the mesh sleeve using a stainless steel snap hook. Determine the depth of the desired monitoring interval from the top of the well casing based on well construction data +/- geologic information. With reference to the center of the passive-diffusion bag, measure an appropriate length of nylon rope to place the center of the passive-diffusion bag 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), cut the rope and tie a reference knot corresponding to the top of the well casing. The reference knot facilitates placing the passive-diffusion bag at the proper depth within the monitoring well.

Step 5 - Place each passive-diffusion bag sampler in a clean, labeled plastic bag, and transport 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 6 - Don appropriate personal protective equipment (as required by the Health and Safety Plan).

Step 7 - Place plastic sheeting around the well.

Step 8 - 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.

Step 9 - 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 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 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 photoionization detector 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 10 - Measure the depth to water.

Step 11 - Remove the appropriate passive-diffusion bag sampler from the labeled plastic bag. Slowly lower the passive-diffusion bag sampler into the monitoring well until the bag is properly positioned, with the reference knot even with the top of the well casing.

Step 12 - 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 13 - Close and lock the well.

Step 14 - Allow an equilibration period of 14 days or more before retrieving the passive-diffusion bag. 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.

<u>Passive-Diffusion Bag Sampler Retrieval and Sample Collection</u>

Step 15 - After the equilibration period, unlock and open the monitoring well following Steps 6 through 10 (above). Slowly remove the passive-diffusion bag sampler from the monitoring well.

Step 16 - Remove the passive-diffusion bag from the mesh sleeve. Create a small hole in the sample-filled polyethylene bag using a decontaminated knife. Pour water from the bag directly into appropriate laboratory glassware.

Step 17 - 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 18 - Secure with packing material and store at 4°C in an insulated transport container provided by the laboratory.

Step 19 - 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 20 - Place the passive-diffusion bag 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 PPE and disposable equipment (see Section VI).

Step 21 - 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, 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 passive-diffusion bag is retrieved and sampled at a given well. This protocol will provide the best practicable comparison between the results of the different sampling methods.

- Each passive-diffusion sampler 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.

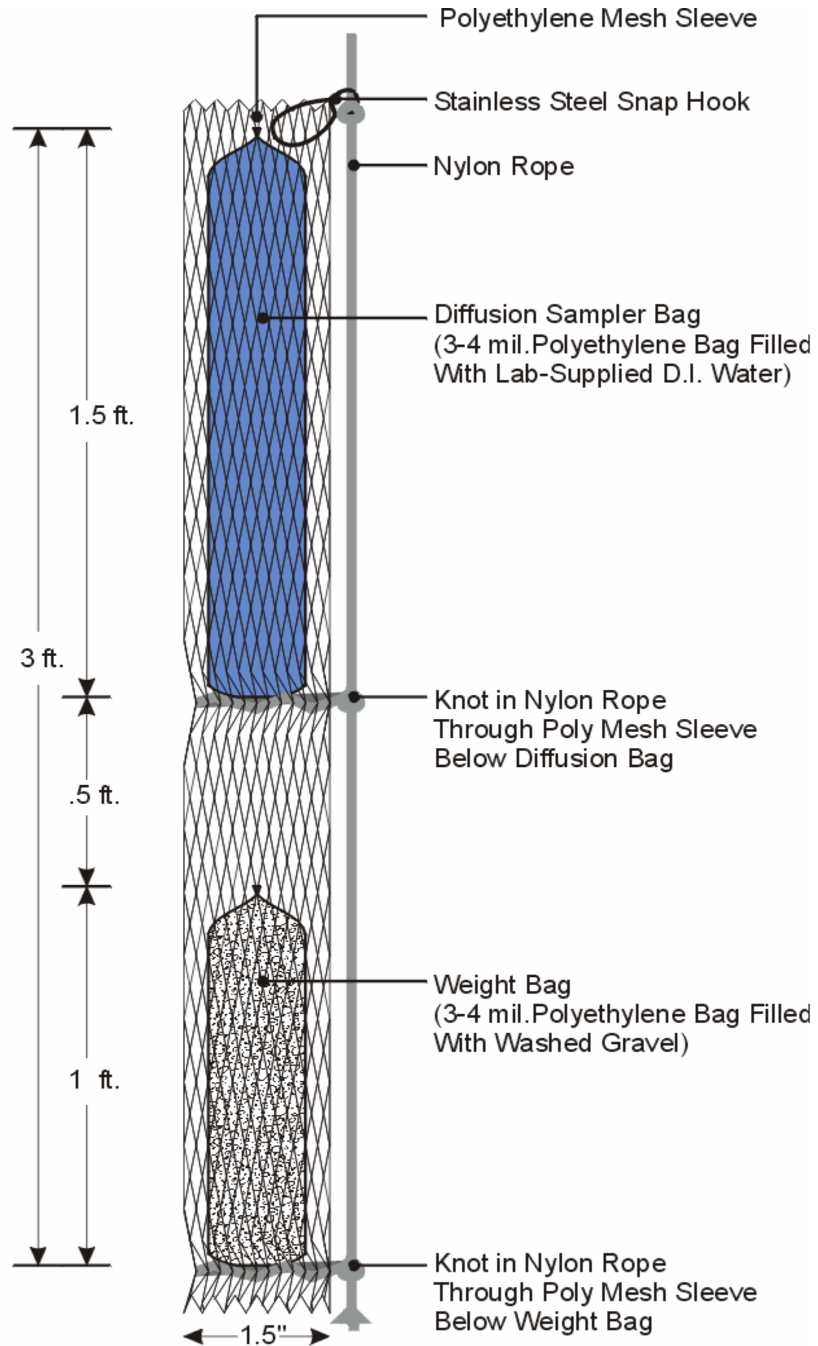
V. Equipment Cleaning

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



NOTE: All dimensions are approximate.

Attachment AA-1
Passive-Diffusion Bag Sampling Record

PASSIVE-DIFFUSION BAG (PDB) SAMPLING RECORD (Page 1 of 2)

Well ID:	Well Diameter:
Project/Task No.:	Top of Casing Stick-Up:
Project Name:	Total Depth of Well (D):
Date:	Well Screen Interval (B to C):
Field Personnel:	Depth to Water (A):
PDB Installation Date:	Depth to Top Bag Attachment (L):
PDB Retrieval Date:	Conventional Sampling Conducted: ☐ Yes ☐ No
PDB Holder Material:	☐ Low Flow Purge Date: ______
PDB Diameter:	☐ Multi-Volume Purge

PDB PREPARATION

Date Prepared	Date Received	Prepared by	DI Water Source	Comments

PDB INSTALLATION

Depth (feet below TOC)	Time	Comments

PDB RETRIEVAL **Sample ID:**

Depth (feet below TOC)	Time	Diffusion Period (days)	Comments (include condition of PDB upon retrieval)

PDB FIELD DUPLICATE ☐ Yes ☐ No **Duplicate ID:**

Depth (feet below TOC)	Time	Comments

FIELD PARAMETERS ***(Not to be measured using PDB sample. Downhole probe, pumping, or bailing required.)***

Date	Time	Sampling Depth (feet)	Temp (°C)	pH (units)	Specific Electrical Conductance (μS/cm)	Dissolved Oxygen (mg/l)	Redox Potential (mV; SSCE)	PDB Status[1]

[1] Note if pre-PDB installation, PDB in well, or post-PDB retrieval (A, B, or C).

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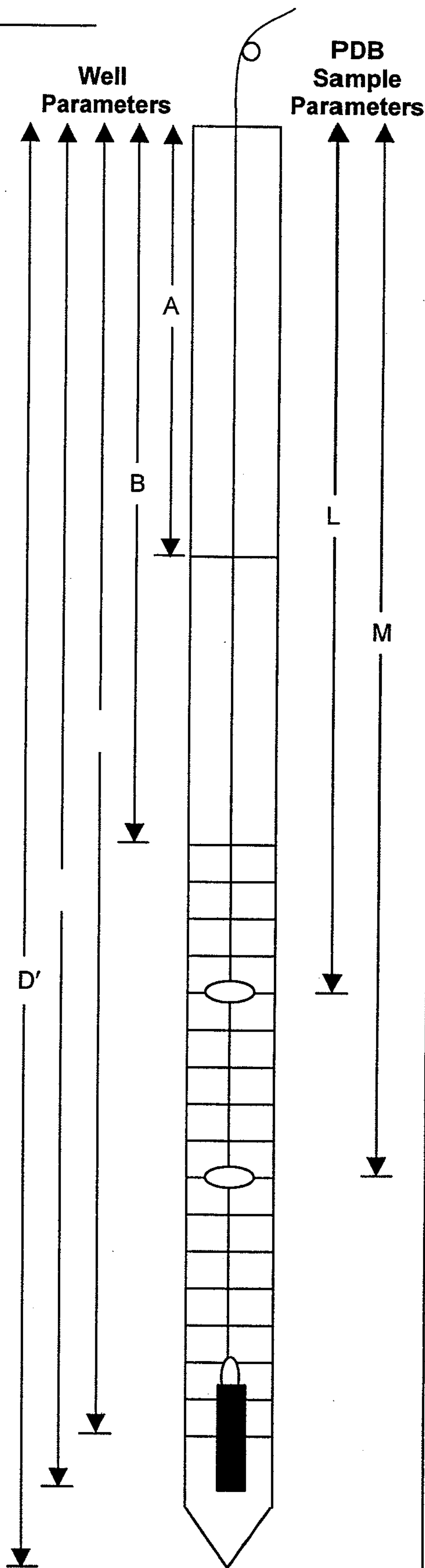
PASSIVE-DIFFUSION BAG (PDB) SAMPLING RECORD (Page 2 of 2)

Well ID: ______________ **Date:** ______________ **Time:** ______________

Project/Task No.: ______________________________

Project Name: ______________________________

		(feet)	
Depth to Water		________	(A)
Depth to Top of Screen		________	(B)
Depth to Bottom of Screen		________	(C)
Total Depth of Well (field measurement)		________	(D)
(initial well installation)		________	(D′)
Depth to Middle of Screen = (B+C)/2		________	(E)
Height of Water above Top of Screen = (B-A)		________	(F)
Depth to Midpoint of Saturated Interval			
F > 0	= E	________	(G)
F ≤ 0	= (A+C)/2	________	(G)
Total Length of PDB		________	(H)
Total Length of Water-Filled Portion of PDB		________	(I)
Distance from Hole for Top Bag Attachment to Top of Water-Filled Portion of PDB		________	(J)
Distance from Hole for Bottom Bag Attachment to Bottom of Water-Filled Portion of PDB		________	(K)
Depth to Top Bag Attachment	= (G - ½ I - J)	________	(L)
Depth to Bottom Bag Attachment	= (G + ½ I + K)	________	(M)
Max. Depth to Bottom Bag Attachment	= (D - 0.6)	________	(N)[1]
Total Length of PDB Holder Material	= (D + 2)	________	



Notes:

All depths to be measured to the nearest 0.01 feet below top of casing (TOC).

[1] Confirm that M < N.

(Based on original sampling form prepared by Geomatrix Consultants.)

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Appendix BB

Soil/Water Shake Test Procedures

Appendix BB

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

Appendix CC

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 Sediment/Water/ NAPL Sampling Procedures

Appendix DD

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 heath 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

Appendix EE

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

Appendix FF

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

Appendix GG

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

BBL
BLASLAND, BOUCK & LEE, INC.
engineers & scientists

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: ______

Appendix HH

Procedure for Determination of Total Organic Carbon in Solid Samples

Appendix HH

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) approved by EPA in fall 2001, will be utilized. A copy of that SOP is provided as Attachment HH-1.

Attachment HH-1
SOP for the Determination of Total Organic Carbon in Solid Samples

STANDARD OPERATING PROCEDURE

Author: John Nicpon
Reviewed by: William A. Kotas

____
Approved by:

____

Northeast Analytical, Inc.
Issuing section: Inorganics Department
NE177_01.SOP
Date: 22-October-2001
Revision Number: 1

1.0 TITLE

Standard operating procedure for the determination of Total Organic Carbon in solid samples according to The US-EPA Lloyd Kahn Method Reference and Tekmar-Dohrmann application note TOC-011 with the triplicate analysis option.

2.0 PURPOSE

The purpose of this SOP is to provide procedures for Particulate Organic Carbon (POC) and Total Organic Carbon (TOC).

3.0 SCOPE

This method is applicable to waste water and ground water for POC, and sediments and filters for TOC.

4.0 COMMENTS

Organic carbon is converted to carbon dioxide (CO_2) by catalytic combustion or wet chemical oxidation. 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.

The fractions of total carbon (TC) are defined as:
1) inorganic carbon (IC)-the carbonate, bicarbonate, and dissolved CO_2;
2) total organic carbon (TOC)-all carbon atoms covalently bonded in organic molecules;
3) dissolved organic carbon (DOC)-the fraction of TOC that passes through a 0.45-µm -pore-diameter filter,
4) particulate organic carbon (POC)-also referred to as non dissolved organic carbon, the fraction of TOC retained by a 0.45-µm filter.

IC interference can be eliminated by acidifying samples to pH 2 or less to convert IC species to CO_2. Subsequently, purging the sample with a purified gas removes the CO_2.

Principle: Depending upon the configuration, TOC can be measured by ultra-violet promoted persulfate oxidation or high-temperature combustion, followed by infrared detection.

1) TOC and POC 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°C in an oxygen atmosphere so that solids as well as liquids can be analyzed.

The sampler consists of a magnetically coupled boat inlet system which delivers the sample to the high temperature furnace. Two ports are provided for sample introduction, a septum port for liquid injections, and a flip-top port for solid samples. The CO_2 from the oxidation of organic carbon is transported in the carrier-gas stream and is measured by means of a nondispersive infrared analyzer (NDIR).

The detection limit for samples is dependent on the amount of sample analyzed.

Sampling and storage: The holding time for analyzing soil samples for TOC is 28 days from the date that the samples are collected. Samples are to be stored at 4°C until the time of analysis.

The holding time for analyzing water samples for POC is 14 days from the date that the sample was collected. Collect samples in one liter containers with Polyseal caps. Do not add any preservative to the bottles or samples. Samples are to be stored at 4°C until the time of analysis.

5.0 SAFETY

5.1 Safety glasses and disposable gloves must be worn when handling chemicals and samples.

5.2 Personnel should familiarize themselves with the necessary safety precautions by reading MSDS information covering any chemicals used to perform SOP.

6.0 REQUIREMENTS

6.1 Method detection limit study.

6.1.1 Seven MDLs samples (spike seven aliquots of the TOC standard into the boat module) The MDL should be determined annually at a spiked concentration of two to three times the estimated instrument detection limit.

6.1.2 Analyze the seven replicate samples according to the procedures set forth in this document. Calculate the MDL by multiplying the standard deviation of seven MDL measurements by 3.14. For the MDL to be valid, it must be greater than 1/10 the amount spiked but not greater than the amount spiked.

6.1.2 Knowledge on the operation and maintenance of the Dohrmann DC-80 series IR-I NDIR detector and sludge/sediment sampler modules.

6.1.3 Trainees are required to read the Instrument manual and take notes on subject matter not covered in SOP. Information about maintenance and replacement on specific parts not covered in SOP should be recorded on the "Notes" page of the SOP for future reference.

7.0 EQUIPMENT

7.1 Equipment.

7.1.1 Dohrmann IR-I NDIR detector module. Located in the main laboratory.

7.1.2 Dohrmann sludge/sediment sampler. Dohrmann (p/n 832-222). Located in the main laboratory.

7.1.3 250 and 1000 µL Rainin autopipets. Rainin (p/n EP-250 and EP-1000).

7.1.4 250 and 1000 µL pipet tips. Rainin (p/n RT-96 and RT-200).

7.1.5 1-5 ml Finn digital pipette with pipet tips. Baxter (p/n P5055-14).

7.1.6 Quartz boats. Dohrmann (p/n 899-624). Located in the main laboratory.

7.1.7 Quartz wool. Dohrmann (p/n 511-735). Located in the main laboratory.

7.1.8 GC oven. Set at 75 ^{O}C. Located in the main laboratory.

7.1.9 Propane tank with torch assembly. Located in the main laboratory.

7.1.10 Tweezers and steel spatula. Located in the main laboratory.

7.1.11 Analytical balance. Located in the main laboratory.

7.1.12 Centrifuge. Located in the main laboratory.

7.1.13 40 ml VOA vials. Located in the bottle storage room.

7.1.14 50, 100 and 250 µl syringe. Located in the main laboratory.

7.1.15 High purity oxygen tank with regulator. Located in the main laboratory.

7.1.16 Aluminum weighing boats. Located in the main laboratory.

7.1.17 Gray septum. Dohrmann (p/n 517-807). Located in the main laboratory.

7.1.18 Pasteur Pipets. Located in all laboratories.

7.1.19 UV-Persulfate Reaction Module. Located in the main laboratory.

7.1.20 Blue injection septum. Dohrmann (p/n 517-811). Located in the main laboratory.

7.1.21 Teflon sleeve reactor, taper joint. Dohrmann (p/n 070-627). Located in the main laboratory.

7.1.22 Lamp, Ultra-violet. Dohrmann (p/n 512-092). Located in the main laboratory.

7.1.23 Peristaltic pump tubing.
- a) PVC Black/Black (p/n 899-641).
- b) PVC Green/Green (p/n 899-645).
- c) Viton A Purple/Purple (p/n 899-651).

7.1.24 High purity nitrogen tank with regulator. Attach plastic tubing to the regulator. Located in the main laboratory.

7.1.25 20-mesh tin. Dohrmann (p/n 511-876). Located in the main laboratory and used for tin/copper scrubber.

7.1.26 Copper. Dohrmann (p/n 511-895). Located in the main laboratory and used for tin/copper scrubber.

7.1.27 Pyrex wool. Dohrmann (p/n 511-895). Located in the main laboratory and used for tin/copper scrubber.

7.2 Reagents.

7.2.1 Laboratory grade water. Located in the cooler room.

7.2.2 ~2500 mg/L TOC stock standard. Mallinkrodt (p/n 6704-1). Dry potassium hydrogen phthalate crystals (primary standard grade) in 104 ^{o}C oven for 2 hours and weigh out approximately 2.65675 grams. Record the weight in the Inorganic standard logbook and dissolve in approximately 400 ml of laboratory grade water, add 2 ml of phosphoric acid and bring to a final volume of 500 ml. Calculate the exact concentration of the solution:
(weight of potassium hydrogen phthalate) X 941 = TOC stock standard {mg/L}

7.2.3 TOCS and POC calibration standards:
Prepare 5 calibration standards of different concentrations ranging from ~120.7 - ~1207 mg/L. Record the date and information related to the preparation of the calibration standards in the Inorganic standard logbook.

7.2.5 7.9N (1+1) nitric acid. Dilute 50 ml of concentrated nitric acid to a final volume of 100 ml. Located in the Inorganics laboratory.

7.2.6 ICV/CCV: TOCS and POC 1000 mg/L TOC control. Ricca (p/n 1847-16). Located in the Inorganics laboratory.

7.2.7 ICV/CCV: TOC in water 10 mg/L TOC control. Ricca (p/n 1847-16). Dilute 1000 mg/L ICV/CCV standard 100x. Located in the Inorganics laboratory.

7.2.8 Concentrated phosphoric acid (H_3PO_4). J.T. Baker. 'Baker analyzed', (Baxter p/n 0260-01*BC). Located in the Inorganics laboratory.

7.3 Glassware and apparatus.

7.3.1 10, 25, 50, 100 ml Class A volumetric flasks. Located in the Inorganics laboratory.

7.3.2 100 ml graduated cylinder. Located in the Inorganics laboratory.

7.3.3 Rinse bottle. Filled with laboratory grade water. Located in the Inorganics laboratory.

7.3.4 TOC logbook. Located next to TOC instrument.

8.0 PROCEDURE

8.1 Operation and maintenance of the Dohrmann IR-I NDIR detector module.

8.1.1 Refer to the instrument manual for specific instructions and part numbers for all components.

8.1.2 To prepare the tin/copper scrubber, fit one end of the Pyrex scrubber tube with a cored gray septum. Insert a tuft of Pyrex wool and then about 2 inches of tin in the other end. Secure the tin with another tuft of Pyrex wool. Then, fill the remaining half of the scrubber tube with an equal amount of copper. Secure the copper with a third tuft of Pyrex wool. Insert a cored gray septum. Inspect the tin/copper scrubber and change the contents of the tube when one-half of the tin is discolored.

8.1.3 The detector must be on for several hours in order to achieve equilibrium. It is recommended that the detector is turned on the day before the analysis is to be performed. Power up the detector and the main unit.

8.1.4 Verify that the printer has sufficient amount of paper before starting the analysis. Reset the printer so that the number "1" will be printed for the first analysis performed for that day.

8.1.5 Select the "TOC" and the "DET" positions. For the detector, select position "3" for high concentrations, "2" for medium concentrations, and "1" for low concentrations of TOC.

8.1.6 The module will not light the green "ready" light if the baseline is above 0.05. Adjust the "zero" control until the baseline is less than 0.02. The "CALIB" light must be off during analysis.

8.2 Operation and maintenance of the Dohrmann sludge/sediment sampler.

8.2.1 Refer to the instrument manual for specific instructions and part numbers for all components.

8.2.2 A portion of sample is weighed into a quartz boat where it is acidified and dried. The boat is placed in the boat carriage of the sampler and it is moved into the combustion chamber. Gas from the combustion tube flows into the flask to the right where it passes through acidified water.
The gas travels to the flask to the left where excess water is removed before traveling to the detector module. The gas passes through the tin and copper scrubber and into the detector.

8.2.3 Before turning on the solid sampler, carefully examine individual components for sign of wear. Adjust the flow of oxygen to 30 psi. The level of acidified water in the right flask must be above the fritted sparging finger. A vigorous flow of gas emitting from the sparging finger should be easily observed, if not, check the gas lines and connections for leaks. The water collection flask should be emptied on a daily basis.

8.2.4 Turn on the furnace unit. When using the module for the first time or after a long period of inactivity, the furnace should be monitored with a voltmeter to verify that the temperature is at 800°C. Place the black (ground) probe in the "com" port. Place the red (positive) probe in the "monitor", set the voltmeter to "volts". The voltage reading should read "0.80", if not, place the red probe in the "adj" port. The voltage reading should read "0.80", if not, adjust the voltage by turning the set screw until the correct voltage is achieved.

8.2.5 If the gray septum (p/n 517-807) at either end of the combustion tube have corroded and require replacement, the furnace must be turned off before replacing the septum.

8.3 Calibration of Dohrmann sludge/sediment sampler and IR-I NDIR detector module.

8.3.1 Calibration standards are prepared as dilutions of the primary stock standard. All preparations are recorded in the standard preparation logbook. A new calibration curve must be generated if either the ICV or CCV (see **8.11 Quality Control**) are unacceptable. The calibration curve is based on 'µg of carbon' versus 'area'. The same volume of each pre-prepared calibration standard is are injected onto a quartz boat that is lined with quartz wool. The calibration standards require duplicate injections. The relative percent difference of the absorbance should be less than 25% for the duplicate injections.

8.3.2 A fresh tuft of quartz wool must be inserted into the boat before calibrating the instrument. The boat is placed inside the sediment sampler module. Hook the loop of the boat with the end of the magnetic boat carriage.

8.3.3 Remove contaminates from the boat by placing it in the furnace until the baseline has started to decrease. Pull the boat out of the furnace.

8.3.4 After the boat has cooled (approximately 30 seconds), place the boat underneath the injection port. Remove septum and inject calibration standard onto the boat. Replace septum.

8.3.5 After the baseline has stabilized, place the boat in the furnace. Press the "Start" button. After the signal has started to decrease, pull the boat out of the furnace.

8.3.6 Repeat **8.3.2-.6** for the remaining calibration standards.

8.3.9 The calibration and continuing check blank consists of 50 ml of laboratory water and one ml of 1+1 nitric acid. Inject 70 µl of the blank solution for the calibration and continuing check blanks.

8.3.10 For TOC solids and POC, inject 70 µl of each calibration standard and the stock standard. If the needle in the IR meter goes past '95' or if the red error light has lit after injecting the stock standard, inject a smaller volume of the standard. Every standard must be within the scale of the detector.

8.3.11 Enter the injection number, standard label, date analyzed, injection volume, and the area printed by the printer in the TOC logbook. See the **Glossary** for information about the correlation coefficient.

8.4 Preparation of solid samples.

8.4.1 Between 1.0 and 20 mg of material can placed in a boat depending on the percent of carbon in the sample. Solid samples are analyzed in duplicate.

8.4.2 The concentration of the samples must be within the range of the calibration curve. If the sample concentration of the sample is outside the range of the calibration curve, repeat the analysis of the sample. If the µg of carbon of the sample was too low, use more sample up to 20 mg. If the sample concentration was too high, use less sample down to 1.0 mg.

8.4.3 Place each boat in a numbered aluminum weigh boat.

8.4.4 Homogenize soil and sediment samples by first decanting any standing water then emptying the contents of the sample jar onto a plastic weighing boat or other container. Remove an gross objects such as twigs, large pebbles, and foreign objects and mix the sample thoroughly. The sample should have a uniform consistency similar to sand, silt or topsoil. If the sample consists entirely of large rocks or other gross objects consult the Inorganics Supervisor or Laboratory Director for furthur homogenization procedures.

8.4.5 Place one boat on the analytical balance and tare the balance. Transfer an aliquot of the sample to the boat and record the NEA #, weight and the boat number in the TOC logbook. Place the boat in the numbered aluminum weigh boat.

8.4.6 Perform **8.4.5** two more times for a triplicate analysis

8.4.7 Add 2 to 3 drops of 1+1 nitric acid to each sample. Turn off the GC oven. Place the aluminum weigh boats in the GC oven. Place a 60 ml beaker over each quartz boat. Turn on the GC oven. Remove samples when dried (minimum of 10 minutes).

8.4.8 Place the boat in the raceway. After the baseline has stabilized, place the boat in the furnace and press the 'Start' button.

8.4.9 Copy the TOC area from the printer into the TOC logbook. Calculate percent relative standard deviation of the area measurements. The percent relative standard deviation for the triplicate analysis should not exceed 25%. If the % RSD exceeds 25% perform an additional determination. Record all measurements in the TOC injection logbook

8.4.10 After each sample analysis, scrape any remaining material from the boat and place the boat in the flame of the propane torch to remove any contaminates.

8.4.11 Repeat **8.4.3-.9** for the remaining samples.

8.5 Percent total solids determination

8.5.1 Determine the percent total solids for each sample as described in NE090.

8.6 The determination of Particulate organic carbon (POC) in water.

8.6.1 The purpose of this procedure is to separate the non dissolved TC compounds from the dissolved TC compounds by centrifuging the water sample. The IC fraction of the sample is removed by the addition of 1+1 nitric acid to the particulate matter.

8.6.2 Shake the sample bottle and measure a maximum of 80 ml aliquot of the sample with a graduated cylinder. Pour the sample into two labeled volatile (VOA) vials. Verify that sample levels in each vial are equal to each other.

8.6.3 Centrifuge the VOA vials at a setting of '7' for 5 minutes.

8.6.4 Remove all of the water from each vial until approximately 10 ml remain in each vial.

8.6.5 Transfer all the material (water and particulates) from the two vials to one vial.

8.6.6 Centrifuge the vial with the water and particulates at a setting of '7' for 5 minutes.

8.6.7 Remove all the water from the vial. Set the 1000 µl Rainin pipet to 650 µl and transfer the particulates to a quartz boat.

Note: If all the material from the VOA vial will not fit inside the boat, transfer a portion of the material from the vial to the boat and dry the boat and the material inside the GC oven. Repeat the process of transferring the sample from the vial to the boat and drying the material until all the sample extract has been transferred to the boat.

8.6.8 Place the boat in the numbered aluminum weigh boat. Record the NEA #, volume of sample centrifuged and the boat number into the TOC logbook.

8.6.9 Add 2 to 3 drops of 1+1 nitric acid to each sample. Turn off the GC oven.. Place the aluminum weigh boats in the GC oven. Place a 60 ml beaker over each quartz boat. Turn on the GC oven. Remove samples when dried (minimum of 10 minutes).

8.6.10 Follow the instructions in **8.3.6** for analyzing samples.

8.6.11 The concentration of the samples must be within the range of the calibration curve. If the sample concentration was too high, extract less than 80 ml of the sample.

8.7 Sample calculations utilizing Lotus spreadsheets.

8.7.1 After the instrument is calibrated, a Lotus spreadsheet is used to construct a calibration curve and the linear regression statistics. Generate a new spreadsheet each time the instrument is calibrated.

8.7.2 Log into the network and access "Lotus 1-2-3". Recall a previous spreadsheet, see the following table for an example of the directories and examples of files saved on November 11, 1996.

Analyte (matrix)	Lotus directory	Example
TOC (solids)	S:\DATA\TOCS*.*	S:\DATA\TOCS\1118.WK6
TOC (water)	S:\DATA\TOC*.*	S:\DATA\TOC\1118.WK6
POC (water)	S:\DATA\POC*.*	S:\DATA\POC\1118.WK6

8.7.3 Enter the average area (subtract the average blank area) for the calibration standards in the box used for constructing the calibration curve. Update the linear regression. For the calibration curve, enter the date of analysis.

8.7.4 The calibration curve standard concentrations are calculated from the TOC area measurements by the spreadsheet. The calculated calibration points are compared with the stated calibration standard concentrations. Except for the lowest calibration standard, the percent recoveries for the calibration standards must be between 90 and 110%.

8.8 Quality control (see attachment B for corrective actions)

8.8.1 A calibration blank is required for each day of analysis. Check blanks are analyzed after every initial and continuing check standard. The concentration of the blank must be less than the MDL for that method.

8.8.2 **Sample duplicate:** A duplicate analysis is performed every 10 samples.
RPD = Abs. {(S1 - S2)/(S1 + S2)} X 200

8.8.3 **Independent and continuing calibration verification standard (ICV) and (CCV)**: A purchased TOC solution of known concentration is analyzed after each calibration curve is generated, after every 10 samples and at the beginning and end of the analysis.
The ICV/CCV is analyzed in replicate.
% recovery = (calculated value/certified value) x 100.

8.8.4 For soil samples, if the sample analyses was off scale <u>and</u> the minimum sample weight of 1.0 mg was used, calculate the maximum concentration of TOC based on the μg of carbon of the highest calibration standard,

average sample weight, and the percent total solids. Report the results as greater than the calculated maximum sample concentration , the detection limit and the standard deviation

8.8.5 **Laboratory fortified sample matrix**. Perform a spike on every 20th soil or water sample. For water samples, spike 10 ml of the sample with an aliquot of the ICV/CCV standard and proceed as in **8.9.2-.4**. For soil samples, weigh the sample and proceed as in **8.4.1-.10**. Place the sample and boat in the boat sampler and spike the sample through the injection port with the ICV/CCV standard. The final concentration of the spiked sample must be within the calibration curve.
% recovery = {(spike sample conc.) - (sample conc.)}/(spike added) X 100

8.9 Entry of data into LIMs.

8.9.1 After the calibration curve has been completed, give the LIMs manager a copy of the Lotus spreadsheet for the calibration curve with the area for blank and area for the lowest standard used in the calibration curve.

8.9.2 Log into LIMS. Click "Win Results" or "Results" from LIMS toolbar. Select the appropriate samples by either typing in the sample ID's or selecting the Login Record File.

8.9.3 Choose the result entry template "TOCSOL", then click "OK". A result entry spreadsheet will then be created with the following columns: TOCBLANK, TOCSLOPE, $TOCAREA, $TOCWTWG, $TOCFINL, %SOLIDS. To find out what should go into these QC data columns, right click on the column heading in gray at the top of the spreadsheet.

8.9.4 The data for samples should be entered into the columns as follows:

$TOCBLANK = Calibration Blank Absorption

$TOCSLOPE = Inverse Slope Absorption

$TOCAREA = Area Counts for Sample

$TOCWTWG = Sample weight in grams

%SOLIDS = % Total Solids for Sample (Enter as a percentage, not a decimal)

$TOCFINL = Final result for TOC in Solids (Fills in automatically) along with the Average and %RSD

8.9.5 Once the field $TOCFINL has been filled in by the computer, right click on that field and select "detailed edit" from the pull down menu. Confirm that the MDL and the date analyzed for the sample are correct. Proceed to the next sample.

8.9.6 Once the data has been entered for all samples, go the QC section of the spreadsheet. If batching was performed correctly there should be some of these fields displayed in white. Right click on the dark gray fields in that same row so that all appropriate QC tests have been added.

(For example, if the sample has a duplicate be sure all the raw data fields for the duplicate have been turned white.) Enter in all appropriate QA/QC data.

9.0 REFERENCES

9.1 "Determination of Total Organic carbon in sediment," Lloyd Kahn, U.S.E.P.A. Region II, Edison NJ.

9.2 Application Note: TOC-011 "Analysis of sludges and solids for carbon," Tekmar-Dohrmann, Cincinnati, OH 10/95.

9.3 NYSDOH ELAP manual item #271, 4/15/94.

9.4 *Standard Methods for the Examination of Water and Wastes*, method #5310B, 17th edition. 1989.

10 ATTACHMENTS

10.1 **Attachment A**: Note pages for analyst.

10.2 **Attachment B**: Quality assurance and corrective action for problems associated with sample preparation and analysis.

10.3 Attachment C: Disposal of samples and waste.

ATTACHMENT A
NOTES

ATTACHMENT A CONTINUED
NOTES

ATTACHMENT B
QUALITY ASSURANCE AND CORRECTIVE ACTIONS.

Calibration curve: If the correlation coefficient is < 0.997 or if the recoveries for any of the calibration standards (which are established by applying the average absorbance measurements to the calibration curve) are not within 10% of the stated value (except for the lowest standard), repeat injections of the outlying standards until curve is within acceptance criteria.

Independent calibration verification (ICV/QCS): Must be within 85 –115% of true value. If the Percent recovery is not within the limits specified, recalibrate the instrument and reanalyze all samples since the last compliant continuing calibration verification standard.

Check standard (CCV/IPC): Use ICV solution as mentioned previously. Must be within 85 –115% of true value. If the Percent recovery is not within the limits specified, recalibrate the instrument and reanalyze all samples since the last compliant continuing calibration verification standard.

Preparation blank: For TOC in water, prepare one blank consisting of laboratory grade water for each batch of samples sparged daily. Prepare blank as described in 8.91-3. If he average area of the blank is greater than the half the value of the lowest standard used to construct the calibration curve, prepare a new blank solution.

Check blank (CCB): For TOC in water**, see Preparation blank**. For TOC in solids, analyze 70 ul of laboratory grade water. Analyze the CCB solution after each ICV/CCV solution. If the average area of the blank is greater than the half the value of the lowest standard used to construct the calibration curve, determine the source of the problem, fix the problem and reanalyze all samples since the last compliant CCB.

Standard Reference Material: Not applicable

Laboratory control sample: Not applicable

Sample duplicate: Prepare and analyze one sample duplicate for every 10th sample. For soil samples, refer to the latest control limit for duplicates. (The default limit is 25% RPD) If the the results for the sample and duplicate are unacceptable, a case narrative explaining why the RPD for a sample and its duplicate was outside the control limits must be written and approved by the quality assurance officer. A copy of the case narrative must be sent with the report to the client.

Triplicate Analysis: Each sample is analyzed in triplicate, if the percent Relative Standard Deviation exceeds 25%, an additional (Quadruplicate) analysis is performed . Results of all four analysis, the mean, and %RSD are reported to the client.

Matrix spike: Prepare and analyze one matrix spike for every 20th sample. **TOC**: For soil samples, refer to the latest control limit for matrix spikes (default recovery limits are 60% to 140%). Spile with an aliquot of the ICV/CCV solution. If the results for the matrix spike is unacceptable, prepare and analyze another matrix spike. If the results for the matrix spike is still unacceptable, a case narrative explaining why the percent recovery for the matrix spike was outside the control limits must be written and approved by the quality assurance officer. A copy of the case narrative must be sent with the report to the client

Serial dilution: Not applicable

Analytical spike: Not applicable

Method of standard additions: Not applicable

Overrange samples: Dilute or redigest samples that are greater than the value of the highest standard used to prepare the calibration curve so that the results are within the calibration curve.

ATTACHMENT C:
DISPOSAL OF SAMPLES AND WASTE

1. Refer to SOP NE054 for procedures for disposing of laboratory waste.

2. Acidified aqueous samples and extracts that do not contain metals or organic compounds above 0.050 mg/L, can be neutralized to a pH above 4.0 before disposal.

3. All client sample containers must be defaced with a permanent marker before disposal.

11 GLOSSARY

11.1 **Laboratory control.:** A standard of known concentration that is independent of the standards used for quantifying samples.

11.2 **Continuing calibration standard (CCV):** Used to assure calibration accuracy during each analysis run. It must be run at a frequency of 10% during the run. It must also be analyzed at the beginning and the end of the run. Its concentration must be at or near the mid-range level of the calibration curve.

11.3 **Correlation coefficient:** The correlation coefficient for the calibration curve must be greater than or equal to 0.997 according to NYSDOH requirements.

Appendix II

Vibracore Sediment Collection Procedures

Appendix II

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 $4^{o}C$);
- 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.

<u>Pre-Processing the Core Prior to Sample Extraction</u>

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

Appendix JJ

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 though 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, 208p.

Appendix KK

Sequential Batch Leach Test Procedures

Appendix KK

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

Appendix LL

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:

Specific Discharge = **__V** x **__t**
____
A

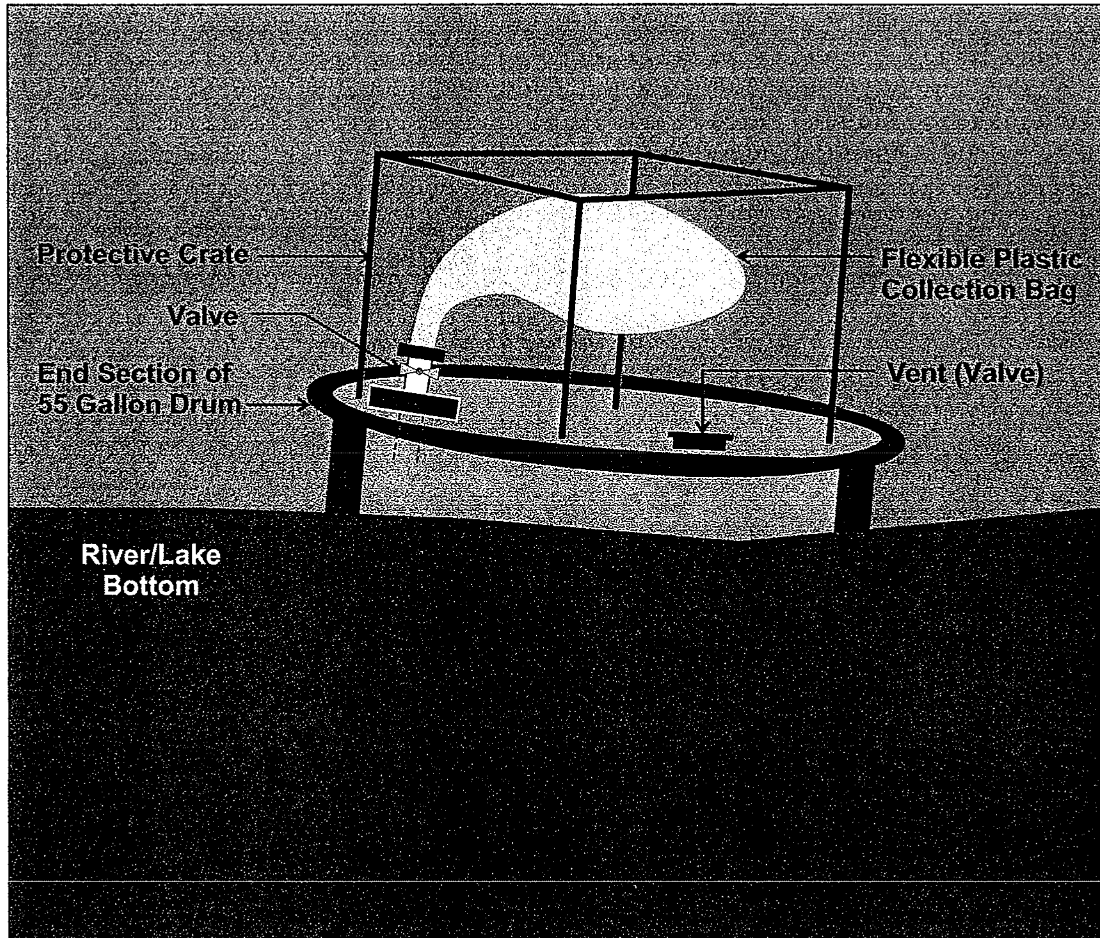
Where **___V** equals the measured change in volume of the water in the collection bag (in cubic centimeters);

___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.



GENERAL ELECTRIC COMPANY
PITTSFIELD, MASSACHUSETTS
FIELD SAMPLING PLAN/
QUALITY ASSURANCE PROJECT PLAN

TYPICAL SEEPAGE METER

BBL®
BLASLAND, BOUCK & LEE, INC.
engineers, scientists, economists

FIGURE
LL-1

06/14/04 SYR-D85-DJH
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