

*Field Sampling Plan/
Quality Assurance Project
Plan*

Volume II of III

General Electric Company
Pittsfield, Massachusetts

January 2000



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Appendix A

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Standard Operating Procedures for Development of Data Quality Objectives (DQOs)

Appendix A

Standard Operating Procedures for Development of Data Quality Objectives (DQOs)

I. Introduction

The procedures described herein provide a framework for the development of investigation or project-specific data quality objectives (DQOs). Project-specific DQOs are statements that specify the objective of the investigation. DQOs are developed to identify the appropriate type of data to collect and to determine the conditions from which to collect the data. DQOs also establish tolerable limits on decision errors that are used to determine the quantity and quality of data needed to support investigation related decisions.

The DQO development process is a strategic planning approach based on the scientific method that is used to prepare for a data collection activity. This process provides a systematic procedure for defining the criteria that a data collection design should satisfy, including when samples are collected, where samples are collected, the tolerable level of decision errors that are allowable, and the total number of samples to be collected.

II. DQO Development Process

In general, the DQO development process consists of several steps to be carried out to the extent necessary or appropriate. During the DQO development process, the planning team will develop the decision performance criteria, i.e., DQOs, that will be used to develop the data collection design. The final step of the process involves developing the data collection design based on the DQOs. A summary of the developmental steps to be considered may include:

- **Problem Definition:** Review prior studies and existing information to gain sufficient information to define the problem.
- **Determine Data Needs:** Identify the information that needs to be obtained and the measurements that need to be taken to provide needed data.
- **Define the Study Boundaries:** Determine where and when data should be collected.
- **Optimize the Design:** Evaluate multiple data needs and choose the most resource-effective sampling design that meets the various DQOs.
- **Establish the Tolerable Limits for Apparent Data Errors:** Specify the tolerable error limit of a specific data collection/analysis effort, if different than specified in Section 5 of the FSP/QAPP.

III. Site-Specific DQO Development

Project-specific DQOs for each field investigation will be developed and presented in each of the project-specific work plans for the areas in question. For many specific investigations at these sites, the data quality objectives will be sufficiently clear that not every step in the process described above will have to be documented in the project-specific work plans. The project-specific work plans will set forth the DQOs to the extent necessary to describe the purpose of the investigation and to identify the appropriate type, locations, and quality of data to be collected to meet that purpose and to support environmental decisions for the site.

Appendix B

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

Appendix B

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 $\mu\text{g/Kg}$, wet weight) and high-level (sample concentrations greater than 200 $\mu\text{g/Kg}$, wet weight) VOC analyses using field preservation techniques, the procedures to collect soil and sediment samples without field preservation, and the procedures for collection of soil and sediment samples using EnCore™, SoilCore™, or equivalent samplers. In order to minimize the handling of preservatives (i.e., sodium bisulfate or methanol) in the field, the preferred sample collection method for VOC analysis will employ the EnCore™, SoilCore™, or equivalent methods. The soil collection procedure to be utilized at each area will be presented in the project-specific work plan.

II. Materials

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

- Health and safety equipment (as required in the Health and Safety Plan);
- Photoionization detector (PID);
- Modified 10 - 30 mL plastic syringe;
 1. Cut off the injection tip,
 2. Depending upon the construction of the syringe, cut small air vents into the plunger or remove the rubber tip and retaining post.
- Stainless steel spatula;
- EnCore™ Sampler T-Handle;
- Resealable-type bags;
- Handiwipes or lint-free paper towels;
- Field notebook;
- Appropriate sample containers depending upon specific methodology (4-oz glass jar with Teflon lined cap, pre-preserved 40 mL vial with septum seal, EnCore™, SoilCore™, or equivalent Sampler); and
- Appropriate transport containers (coolers) with water ice and appropriate labeling, packing, and shipping materials.

III. Field Sampling

Option 1 - Collection of Unpreserved Sample

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

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

- Step 3 - Wipe the threads of the sample jar with a handwipe 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 M.

Option 2A - EnCore™ Sampler

- Step 1 - Place EnCore™ sample container into the EnCore™ T-handle.
- Step 2 - Collect soil sample by pressing the EnCore™ sample container into the soil to be collected.
- Step 3 - Wipe the outside of the EnCore™ sample container with a handwipe or lint free paper towel to ensure an adequate seal.
- Step 4 - Using the T-handle, cap and lock the EnCore™ Sampler for shipment.
- Step 5 - If low-level VOC analysis is to be performed, repeat Steps 1 through 3 two additional times to collect a total of 3 samples (high-level VOC analysis only requires 1 EnCore™ Sampler).
- Step 6 - Collect additional soil in a 4-oz (120 mL) jar for percent moisture determination.
- Step 7 - Place sample container in a transportation cooler on ice immediately after collection. Package and label the sample container following the procedures in Appendix M.

Option 2B - SoilCore™ Sampler

- Step 1 - Collect soil sample by pressing the SoilCore™ sample container into the soil to be collected. A decontaminated stainless steel spatula may be used to assist this procedure.
- Step 2 - Wipe the outside of the SoilCore™ sample container with a handwipe or lint free paper towel to ensure an adequate seal.
- Step 3 - Cap the filled end of the SoilCore™ sample container.
- Step 4 - Fill the opposite side of the SoilCore™ sample container by pushing the empty side of the SoilCore™ into the soil to be collected. A decontaminated stainless steel spatula may be used to assist this procedure.
- Step 5 - Wipe the outside of the SoilCore™ sample container with a handwipe or lint free paper towel to ensure an adequate seal.
- Step 6 - Cap the filled end of the SoilCore™ sample container.
- Step 7 - Place the locking strap around both endcaps and pull the strap tight to secure endcaps. Check the position of endcaps after tightening to verify that the SoilCore™ container is properly sealed.

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

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 M.

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 handwipe 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 handwipe 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 M and N. **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 X. 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 C

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

Appendix C

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

I. Introduction

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

II. Materials

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

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

III. Field Sampling Procedures

VPH

Option 1 - Field Preservation

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

- Step 2 - Prior to sample collection, weigh each sample container and compare the weight to the laboratory supplied tare weight to ensure that loss of preservative has not occurred. **Do not use sample containers that do not agree within ± 0.2 grams of the laboratory tare weight.**
- Step 3 - Soil samples must be collected in a manner that minimizes sample handling and agitation. This is accomplished by using a 30-mL plastic syringe with the end sliced off. The syringe is pushed into the soil sample collected via split-spoon methods until full.
- Step 4 - Extrude approximately 15 grams of soil into the pre-preserved sample vial.
- Step 5 - Wipe the threads of the sample vial with a handwipe 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 M and N. **Do not add tape, custody seals, or any other material to the pre-preserved sample containers that will alter the weight of the container.**

Option 2A - EnCore™ Sampler

- Step 1 - Place EnCore™ sample container into the EnCore™ T-handle.
- Step 2 - Collect soil sample by pressing the EnCore™ sample container into the soil to be collected.
- Step 3 - Wipe the outside of the EnCore™ sample container with a handwipe or lint free paper towel to ensure an adequate seal.
- Step 4 - Using the T-handle, cap and lock the EnCore™ Sampler for shipment.
- Step 5 - Collect additional soil in a 4-oz (120 mL) jar for percent moisture determination.
- Step 6 - Place sample containers in a transportation cooler on ice immediately after collection. Package and label the sample container following the procedures in Appendix M.

Option 2B - SoilCore™ Sampler

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

- Step 2 - Wipe the outside of the SoilCore™ sample container with a handwipe or lint free paper towel to ensure an adequate seal.
- Step 3 - Cap the filled end of the SoilCore™ sample container.
- Step 4 - Fill the opposite side of the SoilCore™ sample container by pushing the empty side of the SoilCore™ into the soil to be collected. A decontaminated stainless steel spatula may be used to assist this procedure.
- Step 5 - Wipe the outside of the SoilCore™ sample container with a handwipe or lint free paper towel to ensure an adequate seal.
- Step 6 - Cap the filled end of the SoilCore™ sample container.
- Step 7 - Place the locking strap around both endcaps and pull the strap tight to secure endcaps. Check the position of endcaps after tightening to verify that the SoilCore™ container is properly sealed.
- Step 8 - Collect additional soil in a 4-oz (120 mL) jar for percent moisture determination.

EPH

- Step 1 - Soil samples are collected directly from the split spoon using a stainless steel, decontaminated, spatula.
- Step 2 - Samples are collected in 4-oz (120 mL) wide-mouth glass jars with Teflon-lined screw caps.
- Step 3 - Wipe the threads of the sample jar with a handwipe 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 M.

IV. Field Cleaning Procedures

Cleaning of EPH/VPH sampling equipment (e.g., stainless-steel sampling tools) is to follow the procedures presented in Appendix X. 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 D

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Soil Boring Installation and Soil Sampling Procedures

Appendix D

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 VOC or EPH/VPH analyses described in Appendices B and C, respectively). Soil samples may be collected through a variety of mechanisms, including 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 a hand driven or a portable power auger depending on the required depth and subsurface material.

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. The sampling method employed will be ASTM D-1588 - standard method for penetration test and split-barrel sampling of soils (Annual Book of ASTM Standards, Volume 04.08).

Personnel will also be responsible for documenting drilling events in the field log notebook. 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).

II. Equipment and Materials

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

- Health and safety equipment (as required in the Health and Safety Plan);
- Cleaning equipment (as required in Appendix X);
- All drilling equipment required by ASTM D-1586;
- Appropriate sample containers and forms;
- Coolers with ice or "blue ice";
- Photoionization detector (PID); and
- Field Notebook.

III. Soil Boring Installation

In conjunction with the use of hollow stem augers or driven casing methodologies, soil cores will be collected using standard 2-inch by 2-foot split spoons driven by a 140-lb hammer, or Standard Shelby tubes, unless otherwise specified. The split spoons or Shelby tubes will be advanced to the depth specified in the project-specific work plan. The sampling method employed will be the American Society of Testing and Materials (ASTM) D1586 - *Standard Method for Penetration Test and Split-Barrel Sampling of Soils*. Specific procedures for the installation of soil borings using hollow stem augers and driven casing methodologies are presented in Appendix T. Soil samples will be collected following the procedures described in the following sections, and those presented in Appendices B and

C for volatile organic compounds (VOCs) and volatile petroleum hydrocarbons (VPH)/extractable petroleum hydrocarbons (EPH), respectively.

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/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. Specific procedures for the installation of soils borings by direct-push drilling are presented in Appendix T. Soil samples will be collected following the procedures described in the following sections, and those presented in Appendices B and C for VOCs and VPH/EPH, respectively.

IV. Subsurface Soil Sampling Procedures

Step 1 - As samples are collected, qualified personnel will describe each soil sample for the following parameters (a sample of which is provided in Attachment D-1).

- soil type;
- color;
- percent recovery;
- moisture content;
- texture;
- grain size and shape;
- consistency; and
- miscellaneous observations

Step 2 - Samples are placed in appropriate sample containers for PID field screening as specified in Appendix O.

Step 3 - PID screening (Appendix O) may be performed to determine which samples will require analysis for volatile organic compounds and in some cases to determine which sample will receive analysis for Appendix IX+3 constituents. This determination, when applicable, will be made in the following manner:

- When the project-specific work plan specifies that PID screening be performed for VOC sample selection, a sample which exhibits a reading of 10 PID units or higher will be collected as specified in Appendix B and analyzed for VOCs by EPA Method 8260B (unless otherwise specified in the work plan).
- When the project-specific work plan specifies that PID screenings be performed for the purpose of selecting samples for analysis for Appendix IX+3 constituents, 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 analysis.

- When using PID screening to determine Appendix IX+3 sample location, VOC samples may be collected using either of the two following procedures:

Option 1 - The VOC samples are collected from each depth interval at the same time that the PID screening samples are collected. After completion of the soil boring, the sample exhibiting the highest PID reading is sent to the laboratory for analysis and the remaining samples are 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 Appendix IX+3 sample. After screening is completed and the Appendix IX+3 sample interval has been determined, the Appendix IX+3 sample (including a VOC sample) can be collected from the appropriate depth interval 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 M.

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 is expected to be utilized for a number of future soil investigations related to the implementation of the agreement in principle (e.g., sampling of 1- to 6-foot and 6- to 15-foot depth increments). 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 with other samples from the specified composite depth interval. Using this process, two composite samples will be obtained -- one representing the 1- to 6-foot depth increment, and the other

representing the 6- to 15-foot depth increment. Both of these composite samples will be analyzed for PCBs.

Step 3 - In addition to analysis for PCBs, a sample from one of the two composite samples may be submitted for Appendix IX+3 analyses, excluding VOCs. The composite sample to be submitted will be that sample corresponding to the highest PID reading from the individual sample depth increments.

Step 4 - The discrete sample depth interval with the highest PID reading will be analyzed for Appendix IX+3 VOCs.

VI. Surficial Soil Sampling

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

Health and safety equipment (as required by the Health and Safety Plan);

- Cleaning equipment (as required in Appendix X);
- 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 which 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 screening is being performed for selection of Appendix IX+3 samples, obtain a split sample for screening with a PID for volatile organic compounds using the procedures set forth in Appendix O and take a PID reading as soon as possible after sample preparation.

Step 4 - Label all sample container 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 M.

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 compositing soil collected at the same time and depth and then filling two sets of sample jars. For VOCs, the samples will not be composited 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.

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 X. The sampling equipment is to be cleaned prior to the start of sampling activities, between samples, and following the completion of sampling activities.

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 on-going disposal practices.

Attachment D-1

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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 E

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Groundwater Purging and Sampling Procedures for Monitoring Wells

Appendix E

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 USEPA 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 E-1). 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 T, 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.

II. Materials

Specific to this activity, the following materials shall be available:

- Site plan, well construction records, prior groundwater sampling records (if available);
- Peristaltic, Rediflow, or equivalent pump and dedicated Teflon® tubing;
- Water level probe;
- Conductivity/temperature meter;
- pH meter;
- Turbidity meter;
- Appropriate water sample containers;
- Appropriate blanks (trip blank supplied by the laboratory);
- 0.45-micron disposable filters;
- Large glass mixing container; and
- Teflon stirring rod.

III. Procedures

After groundwater elevation has been measured, groundwater will be purged from the wells using a low-flow submersible or peristaltic pump. Purge water will be collected and containerized.

- Step 1 - If a submersible pump is being utilized, slowly lower pump, safety cable, tubing, and electrical lines into the well to a depth corresponding to the approximate center of the saturated screen section of the well. If a peristaltic pump is being utilized, slowly lower the sampling tubing into the well to a depth corresponding to the approximate center of the saturated screen section of the well. The pump intake or sampling tube must be kept at least two feet above the bottom of the well to prevent mobilization of any sediment present in the bottom of the well.

- Step 2 - Measure the water level again with the pump in the well before starting the pump. Start pumping the well at 200 to 500 milliliters 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) and the water level should stabilize. The water level should be monitored every three to five 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.

- Step 3 - During purging, monitor the field indicator parameters (e.g., turbidity, temperature, specific conductance, pH, etc.) every three to five minutes (or as appropriate). Field indicator parameters will be measured using a flow-through analytical cell or a clean container such as a glass beaker. Record field indicator parameters on the groundwater sampling log (Attachment E-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 specific conductance and temperature values remain within 3%, 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.

- Step 4 - Complete the sample label according to procedures in Appendix M and cover the label with clear packing tape to secure the label onto the container.

- Step 5 - After the indicator parameters have stabilized, collect groundwater sample by diverting flow out of the unfiltered 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.
- Step 6 - If sampling for total and filtered metals and/or PCBs, a filtered and unfiltered sample will be collected. 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 100-300 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.
- Step 7 - Secure with packing material and store at 4°C in an insulated transport container provided by the laboratory.
- Step 8 - Record on the field log (Attachment E-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 stabilized field indicator parameters, as measured during the final reading during purging.
- Step 9 - Remove pump and tubing from well, secure well, properly dispose of PPE and disposable equipment (see Section VI).
- Step 10 - If tubing is to be dedicated to a well, it should be folded to a length which 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.
- Step 11 - Complete the procedures for packaging, shipping, and handling with associated chain-of-custody (Appendix M).
- Step 12 - Complete cleaning procedures for flow-through analytical cell and submersible pump, as appropriate (see Section V - Equipment Cleaning).

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

- Step 1 - Don appropriate personal protective equipment (as required by the Health and Safety Plan).
- Step 2 - Place plastic sheeting around the well.
- Step 3 - Clean the sampling equipment with the procedures in Appendix X.

- 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 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 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)**
- Step 6 - 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.
- Step 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.
- Step 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. Record field indicator parameters on the groundwater sampling log (Attachment E-2).
- Step 9 - Repeat Step 7 and Step 8 until 3 or 4 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 specific conductance and temperature values remain within 3%, and pH remains within ± 0.1 units for three consecutive readings collected once per well volume removed.
- Step 10 - If the field indicator parameters have not stabilized, remove a maximum of 5 well volumes prior to sample collection. Alternatively, five well volumes may be removed without measurement of the field indicator parameters.
- Step 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.

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

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

Step 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 sediments in the well.

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

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

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

Step 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.

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

Step 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.

Step 20 - If sampling for total and filtered metals and/or PCBs, a filtered and unfiltered sample will be collected. Sample filtration for the filtered sample will be performed in the field utilizing a peristaltic pump prior to preservation. Install new medical-grade silicone tubing in the pump head. Place new Teflon tubing into the sample mixing container and attach to the intake side of pump tubing. Attach (clamp) a new 0.45 micron filter (note the filter flow direction). Turn the pump on and dispense the filtered liquid directly into the laboratory sample bottles.

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

Step 22 - After sample containers have been filled, remove an additional volume of groundwater. Measure the pH, temperature, turbidity, and conductivity. Record on the field log (Attachment E-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.

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

Step 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.

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

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 manufacturers instructions. 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 X.

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 E-1

BLASLAND, BOUCK & LEE, INC.
engineers & scientists

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

**U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I**

BLASLAND, BOUCK & LEE
LIBRARY REFERENCE MATERIAL

**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) permeability 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)

Page of [illegible]

1. Pump dial setting (for example: hertz, cycles/min, etc.).
2. μ Siemens per cm (same as μ mhos/cm) at 25 °C.
3. Oxidation reduction potential (stand in for Eh).

Attachment E-2

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Low-Flow Groundwater Sampling Log

Site Name _____
 Sampling Personnel _____
 Date _____ Time In / Out _____
 Weather _____

Pump Start Time _____
Pump Stop Time _____
Sample Time _____
Sample ID _____
Sampled for:
() VOCs
() SVOCs
() PCBs (Total)
() PCBs (Dissolved)
() Metals (Total)
() Metals (Dissolved)
() Other (Specify) _____

	TIC	TOC	BGL
Reference Point Marked on Casing			
Height of Ref. Pt. Relative to Grade			
Well Diameter			
Well Depth			
Screen Interval Depth			
Water Table Depth			
Intake Depth of pump/tubing			

WELL WATER INFORMATION

Length of Water Column	
Volume of Water in Well	
Minutes of Pumping	

Evacuation Method: Bailer () Pump ()

Pump Type:

Water Quality Meter Type(s) / Serial Numbers:

[illegible]**MISCELLANEOUS OBSERVATIONS/PROBLEMS**

Laboratory: _____
Delivered Via: _____
Airbill #: _____

Field Sampling Coordinator: _____

Attachment E-3

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Representative Sample Label



PROJECT #

SAMPLE I.D.

DATE

SAMPLE TYPE

☐ Soil/Sediment

☐ Water

COLLECTION MODE

☐ Composite

☐ Grab

TIME

ANALYSIS

SAMPLER(S)

PRESERVATIVE

Appendix F

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Surface Water Sampling Procedures

Appendix F

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 X);
- 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;
- Teflon tubing;
- Teflon 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 “blue” ice; and
- Appropriate water sampler as specified in the project-specific work plan, which may include following:
 - Surface water grab sampler (Attachment F-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 Teflon tubing; or
 - Kemmerer stainless steel bottle sampler (Attachment F-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 F-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 X;
- Step 4 - Assemble the water sampler (Attachment F-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 teflon stirring rod and transfer the appropriate volume into the laboratory sample jar. (Volatile samples will not be homogenized.);
- Step 11 - When sampling for volatiles, surface water samples will be collected directly from the water sampler to 40 ml vials with teflon liners;
- Step 12 - The sample collection order (as appropriate) will be as follows:

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

- Step 13 - If sampling for total and filtered metals, a filtered and unfiltered sample will be collected. Sample filtration for the filtered sample will be performed in the field utilizing a peristaltic pump prior to preservation. Install new medical-grade silicone tubing in the pump head. Place new teflon tubing into the sample mixing container and attach to the intake side of pump tubing. Attach (clamp) a new 0.45 micron filter to the discharge side of the pump tubing (noting the correct filter flow direction). Turn the pump on and dispense the filtered liquid directly into the laboratory sample bottles.
- 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 M;
- 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 P.
- 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 M.
- B. To obtain surface water samples at depth from lakes (including Silver Lake), ponds, and impoundments, a peristaltic pump (Attachment F-4) will be used, and the following procedures will be followed:
- Step 1 - Identify sampling location on appropriate sampling log sheet (Attachment F-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 X;
- 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 teflon tubing necessary to reach the required sample depth and attach to intake side of pump tubing. Taping the teflon 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 F-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 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/stream.
- 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 "velocity-area method"). The total flow rate is the sum of the flow of the partial sections.

$$Q_T = V_1 A_1 + V_2 A_2 + \dots + V_n A_n$$

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

Field duplicates will be prepared by filling a composite container with water collected at the same time and depth and then two sets of sample jars. For VOCs, the samples will not be composited prior to placement in the sample jars. The duplicate sample will be labeled 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 X.

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 F-1

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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 Solids _____
Odor _____	Film _____

IV.	Container	Analysis

V. Surface Water Characteristics

Color _____	Suspended Solids _____
Temperature _____	Film _____
	Odor _____

Attachment F-2

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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 _____

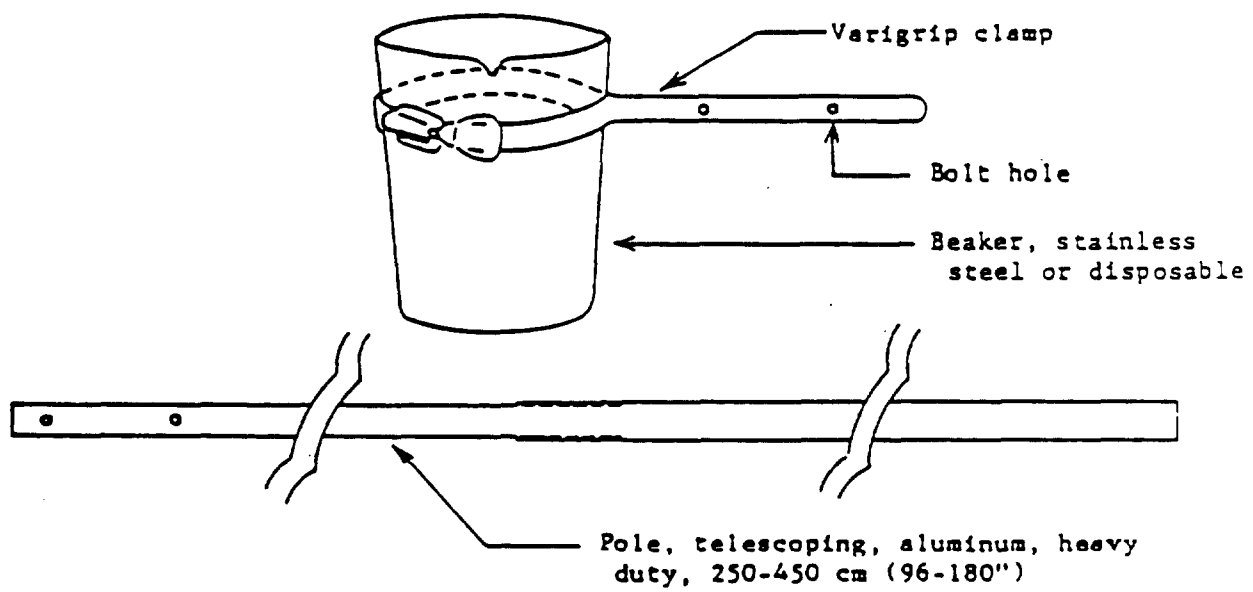
Sample ID	Date	Time	Sample Depth (feet off bottom)	Analysis

Comments _____

Attachment F-3

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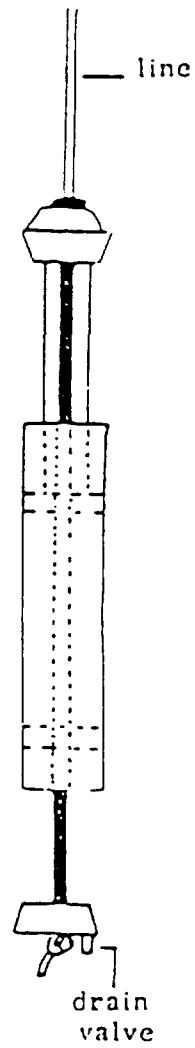
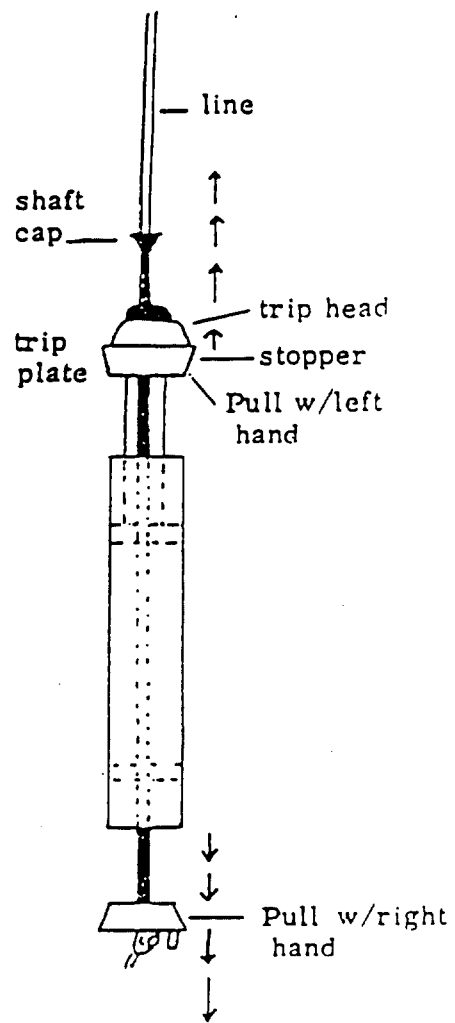
Surface Water Grab Sample



Attachment F-4

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Kemmerer Bottle



Appendix G

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Sediment Sampling Procedures

Appendix G

Sediment Sampling Procedures

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 G-1, G-2, and G-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);
- Boat;
- Flagging;
- Field book;
- 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 (as required by the Health and Safety Plan);
- 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 then 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; and

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 Health and Safety Plan);
- Cleaning equipment (as required in Appendix X);
- 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;
- Endcap 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 G-4) and/or field notebook along with other appropriate information collected during sediment sampling activities.

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

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 endcap 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 and 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 project-specific 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 B.
- 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 X) between each cut.
- Step 22 - Prepare equipment blank samples at the frequency specified in Table 4 of the 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 M.
- 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 X. Equipment cleaning of any sampling equipment which is re-used at another sample location, will be performed as described in Appendix X.

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 G-1

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Sediment Harvesting Procedures

Attachment G-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;
- Cleaning equipment;
- Surface water composite sample (5 gallons) collected at mid-depth and mid-channel utilizing a peristaltic pump and the procedures in Appendix F;
- 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 1-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 1-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 wrapped filter for shipment in cooler to the laboratory in accordance with procedures in Appendix M.

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

Attachment G-2

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Sediment Trap Procedures

Attachment G-2

Sediment Trap Procedures

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

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);
- 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.

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 tope from the sediment trap to the buoy.

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 M.
- 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 M.

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 one foot off the bottom.

Attachment G-3

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Settleability Testing

SETTLING TEST PROCEDURES*

PART I: TESTING EQUIPMENT AND PROCEDURES

Test Objective

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

Test Equipment

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

Samples

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

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

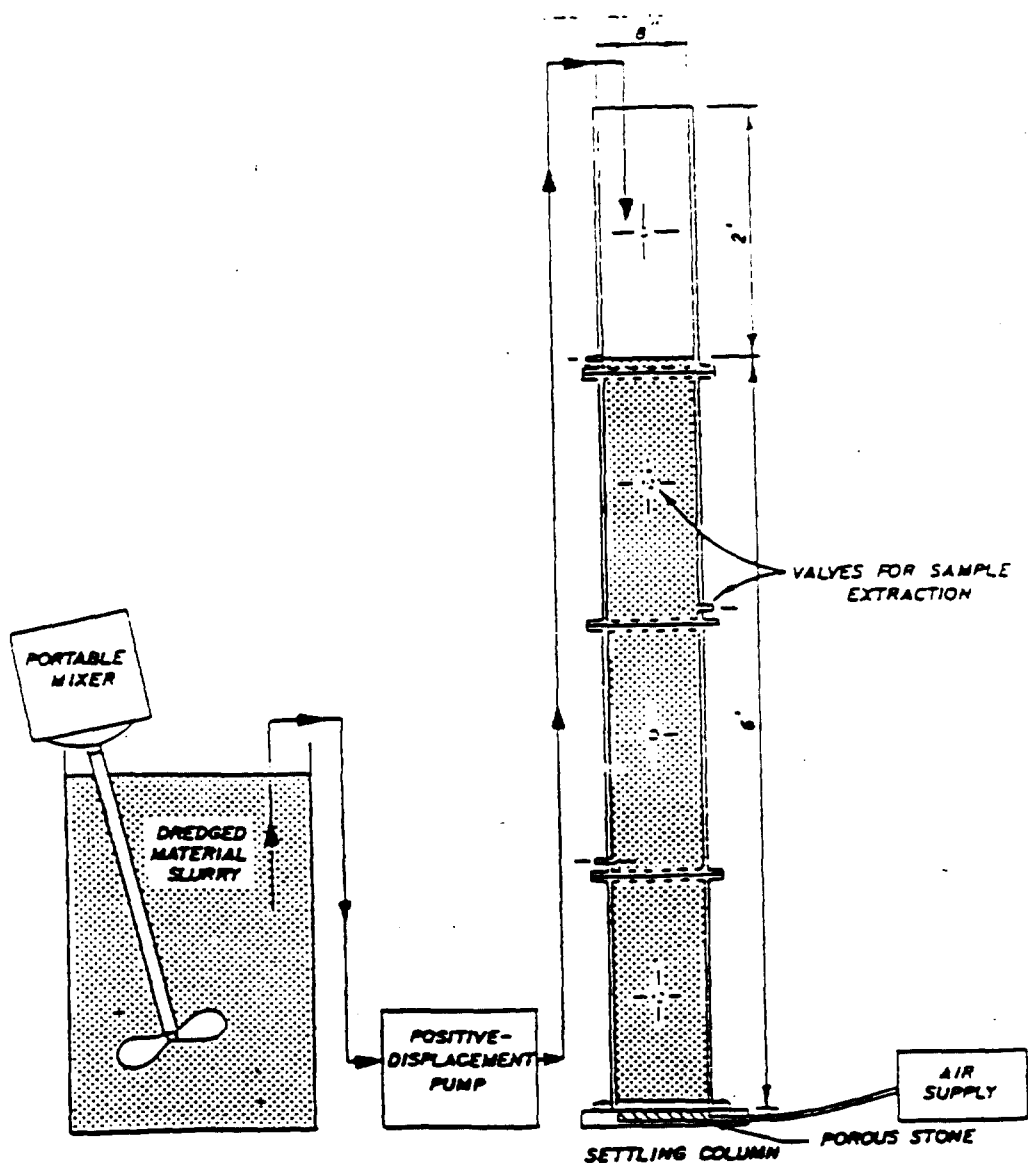
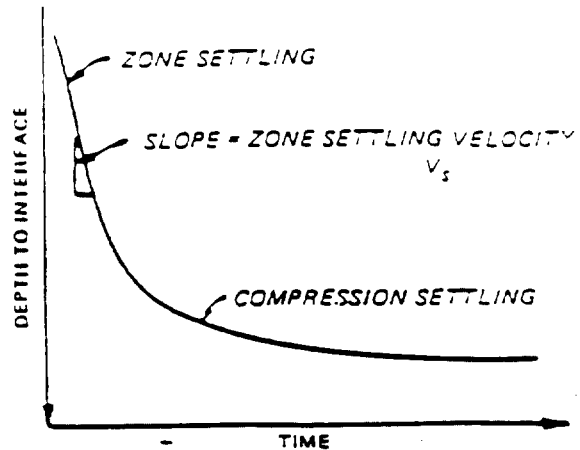


Figure 1. Schematic diagram of apparatus for settling tests

Figure A1. Conceptual plot of interface height versus time



Test Procedures

Pilot test

4. A pilot test conducted in a small graduated cylinder (12 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 compression settling.

Required number of
column loadings for tests

7. Three types of settling tests may be needed to fully define the settling properties of the dredged material. However, in many cases the 8-in. settling column used for the settling tests need only be loaded with slurry once. A compression settling test is needed to define the volume which will be occupied in the disposal area by a newly deposited dredged material layer. Also, a flocculent settling test for either the slurry mass or for the supernatant water above any interface is required to predict effluent suspended solids concentrations. Both of these tests should be conducted at a slurry concentration equal to the expected influent concentration. Therefore, only one loading of the test column would be required to collect data for both purposes. A series of zone settling tests is required to define the minimum required surface area needed for effective zone settling. For the zone settling 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 agitating 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_0 . 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

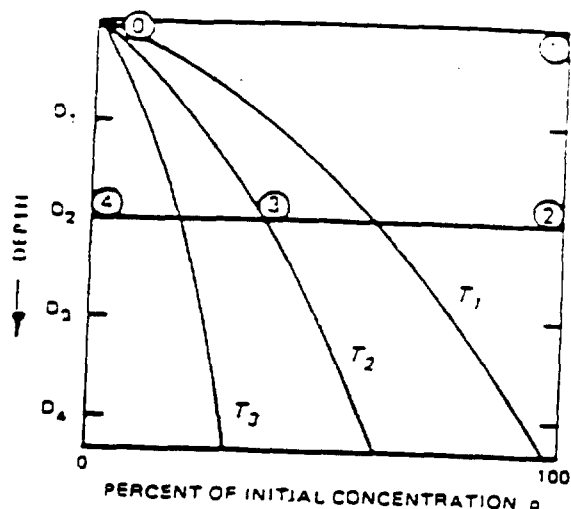


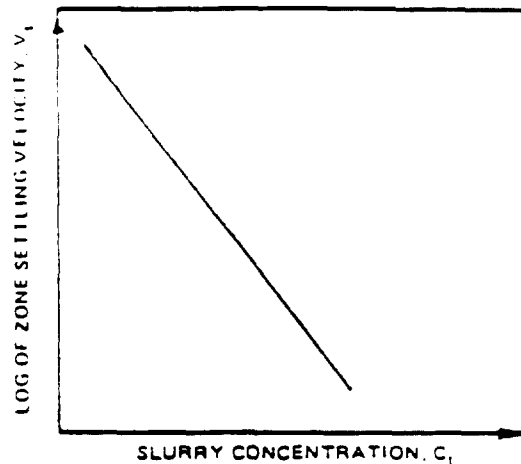
Figure A2. Conceptual concentration profile diagram

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.

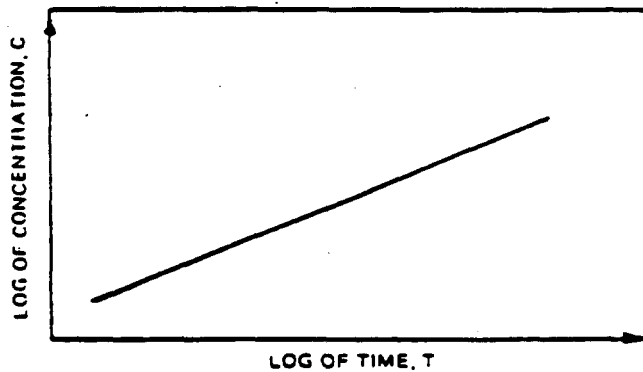


Figure A4. Conceptual time-versus-concentration plot

Attachment G-4

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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

<u>Section Number</u>	<u>Representative Length of Section</u>	<u>Actual Length of Section</u>
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

IV. Sediment Characteristics

Color _____ Material _____
Grain Size _____ Odor _____

Remarks:

Appendix H

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

Appendix H

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;
- Health and safety equipment (as required in the Health and Safety Plan);
- Cleaning Equipment (as required in Appendix X);
- Plastic sheeting;
- Field book or appropriate log forms;
- Absorbent pads;
- Peristaltic pump and pump tubing or bailer (stainless steel or Teflon®);
- 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 forms;
- Large heavy duty garbage bags;
- Teflon® 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 R.
- 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 H-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 Teflon® or stainless steel bailer decontaminated in accordance with Appendix X. Alternatively, it may be removed utilizing a peristaltic pump with new Teflon® 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 Teflon® tubing or a Teflon® 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 Teflon® 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 X 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 M. LNAPL/DNAPL may require additional packaging and labeling procedures as specified in Appendix N.

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 H-1

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LNAPL/DNAPL Sampling Field Log

Example

LNAPL/DNAPL Sampling Field Log

Project: _____
Site _____
Name: _____

Project No.: _____
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 -

DNAPL Thickness -

Water Thickness -

Vol. LNAPL Removed _____

Vol. LNAPL Removed _____

II. WELL SAMPLING

Lab Sample No.

Time Sampled

Material Sampled

III. MISC. OBSERVATIONS

Appendix I

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Biota Sampling Procedures

Appendix I

Biota Sampling Procedures

I. Introduction

This appendix specifies the procedures for certain types of biota sampling that may be conducted in Massachusetts. These include procedures for the collection and processing of samples of aquatic vertebrates (fish and frogs) for chemical analysis of their tissue, as well as procedures for aging fish (if appropriate). For other types of biota sampling (if appropriate), the sampling and analysis procedures will be presented in the work plan providing for such sampling.

The procedures for collection and analysis of biota samples from the Connecticut portion of the Housatonic River are presented in Attachment I-2.

II. Sampling of Aquatic Vertebrates (Fish and Frogs) 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.

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);
- 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.

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

Step 3 - Bullfrogs will be collected using angling methods.

Step 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 log book:
 - 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 otherwise provided in a project-specific work plan.
- d. Wrap each 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 M.

C. Processing

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

Step 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.

Step 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 forms for specific parameters to be run.

Step 3 - Processing the specimen samples

- a. All samples will be prepared as one of the following sample 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 side 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 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; and
 - 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

- Step 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 a forceps.
- Step 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.
- Step 3 - Each sample envelope will be labeled with the collection date and sample number.
- Step 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

- Step 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.
- Step 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.
- Step 3 - Regenerated scales should not be mounted.
- Step 4 - Scale impressions can be made by 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

- Step 1 - Scales should be aged using a microprojector, with the sculpted surface of the scale or impression facing the light source.
- Step 2 - Regenerated scales should not be aged.
- Step 3 - Each age should be based upon the reading of at least two scales, both aged by independent observers, and a consensus reach by consultation if different results occur.
- Step 4 - Age should be reported as the number of annuli in Roman numerals.

IV. Sample Analysis

Samples of the biota collected may be analyzed for Aroclor-specific polychlorinated biphenyls (PCBs), and/or polychlorinated dibenzo-p-dioxins (PCDDs)/polychlorinated dibenzofurans (PCDFs) following the methods presented in Table 1. Biota samples may also be analyzed for lipid content and/or congener-specific PCBs following the procedures presented in Attachment I-1. The protocols for additional parameters (if proposed) will be presented in the applicable work plan.

Attachment I-1

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Extraction of Lipids

Extraction and Cleanup of PCBs

Congener - Specific PCB Analysis - Method NEA-608CAP

STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

**NE158_01.SOP
REVISION NUMBER: 01**

**STANDARD OPERATING PROCEDURE FOR THE
EXTRACTION OF LIPIDS FROM FISH AND BIOTA MATERIAL**

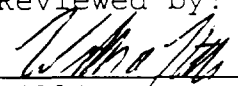
MAY 20, 1998

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STANDARD OPERATING PROCEDURE

Author: Dean J. Morrissey

Reviewed by:



William A. Kotas

Northeast Analytical, Inc.

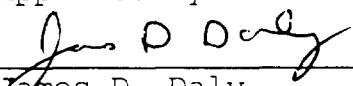
Issuing Section: Organics

SOP Name: NE158_1.SOP

Date: 20-May-1998

Revision: 01

Approved by:



James D. Daly
Assistant Laboratory Director

1.0 TITLE

Standard Operating Procedure for the extraction of lipids from fish and biota material.

2.0 PURPOSE

This SOP provides the method for the extraction of lipids from fish tissue.

3.0 SCOPE

The following procedure is used by NEA for the extraction of fish tissue for lipid analysis, though it may in part be adapted for other biota extractions.

4.0 COMMENTS

This method may be restricted to use by or under the supervision of a chemist knowledgeable in the area of sample extraction and clean-up. The chemist should further be aware of the proper care and handling of Polychlorinated Biphenyls (PCBs) as well.

5.0 SAFETY

Polychlorinated biphenyls should be treated with extreme caution; as a class of chemical compounds they possess both toxic and carcinogenic properties. 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.

NORTHEAST ANALYTICAL, INC
STANDARD OPERATING PROCEDURES

SOP Name: NE158_01.SOP

Revision: 1

Date: 05/20/98

Page: 1 of 9

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, 2500A. The chemist must also be certified to perform the procedure by an approved instructor.

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 Analytical Balance: Mettler ER-180A used to determine sample mass.
- 7.0.7 Cellulose Extraction Thimble: Contains sample during soxhlet extraction.
- 7.0.8 Sodium Sulfate: J.T.Baker #3375-05 Anhydrous, Granular (12-60 Mesh). Washed with dichloromethane and baked overnight at 200°C. Used for the laboratory method blank.
- 7.0.9 Boiling Chips: Hengar #5785 Alltech Associates, Inc. (or equivalent)
- 7.0.10 Chiller: Pump driven water circulating cooling system cool flow #75 NESLABS Instruments, Inc. (or equivalent)
- 7.0.11 Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208. (or equivalent)
- 7.0.12 Acetone: High Purity Solvent Baxter (Burdick/Jackson) #UN1090. (or equivalent)
- 7.0.13 Turbo Vap Evaporator: Zymark #ZW640-3.
- 7.0.14 Turbo Vap Evaporator concentrator tubes: Zymark 250mL, 0.5mL endpoint.
- 7.0.15 Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL, used for liquid containment and pipet storage.
- 7.0.16 Silanized Glass Wool: Supelco, Ic. #2-0410 solvent washed 3 times with hexane, baked at 130°C for >16 hours. Used to contain sample within the thimble during extraction.
- 7.0.17 1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.
- 7.0.18 Vials: glass, 8 dram & 4 dram (with polyseal sealed cap) (20 mL & 10 mL) capacity, for sample extracts.
- 7.0.19 Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.

NORTHEAST ANALYTICAL, INC

STANDARD OPERATING PROCEDURES

SOP Name: NE158_01.SOP

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- 7.0.20 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)
- 7.0.21 Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL.
- 7.0.22 Weighing Boats: Tin weighing boats for percent lipid weight determination.

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 on the sample tracking sheet.
- 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 The fish samples are usually received as fillets and must be processed to produce a homogenous material prior to extraction. Once the sample has been processed, the sample is cut up into small enough pieces to fit into a standard manual meat grinder. An acetone rinsed Pyrex tray should be placed under the grinder exit to catch the minced tissue. After the fish has been ground once, it should be ground again to ensure complete homogenization of the sample. The ground fish is then placed into an appropriate-sized jar and labeled.
The sample is then placed in the freezer for storage until the extraction process is begun.

8.2 Procedure: Sample Extraction

- 8.2.1 Rinse the thimbles with hexane, allow them to dry out in a pre-cleaned 100mL beaker in a fume hood.

- 8.2.2 Fill a pyrex pan with ice cubes and cold water about 1/2 inch deep. As the samples are weighed out, place the beakers in the pyrex pan to chill for at least 15 minutes prior to the drying step.
- 8.2.3 Into a pre-cleaned and tared 300mL beaker accurately weigh to the nearest 0.0001g using an analytical balance about 10 grams of tissue. Record this weight in the laboratory sample log book. Place the beaker containing the sample into the pyrex pan to chill. Repeat for remaining samples.
- 8.2.4 After the sample has been sufficiently chilled, add approximately 10 g. of a 1:1 mix of magnesium sulfate/sodium sulfate to the sample and mix well with a clean wooden tongue depressor. If the sample has not dried after a few minutes, another 10 g. may be added. Once the sample is well-dried and free flowing, transfer the sample to an extraction thimble. Add a plug of glass wool to the thimble and set aside. Repeat with remaining samples. Set empty mixing beaker and stirring utensil aside for later rinsing into soxhlet extractor to complete sample transfer. **Be careful not to add too much drying agent to the sample; if too much is added, the sample may not fit completely in the thimble. In this case the sample will have to be split into two different soxhlets.**
- 8.2.5 Add 200 mL of a 1:1 mixture of hexane/acetone to a 250 mL round bottom flask. Add several boiling chips. Place a soxhlet extractor on top of the round bottom flask. Label the round bottom with a sample number and use a pair of long-handled tweezers to place the corresponding thimble into the soxhlet extractor. Rinse corresponding beaker & tongue depressor with a few pipets of hexane. Transfer into thimble. Repeat this step twice more with the same sample, and then repeat all preceding steps with remaining samples. After all samples have been processed add the specified surrogate and matrix spikes required directly into thimble.
- 8.2.6 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.
- 8.2.7 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 once every two to three minutes should be achieved.

- 8.2.8 The samples should be extracted overnight for a minimum of 16 hours. Once the sample has finished extracting (usually in the morning), turn the heating mantle off and allow samples to cool to room temperature. Once cool, rinse the inside of the condenser with several pipette volumes of hexane. Disengage the soxhlet and condenser unit and rinse the joint off as well into the soxhlet.
- 8.2.9 Move soxhlet units into a chemical fume hood and flush the remaining solvent from the soxhlet extractor by tipping the soxhlet. Using a pair of long-handled tweezers, pull the thimbles out of the soxhlets one at a time and allow them to drip dry by balancing the thimbles on the tops of the soxhlets. Once dry, remove the thimbles to a pyrex drying pan for total solvent evaporation.
- 8.2.10 Rinse the soxhlet with several pipetfuls of hexane and tip again to drain into the round bottom. Set aside the soxhlet at this time. Procure the same number of turbo tubes as there are samples. Using an individual turbotube stand, label a turbotube with the corresponding sample ID number and place in the holder. Pour the contents of the round bottom into the turbotube, using a pipette and hexane to rinse the last drop out of the mouth of the round bottom. 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.
- 8.2.11 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. The temperature of the unit should be set at $38 \pm 2^{\circ}\text{C}$.
- 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 .
- 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/acetone) reduction takes approximately 20-30 minutes. Do not leave the unit unattended as extracts may be 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 acetone component is fractionally removed at a faster rate than hexane; however, a solvent exchange with hexane should be completed 3x adding approximately 2-4 mL of hexane each

time to ensure the acetone has been entirely removed.
NOTE: Not all samples will evaporate at the same rate; sample extracts containing large amounts of lipids or other non-volatile liquids may stop reducing before the 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 (25mL for biota extracts) with three 2mL hexane rinses. 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-labelled 8 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 concentration

NOTE NO CLEAN-UP STEPS ARE PERFORMED FOR PERCENT LIPID DETERMINATION.

- 8.4.1 Weigh a pre-labelled tin weighing boat for each sample extract. Record the weight in the percent lipid log book. Place the weighing boat into the desicator for 1 hour.
- 8.4.2 Using a 10mL pipet, transfer 10mL of the extract into the corresponding weighing boat.
- 8.4.3 Concentrate the extract in the weighing boat using the nitrogen micro-blowdown. Place a maximum of 3 weighing boats at one time on the nitrogen blow down. Set the temperature at 40C and the pressure at 40 PSI.
- 8.4.4 After concentration has been completed, place the weighing boats into the desicator for 1 hour.
- 8.4.5 Weigh sample weighing boats and record the weight in the percent lipid log book.

8.4.6 Calculate percent lipids as follows:

$$\text{PERCENT LIPIDS} = \frac{V_f - V_e \times 2.5 \times 100}{\text{sample weight (g)}}$$

V_f = weight of tin tray with sample(g)

V_e = weight of empty tin tray(g)

10.0 References

1. SW-846 methods 3500A & 3600A; United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Vol.1B, Cincinnati, OH 45268.
2. Guide to Environmental Analytical Methods, Genium Publishing Corporation, Schenectady, NY 12304.

11.0 Attachments

1. Note page for analyst

Note page for analyst

STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

**NE017_03.SOP
REVISION NUMBER: 03**

**STANDARD OPERATING PROCEDURE FOR THE
EXTRACTION AND CLEANUP OF POLYCHLORINATED
BIPHENYLS FROM FISH AND BIOTA MATERIAL**

JANUARY 29, 1998

COPY # (UNCONTROLLED)

STANDARD OPERATING PROCEDURE

Author: Dean J. Morrissey

Reviewed by:

 QA OFFICER
William A. Kotas

Northeast Analytical, Inc.

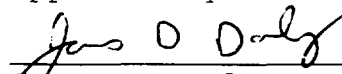
Issuing Section: Organics

SOP Name: NE017_03.SOP

Date: 29-JANUARY-1998

Revision: 03

Approved by:


James D. Daly
Assistant Laboratory Director

1.0 TITLE

Standard Operating Procedure for the extraction and cleanup of polychlorinated biphenyls from fish and biota material.

2.0 PURPOSE

This SOP provides the method for the extraction of polychlorinated biphenyls from fish tissue.

3.0 SCOPE

The following procedure is used by NEA for the extraction of fish tissue for PCB analysis, though it may in part be adapted for other biota extractions.

4.0 COMMENTS

This method may be restricted to use by or under the supervision of an extraction chemist knowledgeable in the area of extraction and clean-up. The chemist should further be aware of the proper care and handling of PCBs as well.

5.0 SAFETY

Polychlorinated biphenyls should be treated with extreme caution; as a class of chemical compounds they possess both toxic and carcinogenic properties. 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.

NORTHEAST ANALYTICAL, INC

STANDARD OPERATING PROCEDURES

SOP Name: NE017_03.SOP

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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, 2500A. The chemist must also be certified to perform the procedure by an approved instructor.

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 Analytical Balance: Mettler ER-180A used to determine sample mass.
- 7.0.7 Cellulose Extraction Thimble: Contains sample during soxhlet extraction.
- 7.0.8 Sodium Sulfate: J.T.Baker #3375-05 Anhydrous, Granular (12-60 Mesh). Washed with dichloromethane and baked overnight at 200°C. Used for the laboratory method blank.
- 7.0.9 Boiling Chips: Hengar #5785 Alltech Associates, Inc. (or equivalent)
- 7.0.10 Chiller: Pump driven water circulating cooling system cool flow #75 NESLABS Instruments, Inc. (or equivalent)
- 7.0.11 Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208. (or equivalent)
- 7.0.12 Acetone: High Purity Solvent Baxter (Burdick/Jackson) #UN1090. (or equivalent)
- 7.0.13 Turbo Vap Evaporator: Zymark #ZW640-3.
- 7.0.14 Turbo Vap Evaporator concentrator tubes: Zymark 250mL, 0.5mL endpoint.
- 7.0.15 Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL, used for liquid containment and pipet storage.
- 7.0.16 Silanized Glass Wool: Supelco, Ic. #2-0410 solvent washed 3 times with hexane, baked at 130°C for >16 hours. Used to contain sample within the thimble during extraction.
- 7.0.17 1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.
- 7.0.18 Vials: glass, 8 dram & 4 dram (with polyseal sealed cap) (20 mL & 10 mL) capacity, for sample extracts.
- 7.0.19 Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.

- 7.0.20 Centrifuge: International Equipment Co., Model CL. (or equivalent)
- 7.0.21 Wrist Shaker: Burrell wrist action shaker, Model 75 and 88. (or equivalent)
- 7.0.22 Florisil: 4% deactivated, solvent washed with 1:1 hexane/ether, baked at 130°C for 16 hours. Deactivated with 4% D.I. water. EM Science #FX0282-1.
- 7.0.23 TBA Reagent: Tetrabutylammonium Hydrogen-Sulfite Reagent. (prepared in the laboratory)
- 7.0.24 Mercury: Triple distilled Mercury Refining Co, Albany, NY #328502. (or equivalent)
- 7.0.25 Sulfuric Acid: Na₂SO₄ (concentrated) Malinkrodt #2468 #UN1830. (or equivalent)
- 7.0.26 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)
- 7.0.27 Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL.

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 on the sample tracking sheet.
- 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 The fish samples are usually received as fillets and must be processed to produce a homogenous material prior to extraction. Once the sample has been processed by the sample custodian, the sample is to be cut up into small enough pieces to fit into a standard manual meat grinder. An acetone rinsed Pyrex tray should be placed under the grinder exit to catch the minced tissue. After the fish has been ground once, it should be ground again to ensure complete homogenization of the sample. The ground fish is then placed into an appropriate-sized jar and labeled.

The sample is then placed in the freezer for storage until the extraction process is begun.

8.2 Procedure: Sample Extraction

- 8.2.1 Rinse the thimbles with hexane, allow them to dry out in a pre-cleaned 100mL beaker in a fume hood.
- 8.2.2 Fill a pyrex pan with ice cubes and cold water about 1/2 inch deep. As the samples are weighed out, place the beakers in the pyrex pan to chill for at least 15 minutes prior to the drying step.
- 8.2.3 Into a pre-cleaned and tared 300mL beaker accurately weigh to the nearest 0.0001g using an analytical balance about 10 grams of tissue. Record this weight in the laboratory sample log book. Place the beaker containing the sample into the pyrex pan to chill. Repeat for remaining samples.
- 8.2.4 After the sample has been sufficiently chilled, add approximately 10 g. of a 1:1 mix of magnesium sulfate/sodium sulfate to the sample and mix well with a clean wooden tongue depressor. If the sample has not dried after a few minutes, another 10 g. may be added. Once the sample is well-dried and free flowing, transfer the sample to an extraction thimble. Add a plug of glass wool to the thimble and set aside. Repeat with remaining samples. Set empty mixing beaker and stirring utensil aside for later rinsing into soxhlet extractor to complete sample transfer. **Be careful not to add too much drying agent to the sample; if too much is added, the sample may not fit completely in the thimble. In this case the sample will have to be split into two different soxhlets.**
- 8.2.5 Add 200 mL of a 1:1 mixture of hexane/acetone to a 250 mL round bottom flask. Add several boiling chips. Place a soxhlet extractor on top of the round bottom flask. Label the round bottom with a sample number and use a pair of long-handled tweezers to place the corresponding thimble into the soxhlet extractor. Rinse corresponding beaker & tongue depressor with a few pipets of hexane. Transfer into thimble. Repeat this step twice more with the same sample, and then repeat all preceding steps with remaining samples. After all samples have been processed add the specified surrogate and matrix spikes required directly into thimble.

- 8.2.6 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.
- 8.2.7 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 once every two to three minutes should be achieved.
- 8.2.8 The samples should be extracted overnight for a minimum of 16 hours. Once the sample has finished extracting (usually in the morning), turn the heating mantle off and allow samples to cool to room temperature. Once cool, rinse the inside of the condenser with several pipette volumes of hexane. Disengage the soxhlet and condenser unit and rinse the joint off as well into the soxhlet.
- 8.2.9 Move soxhlet units into a chemical fume hood and flush the remaining solvent from the soxhlet extractor by tipping the soxhlet. Using a pair of long-handled tweezers, pull the thimbles out of the soxhlets one at a time and allow them to drip dry by balancing the thimbles on the tops of the soxhlets. Once dry, remove the thimbles to a pyrex drying pan for total solvent evaporation.
- 8.2.10 Rinse the soxhlet with several pipetfuls of hexane and tip again to drain into the round bottom. Set aside the soxhlet at this time. Procure the same number of turbo tubes as there are samples. Using an individual turbotube stand, label a turbotube with the corresponding sample ID number and place in the holder. Pour the contents of the round bottom into the turbotube, using a pipette and hexane to rinse the last drop out of the mouth of the round bottom. 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.
- 8.2.11 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 .
- 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/acetone) 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 acetone component is fractionally removed at a faster rate than hexane; however, a solvent exchange with hexane should be completed 3x to ensure the acetone 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 (25mL for biota extracts) with three 2mL hexane rinses. 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-labelled 8 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 on the sample tracking log sheet.

Sample extract cleanups are performed on set volume extracts. The set volume is 25 mL for biota samples.

8.4.0 Sulfuric Acid Wash

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

8.4.2 Chill the sample to approximately 0°C. Add 5.0 mL concentrated H₂SO₄ and shake for 30 seconds by hand, centrifuge for approximately 1 minute at a setting of 4, transfer 20mL of the hexane layer(upper layer) to an 8 dram vial.

8.4.3 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.

8.5.0 Elemental Sulfur Clean-up

8.5.1 Elemental sulfur is soluble in the extract solvents used for biota samples. It is commonly found in many sediment/soil samples, decaying organic material, and some industrial wastes. Large amounts of sulfur can cause the electron capture detector (ECD) to signal saturate for long periods during the elution envelope of PCBs. Even small amounts of sulfur can interfere with PCB measurement as a co-eluting chromatographic peak.

8.5.2 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.

8.6.0 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.

8.6.1 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.

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

8.6.3 Transfer the sample extract to a clean 8 dram vial.

8.6.4 The precipitated sulfur can be removed from the extract by performing a sulfuric acid clean-up or a florisil slurry (discussed in 8.8.0).

8.7.0 Removal of Sulfur using TBA Sulfite

8.7.1 The TBA procedure removes elemental sulfur by conversion to the thiosulfate ion which is water soluble.

8.7.2 Add 2.0mL TBA Sulfite Reagent, 1.0 mL 2-propanol, and approximately 3 aliquots (using the dedicated dispenser) of sodium sulfite crystals to the extract and shake for at least 5 minutes on the wrist shaker and observe. An excess of sodium sulfite must remain in the sample extract during the procedure. If the sodium sulfite crystals are entirely consumed add one or two more aliquots to the extract and observe.

- 8.7.3 Place the samples on the wrist shaker for one hour observing at 15 minute intervals to make sure that the sodium sulfite is not consumed. Add 5mL organic free water and shake for five minutes.
- 8.7.4 Place the samples into the centrifuge and spin for approximately 2 minutes on setting #4.
- 8.7.5 Transfer the hexane layer to a new 8 dram vial and cap.
- 8.8.0 Florisil Adsorption (Slurry)
- 8.8.1 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.
- 8.8.2 Add approximately 3 grams of 4% deactivated florisil to each vial containing the sample extract.
- 8.8.3 Vigorously shake the vial for approximately 1 min by hand or on the wrist shaker.
- 8.8.4 Place the vial(s) into the centrifuge for 2 minutes on setting #4.
- 8.8.5 Transfer the extract to a clean 8 dram vial.
- 9.1.0 **Extract Screening and Dilution:**
- 9.1.1 PCB extracts are generally screened by GC initially to determine the approximate concentration before final analysis. Prior site history and client supplied estimates of sample concentration may be used to determine what, if any, extract dilution is necessary. Extracts of unknown concentration are generally screened at a 10 to 100 fold dilution.
- 9.1.2 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 labelled denoting

the equivalent extract volume after the dilution; e.g. a 25mL extract diluted 1 to 10 is labelled "250X", an undiluted 25mL extract is labelled "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.

- 9.1.3 Perform the dilution using an appropriate 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 labelled. Cap and invert the vial at least three times to thoroughly mix the extract with the solvent.
- 9.1.4 Transfer 1 mL of the extract to a labelled 1.5 mL GC autosampler vial. Record the screening dilution on the GC Queue Lab Sheet (Lotus-123 spreadsheet) along with the extract volume, and the sample mass. Submit the GC Queue Lab Sheet with the sample extracts to the GC analyst.

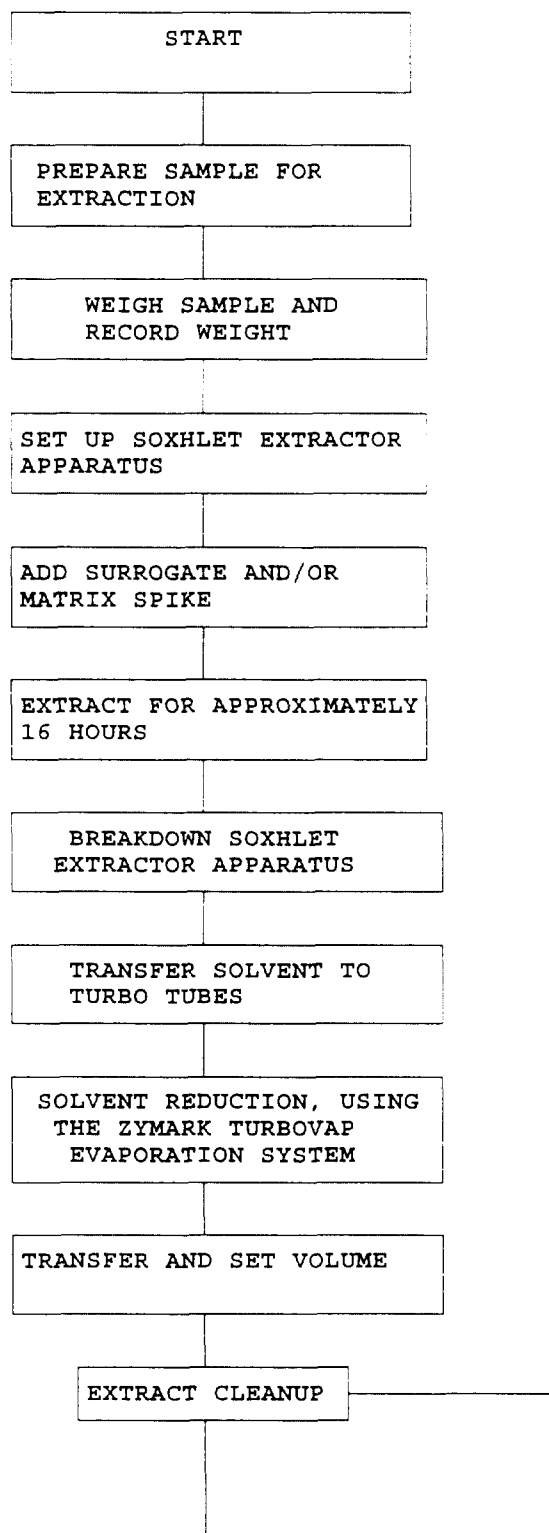
10.0 References

- 1. SW-846 methods 3500A & 3600A; United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Vol.1B, Cincinnati, OH 45268.
- 2. Guide to Environmental Analytical Methods, Genium Publishing Corporation, Schenectady, NY 12304.

11.0 Attachments

- 1. Method flow chart
- 2. Note page for analyst

PCBS IN FISH TISSUE/BIOTA
SOXHLET EXTRACTION/TURBO VAP EVAPORATOR



EXTRACT DILUTION

GC SCREENING/
ANALYSIS

Note page for analyst

STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

NE013_04.SOP

REVISION NUMBER: 04

STANDARD OPERATING PROCEDURE FOR CONGENER-
SPECIFIC POLYCHLORINATED BIPHENYL (PCB) ANALYSIS

METHOD FOR CONGENER-SPECIFIC POLYCHLORINATED
BIPHENYL (PCB) QUANTIFICATION AND IDENTIFICATION BY
CAPILLARY COLUMN/GAS CHROMATOGRAPHY WITH
ELECTRON CAPTURE DETECTION

MAY 01,1999

COPY # _____

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STANDARD OPERATING PROCEDURE
LABORATORY METHOD NE013_04.SOP

REVISION 4 (5/01/1999)

NORTHEAST ANALYTICAL INC.

STANDARD OPERATING PROCEDURES

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Lab Method NE013_04.SOP

Congener-Specific Polychlorinated Biphenyl (PCB) Analysis

Method for Congener-Specific Polychlorinated Biphenyl (PCB)
Quantification and Identification by Capillary Column/Gas
Chromatography with Electron Capture Detection

1.0 Scope

1.1 This method is applicable in the determination and quantification of Polychlorinated Biphenyls (PCB) in sediments, soils, biota, oils, water, and other solid and liquid matrices. This method is a congener-specific determination, employing a high resolution fused-silica capillary chromatographic column. The method has been, in part, developed from the following documents:

1.) "Quality Assurance Plan, Green Bay Mass Balance Study, 1. PCBs and Dieldrin, US EPA Great Lakes National Program Office", prepared by Deborah L. Swackhamer, Quality Assurance Coordinator, Field and Analytical Methods Committees, University of Minnesota, December 11, 1987. This document outlines quality assurance and quality control procedures to be followed by laboratories participating in the Green Bay Mass Balance Study. Where applicable, Northeast Analytical, Inc., will incorporate and utilize this information in quality control of data generated. Instrumental analysis and conditions (Mullin, M.D., 1985, PCB Workshop, US EPA Large Lakes Research Station, Gross Ile, MI, June.) cited in the Green Bay Mass Balance Study document will be refined to be applicable to an in-house data management software package.

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2.) "Comprehensive, Quantitative, Congener-Specific Analyses of Eight Aroclors and Complete PCB Congener Assignments on DB-1 Capillary GC Columns", George M. Frame, Robert E. Wagner, James C. Carnahan, John F. Brown, Jr., Ralph J. May, Lynn A. Smullen, and Donna L. Bedard, *Chemosphere*, Vol. 33, No. 4, pp. 603-623, 1996. This journal publication provides complete assignment of all 209 PCB congeners to the GC peaks separable on a DB-1 capillary column. It also provides weight percent information for PCB congeners in Aroclor formulations used in labeling protocols for reporting purposes.

3.) "Standard Operating Procedure for the Gas Chromatographic Analysis of Hydrophobic Organic Contaminant Extracts from Great Lakes Water Samples", USEPA Great Lakes National Program Office, 77 West Jackson Boulevard, Chicago, IL 60604-2590, GLNPO Organics SOP – 10, 6/1/94:Revision 2. This USEPA SOP summarizes M. Mullin's congener composition of the mixed Aroclor standard used for calibration.

4.) "Biphenyls and Halogenated Pesticides by High Resolution Gas Chromatography", M.D. Mullin, Large Lakes Research Station, LLRS-SOP-ORG-013, revision 2, August 3, 1990, p 1-10. This LLRS SOP written by M. Mullin summarizes the calibration composition of the mixed Aroclor standard reported in "Mullin, M.D., PCB Workshop, U.S. EPA Large Lakes Research Station, Grosse Ile, MI, June 1985.

1.2 This gas chromatographic capillary column method, utilizing an electron capture detector, will effectively separate 112 or more peaks representing 209 PCB congeners.

2.0 Summary of Method

2.1 This method provides detailed instructions for gas chromatographic conditions for analysis of PCBs by capillary gas chromatography.

2.2 This method utilizes a mixed Aroclor standard (Aroclor 1232/1248/1262 in the ratio of 25:18:18) for calibration. Method detection limit and practical quantitation limit will be established experimentally using the procedure in USEPA 40 CFR, Part 136, App. B.

2.3 In general, samples are first extracted with a pesticide-grade solvent. The extracts are further processed through a series of clean-up techniques. The sample is then analyzed by direct liquid injection onto the gas chromatographic column and detected by an electron capture detector. This method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.

2.4 A key component of this method is the importance placed on the chromatographic separation that must be achieved for this congener specific technique. A total of 112 chromatographic peaks are detected, containing 209 PCB congeners in various ratios. This allows an almost complete profile of environmentally occurring PCBs.

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2.5 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 himself with the hazards of handling the compounds used for standards and samples themselves.

3.0 Interference

3.1 One of the major sources of interference in the analysis of PCBs is that organochlorine pesticides are coextracted from the samples. A few of these ECD responding pesticides can be separated cleanly from the PCB profile by the resolving characteristics of the capillary column. Several of the commonly found pesticides and degradation products (DDT, DDE, DD) overlap the PCB profile envelope and co-elute with several of the PCB congeners found in environmental samples. 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 total PCB values generated.

3.2 Sample preparation, extraction procedures, and extract clean-up protocols are covered in separate SOPs that deal exclusively with sample extraction.

3.3 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 give response on electron capture detectors, usually as late eluting peaks and can interfere in PCB quantification.

4.0 Sample Archiving

4.1 Depending on program requirements, sample extracts and certain original samples (i.e. Soil, sediment, tissue, and oil samples) can be retained after analysis. The sample extracts and original samples will be stored in a freezer.

5.0 Equipment and Apparatus

5.1 Gas Chromatograph: Complete system for high resolution, capillary column capability and all required accessories. Northeast Analytical, Inc. will use a Varian Model 3800 gas chromatograph, equipped with capillary on-column injection (Septum Programmable Injector), temperature programmable oven, Model 8200 automatic sampler, and fast time constant electron capture detector. A data system (Waters Associates, Millennium_32 Workstation) for chromatographic operations and integration of detector signal is interfaced to the gas chromatograph.

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5.1.1 GC Column: The gas chromatograph column to be used for analysis will be a DB-1 (J&W Company), bonded polydimethylsilicone, 30 meter fused silica capillary column with an internal diameter of 0.25mm and phase coating thickness of 0.25 microns. This column is capable of resolving 112 chromatographic peaks from the full spectrum of all PCB congeners that could be expected in an environmental sample. Refer to Appendix A and Appendix B for a complete description of PCB congeners identified in each GC chromatographic peak and achievable chromatographic separations.

5.2 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.

5.3 Volumetric Flasks: 10 and 100mL, ground-glass stopper. For standard preparation.

5.4 Microsyringe: 10 and 100uL for standard preparation.

5.5 Pipettes: Class A volumetric, 2mL, 5mL, and 10mL.

5.6 Vials: Glass, 10 and 20mL capacity for sample extracts.

5.7 Bottles: Glass, 120mL capacity for standard storage.

6.0 Reagents and Standards:

6.1 Solvents: Pesticide grade quality. Hexane, acetone, toluene, methylene chloride.

6.2 Octachloronaphthalene: Obtained from Ultra Scientific (Hope, RI) with a purity greater than 95%.

6.3 Polychlorinated Biphenyls: Neat commercial material or solutions for standard preparation. These materials are multi-component mixtures of PCB congeners and are the actual materials that were used in products such as transformers and capacitors. Monsanto was the largest producer of PCB formulations and sold them under the tradename Aroclor.

6.4 PCB Congeners: A complete set of all 209 PCB congeners to individually verify the exact elution on the chosen chromatographic system. A subset of congeners are also used as a secondary supplemental calibration standard for those congeners that do not exist at a high enough level in the Aroclor based calibration standard. Obtained from AccuStandard or Ultra Scientific.

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6.5 Stock Standard Solutions:

6.5.1 Stock standards are prepared from individual neat Aroclor formulations by weighing approximately 0.1000g to the nearest 0.2 mg and dissolving and diluting to volume in a 100mL volumetric flask with hexane. This will give a stock concentration of 1,000ug/mL.

6.5.2 The stock standard is transferred into screw-cap 120mL boston bottles and stored in a freezer, protected from light. Stock standards should be checked at frequent intervals for signs of evaporation, especially just prior to preparing calibration standards.

6.5.3 Stock PCB standards must be replaced after one year, or sooner if comparison with continuing calibration check standards indicate a problem.

6.5.4 Stock standards for the following are prepared by the above procedure:

Aroclor 1232
Aroclor 1248
Aroclor 1262

6.6 Mixed Aroclor Stock Standard at 62.7ug/mL: A stock standard is prepared at 62.7ug/mL that is used for preparing linearity standards and the calibration standard. Using a 5.0mL Class A pipette, accurately add 2.49mL of stock Aroclor 1232 standard (1,033ug/mL) to a 100mL volumetric flask. Using a 2.0mL Class A pipette, accurately add 1.82mL of stock Aroclor 1248 standard (1,019ug/mL) and 1.80mL of stock Aroclor 1262 standard (1,024ug/mL) to the same 100mL volumetric flask. Make volume to the 100mL mark with hexane.

6.6.1 Store the Mixed Aroclor Stock Standard at 62.7ug/mL in a freezer in a tightly capped bottle. This standard must be replaced after one year, or sooner, if comparison with continuing check standards indicate a problem.

6.6.2 High Linearity Standard at 31.35ug/mL: The High Linearity Standard is prepared from the 62.7ug/mL mixed Aroclor stock standard. Using a 5.0mL Class A pipette, accurately add 5.0mL of the 62.7 stock standard into a 10mL volumetric flask. Using a 10 microliter syringe add 9.0uL of 202ug/mL octachloronaphthalene into the same 10mL volumetric flask. The octachloronaphthalene is used as an internal standard for instrument calibration. Transfer the standard solution to a 10ml vial and store in a freezer.

6.6.3 The 31.35ug/mL High Linearity Standard must be replaced after one year.

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6.7 Calibration Standard at 6.27ug/mL: The calibration standard is prepared by combining Aroclor 1232, Aroclor 1248, and Aroclor 1262 in a 25:18:18 ratio with a final mixture concentration of 2.57ug/mL, 1.86ug/mL, and 1.84ug/mL respectively (total = 6.27ug/mL). The final concentration of the mixed standard may vary to accommodate instrument sensitivity or more closely represent sample concentrations, but the same ratio values must be maintained. These ratios are strictly maintained so that the percent composition data remains applicable, since it was developed for use under these fixed mixture parameters. The calibration standard is prepared from the 62.7ug/mL mixed Aroclor stock standard. Using a 10.0mL Class A pipette, accurately add 10.0mL of the 62.7 stock standard into a 100mL volumetric flask. Using a 100 microliter syringe add 90.0uL of 202ug/mL octachloronaphthalene into the same 100mL volumetric flask. Make to volume with hexane. The octachloronaphthalene is used as an internal standard for instrument calibration. Transfer the standard solution to a 120-ml boston bottle and store in a freezer.

6.7.1 The 6.27ug/mL Calibration Standard must be replaced after one year, or sooner, if comparison with continuing check standards indicate a problem.

6.8 Supplemental Congener Standard: A Supplemental Congener Standard is analyzed along with the 6.27ug/mL Calibration Standard. This standard contains congeners that exist at low levels in the mixed Aroclor standard and comprises congeners that are not typically found in Aroclor formulations, but could become important in by-product PCB analysis or the study of model experiments that use unusual PCB congeners. This standard is analyzed to supply accurate retention time information and response factors for quantification. All stock standards are purchased as solutions at 100ug/mL. All supplemental congeners are diluted (except 3-Chlorobiphenyl) to 10ug/ml in hexane by pipetting 1.0mL of the 100ug/mL stock standard into a 10mL volumetric flask and making to volume with hexane. For each congener, pipette 0.5mL of the 10ug/mL secondary stock standard into the same 100mL volumetric flask. For the 3-Chlorobiphenyl, pipette 2.0mL of the 100ug/mL stock standard into the same 100mL volumetric flask. Using a 100 microliter syringe add 90.0uL of 202ug/mL octachloronaphthalene into the same 100mL volumetric flask. Make to volume with hexane. The octachloronaphthalene is used as an internal standard for instrument calibration. Transfer the standard solution to a 120mL boston bottle and store in a freezer. The standard concentration is 2.00ug/mL for 3-Chlorobiphenyl and 0.050ug/mL for all other congeners in the standard. The following table lists the congeners included in this standard and the DB-1 peaks where they elute and also lists the other non-Aroclor congeners that co-elute with these 14 congeners. The Stock Standard and Supplemental Congener Standard must be replaced after one year.

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Supplemental Congener Standard

DB-1 Peak Number	IUPAC Congener Number	(IUPAC #)	PCB Congener Analyzed	Conc ug/mL
3	2	(2)	3-Chlorobiphenyl	2.000
9	14	(14)	3,5-Dichlorobiphenyl	0.050
11	30	(30)	2,4,6-Trichlorobiphenyl	0.050
12	11	(11)	3,3'-Dichlorobiphenyl	0.050
19	23,34,54	(34)	2',3,5-Trichlorobiphenyl	0.050
28	36	(36)	3,3',5-Trichlorobiphenyl	0.050
30	39	(39)	3,4',5-Trichlorobiphenyl	0.050
35	62,65	(65)	2,3,5,6-Tetrachlorobiphenyl	0.050
36	35	(35)	3,3',4-Trichlorobiphenyl	0.050
41	68,96	(96)	2,2',3,6,6'-Pentachlorobiphenyl	0.050
43	57,103	(103)	2,2',4,5',6-Pentachlorobiphenyl	0.050
62	154	(154)	2,2',4,4',5,6'-Hexachlorobiphenyl	0.050
68	123	(123)	2',3,4,4',5-Pentachlorobiphenyl	0.050
70	140	(140)	2,2',3,4,4',6'-Hexachlorobiphenyl	0.050
76	127,168,184	(127)	3,3',4,5,5'-Pentachlorobiphenyl	0.050

6.9 **Internal Standard:** The internal standard used for capillary gas chromatography of PCBs will be octachloronaphthalene (OCN). Weigh, to the nearest 0.1 mg, 10.1mg of solid octachloronaphthalene (OCN) into a 5mL vial. Quantitatively transfer the OCN using six successive 2-mL washings of toluene to a 50mL volumetric flask. Be sure to rinse the 5mL vial walls carefully so that all OCN is completely transferred to the 50mL volumetric flask. Make the solution to volume using toluene and mix the internal standard solution by shaking the flask several times. This will give a concentration of OCN of 202ug/mL. Carefully transfer the internal standard solution to 25mL vials, tightly cap, and store in a freezer. A portion of the internal standard is transferred to a 5mL reacti-vial with a Teflon syringe-valve cap to use on a daily basis. The react-vial minimizes evaporation since the cap does not have to be removed.

6.9.1 The OCN internal standard is added to all calibration standards, continuing check standards, blanks, samples, and QC samples at the same amount. In most cases this will be achieved by spiking 9.0uL of OCN internal standard solution to 10mL of standard or sample extract to give a concentration of 0.1818ug/mL.

6.9.2 The internal standard will be added to calibration standards, sample extracts, blanks, and QC samples prior to gas chromatographic analysis. Thus, the internal standard is used as a quantification spiking standard and will eliminate sample injection volume variations, but will not correct for analytical losses during sample preparation.

6.9.3 OCN internal standard must be replaced after one year.

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6.10 Continuing Check Standards: Continuing check standards at 1.27ug/mL and 0.127ug/mL are prepared from Aroclor solutions obtained from a different source (ULTRA Scientific) than the calibration standard. The continuing standard is a mixed Aroclor 1232, 1248, and 1262 in the fixed ratio used to prepare the calibration standard and must be strictly adhered to.

6.10.1 50.0ug/mL Continuing Check Stock Standards: Aroclor 1232, Aroclor 1248, and Aroclor 1262 are obtained from ULTRA Scientific at 1000ug/mL in isooctane. Using a 1.0mL Class A pipette, 0.5mL of 1000ug/mL Aroclor 1232 is transferred to a 10.0mL volumetric flask and made to volume with hexane. This procedure is repeated for Aroclor 1248 and Aroclor 1262. The 50ug/mL stock standards are transferred to a 10mL vial, tightly capped and stored in a freezer. These stock standards must be replaced after one year.

6.10.2 1.27ug/mL Continuing Check Standard with OCN: Using a 1.0mL Class A pipette transfer 1.0mL of 50.0ug/mL Aroclor 1232, 0.5mL of 50.0ug/mL Aroclor 1248, and 0.5mL of 50.0ug/mL Aroclor 1262 into a 100mL volumetric flask. Using a 100 uL microsyringe, add 90.0uL of OCN internal standard (final concentration of 0.1818 ug/mL). Make to volume with hexane and mix well by shaking and inverting flask several times. The prepared continuing check solution will contain a total of 1.27ug/mL PCB (0.500ug/mL Aroclor 1232, 0.360ug/mL Aroclor 1248, and 0.360ug/mL Aroclor 1262).

6.10.3 Transfer the 1.27ug/mL Continuing Check Standard with OCN to a 120mL boston bottle, cap tightly, and store in a freezer. A new continuing check standard must be prepared every six months.

6.10.4 1.27ug/mL Continuing Check Standard without OCN: Using a 1.0mL Class A pipette transfer 1.0mL of 50.0ug/mL Aroclor 1232, 0.5mL of 50.0ug/mL Aroclor 1248, and 0.5mL of 50.0ug/mL Aroclor 1262 into a 100mL volumetric flask. Make to volume with hexane and mix well by shaking and inverting flask several times. The prepared continuing check solution will contain a total of 1.27ug/mL PCB (0.500ug/mL Aroclor 1232, 0.360ug/mL Aroclor 1248, and 0.360ug/mL Aroclor 1262).

6.10.5 Transfer the 1.27ug/mL Continuing Check Standard without OCN to a 120mL boston bottle, cap tightly, and store in a freezer. A new continuing check standard must be prepared every six months. This continuing check standard without OCN is used to prepare the 0.127ug/mL Continuing Check Standard with OCN.

6.10.6 0.127ug/mL Continuing Check Standard with OCN: Using a 10.0mL Class A pipette transfer 10.0mL of 1.27ug/mL Continuing Check Standard without OCN to a 100mL volumetric flask. Using a 100uL microsyringe, add 90.0uL of OCN internal standard (final concentration of 0.1818ug/mL). Make to volume with hexane and mix well by shaking and inverting flask several times. The prepared continuing check solution will contain a total of 0.127ug/mL PCB (0.050ug/mL Aroclor 1232, 0.036ug/mL Aroclor 1248, and 0.036ug/mL Aroclor 1262).

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6.10.7 Transfer the 0.127ug/mL Continuing Check Standard with OCN to a 120mL boston bottle, cap tightly, and store in a freezer. A new continuing check standard must be prepared every six months.

7.0 Procedure

7.1 Calibration:

7.1.1 Gas chromatographic Operation Parameters: Establish the gas chromatographic operation parameters as follows:

GC Column: DB-1 (J&W, bonded polydimethylsilicone), 30 meters,
0.25 mm internal diameter, 0.25 micron phase coating.

Oven Temperature Program: 50°C for 1.0 min hold time, 50°C to 220°C at
6.0°C/min hold at 220°C for 39.70 min.

GC Column Velocity: Approximately 30 cm/sec Helium. Column flow adjusted
to elute OCN Internal Standard near 52.0 minutes.

Detector: Electron Capture Detector (ECD), attenuation 1, range 10,
autozero on.

Detector Temperature: 300°C.

Injector Temperature Program: 45°C for 0.10 min hold time, 45°C to 250°C at
200°C/min, hold at 200°C for 62.0 min.

Detector Make-up Gas: Approximately 30mL/min Nitrogen. Adjusted for signal
sensitivity.

Autosampler: Multi-vial mode, 0.5uL sample volume (adjustable based on signal
sensitivity). Injection rate (3.0uL/sec), 0.04 min injection time, solvent plug size
0.1uL, upper air gap – yes, lower air gap - no.

7.1.2 Initial GC Calibration: Prior to running samples the system must be
calibrated and the Continuing Calibration Check Standard must be verified.

7.1.2.1 Establish the gas chromatographic operation parameters
outlined in Section 7.1.1 and prepare the appropriate calibration standards
composed of a mixture of Aroclors 1232, 1248, and 1262 as outlined in Sections
6.5 through 6.10.

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7.1.2.2 Chromatographic Resolution Criteria: Chromatographic resolution is measured by peak height to valley height for two pairs of closely eluting peaks. The peak valley height formed between DB-1 peaks 14 and 15 must be equal to or less than the half height of peak 15. The peak valley height formed between DB-1 peaks 74 and 75 must be equal to or less than one-third the height of peak 74. This peak resolution must be established initially and maintained throughout sample analysis.

7.1.2.3 Initial High Level Linearity Verification: The gas chromatograph must undergo a linearity study. A high-level three-point initial calibration check for linearity is analyzed and calculated relative response factors must meet a limit for relative standard deviation for each GC peak of less than 20%. This high level linearity verification is associated with samples with medium to high levels of PCBs for sample matrices such as biota, sediment, soil, oil, and other solid or liquid samples that contain appreciable levels of PCBs. The high level initial linearity is comprised of the following standards: 31.35ug/mL High-Level Linearity Standard, 6.27ug/mL Calibration Standard, and the 1.27ug/mL Continuing Calibration Standard.

7.1.2.4 Initial Low Level Linearity Verification: The gas chromatograph must undergo a linearity study. A low-level three-point initial calibration check for linearity is analyzed and calculated relative response factors must meet a limit for relative standard deviation for each GC peak of less than 20%. This low level linearity verification is associated with samples with low levels of PCBs for sample matrices such as water and other solid or liquid samples that contain low levels of PCBs. The low-level initial linearity is comprised of the following standards: 6.27ug/mL Calibration Standard, the 1.27ug/mL Continuing Calibration Standard, and the 0.127ug/mL Continuing Calibration Standard.

7.1.2.5 Initial 72-Hour Retention Time Window Measurement: An initial retention time study must be performed to establish retention time windows to assist in PCB peak assignment. Three high level Continuing Calibration Check standards must be analyzed over at least a 72-hour period. In addition, the Supplemental Congener Standard is analyzed three times over at least a 72-hour period. The mean retention time is calculated from these standards along with the standard deviation for each GC peak. The retention time window is established as ± 3 times the determined standard deviation. If the 3 times the standard deviation value is less than 0.07 minutes a default of 0.07 minutes is applied to that GC peak. The default 0.07 minutes retention time window is the default window used in Contract Laboratory Program, Statement of Work for Organic Analysis for PCB/Pesticide analysis.

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7.1.2.6 Our laboratory will use a computer based data acquisition workstation (Waters Associates, Millennium_32 workstation software), interfaced to the gas chromatograph. The workstation processes the detector signal, performs an analog to digital conversion, and stores the digitized chromatograms on the computer hard disk. All data analysis will be done on the specialized software package including peak integration, calculating calibration curves/response factors, report generation, chromatogram hardcopies, and archival of data. Calculate the response factor for each separated and identified peak.

7.1.2.7 After the above criteria are met system calibration for sample analysis can be performed. Appendix A identifies which congener and or congeners compose each resolvable GC peak in the calibration standard, along with the amount that each congener or co-eluting group of congeners are represented in the calibration standard. Throughout this document the IUPAC PCB numbering system will be used for congener identification. Appendix B is an example of an acceptable chromatogram of the calibration standard, along with peak congener labels for cross-reference to data in Appendix A. Analyze the 6.27ug/mL Calibration Standard to initiate calibration of the GC system. Also analyze the Supplemental Congener Standard to calculate relative response factors for congeners that do not exist in the 6.27ug/mL Calibration Standard. Response factors are calculated relative to the internal standard by the following equation:

$$RRF = (A_x/A_{is}) \times (C_{is}/C_x)$$

Where:

RRF	=	Relative response factor of congener(s)
A _x	=	Area of peak for the congener(s).
A _{is}	=	Area of peak for the internal standard
C _x	=	Concentration of the congener(s).
C _{is}	=	Concentration of the internal standard.

7.2 On-going Calibration:

7.2.1 Chromatographic Resolution:

7.2.1.1 Chromatographic resolution is measured by peak height to valley height for two pairs of closely eluting peaks. The peak valley height formed between DB-1 peaks 14 and 15 must be equal to or less than the half height of peak 15. The peak valley height formed between DB-1 peaks 74 and 75 must be equal to or less than one-third the height of peak 74. This peak resolution must be established initially and maintained throughout sample analysis.

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7.2.2 Response Factors:

7.2.2.1 The relative response factors calculated from the calibration standard will be verified on each working day by analyzing a Continuing Calibration Standard, calculating the selected congener concentrations and comparing to their known concentration. A subset of six congeners will be used to verify the relative response factors before samples are processed. The six congeners include:

DB-1 PEAK NUMBER	IUPAC CONGENER NUMBER	RELATIVE PEAK LEVEL IN CALIBRATION STANDARD	PEAK CONC 1.27ug/mL CONTINUING CALIBRATION STD (ng/mL)	PERCENT DIFFERENCE LIMITS
7	6	Low level peak in standard	14.10	<30
116	205	Low level peak in standard	0.820	<30
47	70	Medium level peak in standard	25.22	<10
93	174,181	Medium level peak in standard	23.74	<10
37	104,44	high level peak in standard	31.90	<10
102	180	high level peak in standard	45.26	<10

7.2.3 After the Continuing Calibration Standard is analyzed, calculate the amount for these six congeners and compare those values to the known concentrations by the following equation:

$$\text{Percent Difference} = [\text{Amt}(1) - \text{Amt}(2)] / \text{Amt}(2) \times 100$$

Where: Amt(1) = Amount calculated for congener.
Amt(2) = Known amount for congener.

7.2.4 A percent difference greater than $\pm 30\%$ for the two low-level peaks (7 and 116) indicates an instrument problem or unacceptable relative response factors. A percent difference greater than $\pm 10\%$ for the medium level (47 and 93) and high level (37 and 102) peaks also indicates an instrument problem or unacceptable relative response factors. If any of the evaluation congeners fail to meet the percent difference acceptance criteria, the calibration standard must be re-analyzed and new relative response factors generated.

7.2.5 The Continuing Calibration Standard must be analyzed again and values calculated using the new relative response factors. If the Continuing Calibration Standard fails to meet the percent difference criteria after re-calibration, sample analysis must not proceed until the problem is found and corrected (i.e., GC gas leak, autosampler lines plugged, broken injector liner).

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7.3 Sample Preparation and Extraction

7.3.1 The following SOPs detail sample extraction procedures that are utilized in preparing samples for analysis by this analytical method:

SOP NAME	TITLE
NE005	SOXHLET EXTRACTION: SOLIDS
NE006	WATER EXTRACTION
NE017	FISH & BIOTA EXTRACTION
NE049	ANIMAL TISSUE EXTRACTION SMALL MASS PROCEDURE
NE087	EXTRACTION FOR OIL
NE088	WIPE EXTRACTION FOR PCB
NE124	CLLE PCB EXTRACTION
NE132	FISH/BIOTA GRINDING PROCEDURES
NE140	PCB SCREENING BY GC
NE143	ASE EXTRACTION FOR PCB: SOLIDS
NE144	ASE EXTRACTION FOR WIPE: PCB
NE158	% LIPID DETERMINATION: FISH & BIOTA

7.4 Calculations:

7.4.1 SCREENING GC: External Standard Calibration:

7.4.1.1 The GC screening analysis will be done by the external standard calibration technique. See standard operating procedure NE140 for details on the PCB screening procedures used prior to final analysis by this method. The GC will be standardized by using Aroclor 1242 and Aroclor 1260. These two Aroclor formulations incorporate most environmental PCBs found in sample extracts and provide a good estimate of PCB amount for final dilution for capillary analysis. A two level calibration curve is utilized (0.50ug/mL and 2.5ug/mL standards).

7.4.1.2 The calibration curves for each section of the PCB elution profile will be calculated using the following formula:

$$\text{Calibration factor} = \frac{\text{Amount (ug) of Aroclor}}{\text{Total area of Aroclor}}$$

The calibration curve will be a linear fit forced through zero.

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7.4.2 SCREENING GC: Sample Calculations

7.4.2.1 The concentration of each Aroclor (grouped as Aroclor 1242 and Aroclor 1260 only) in a sample will be calculated based on the extract volume (not the sample weight or volume) to supply solution concentration values that show if the extract needs to be diluted for final capillary GC analysis. The solution concentration of either Aroclor 1242 or Aroclor 1260 (or both) in a sample is calculated as follows:

$$\text{Concentration (ug/mL)} = (\text{Ax}) \times (\text{CF})$$

Where: Ax = Area of Aroclor of interest in sample
CF = Calibration factor in standard

7.4.3 Capillary GC: Internal Standard Calibration

7.4.3.1 The capillary column GC analysis will be done by the internal calibration technique. Calibration and sample quantification will be performed by a commercial GC software package. The capillary GC will be standardized by using an Aroclor mixture that encompasses all the possible PCB congeners present in environmental samples. Refer to Section 6.6 for details on the calibration standard and Aroclor ratios.

7.4.3.2 Response factors for each separated and identified peak in the standard will be calculated using the following formula:

$$\text{RRF} = (\text{Ax}/\text{Ais}) \times (\text{Cis}/\text{Cx})$$

Where: RRF = Relative response factor of congener(s).
Ax = Area of peak for the congener(s).
Ais = Area of peak for the internal standard.
Cx = Concentration of the congener(s).
Cis = Concentration of the internal standard

7.4.4 Capillary GC: Sample Calculations

7.4.4.1 The concentration of each identified PCB peak in a sample will be calculated based on the sample dry weight in the case of soils and sediments and wet weight for fish and biota samples.

7.4.4.2 The sample PCB concentration of each standardized PCB peak is calculated as follows:

$$\text{Concentration (ng/g)} = \frac{[(A_x)(C_{is})(V)(D)]}{[(A_{is})(RRF)(W_s)]}$$

Where:

- A_x = Peak area for congener(s) being measured.
- C_{is} = Amount of internal standard added to sample extract.
- D = Dilution factor, if sample was diluted prior to analysis.
- V = Extract volume.
- A_{is} = Peak area of added internal standard.
- RRF = Relative response factor for congener(s) being measured as determined in Section 7.4.3.2.
- W_s = Dry or wet weight of sample.

7.4.5 Data Output and Reporting Format:

7.4.5.1 Several specialized software routines have been developed for high resolution PCB analysis to aid the data user in understanding and organizing the complex data generated from this extremely detailed analysis. Appendix C contains examples of the sample hard copy format that will be used in reporting sample information. This data is also available in electronic format as an Excel™ file.

8.0 Quality Control

8.1 The following table lists the Quality Control samples required for capillary gas chromatography analysis of PCBs in soils and sediments.

Quality Control Requirements

<u>QC Sample</u>	<u>Frequency</u>
Lab Blank	With each sample batch (up to 20 samples)
Lab Control Spike	With each sample batch (up to 20 samples)
Continuing Check	Analyzed prior to each sample batch (up to 10 samples)
Duplicate Analysis	Field generated sample – analyzed at discretion of client
Matrix Spike	One matrix spike per 20 field samples
Matrix Spike Duplicate	One matrix spike duplicate per 20 field samples

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8.1.2 Laboratory Blank: The laboratory blank will monitor and assess whether the contamination or excessive interference is occurring from laboratory solvents, reagents, and glassware used in processing samples for analysis. The laboratory blank is taken through the sample extraction and clean-up procedures to include all manipulations exposed to actual samples (required volume of solvents, concentration steps, clean-up procedures, etc.) If the laboratory blank is positive for PCB above the reporting limit, the contamination must be traced down and eliminated before samples can be processed and analyzed. If non-PCB contamination occurs that interferes with PCB quantification, it too must be traced down and eliminated before proceeding with sample analysis. The laboratory blank will consist of granular sodium sulfate for solids and organic free water for water samples.

8.1.2.1 Samples associated with a positive laboratory blank should be flagged when the data is reported. If a laboratory blank is positive for PCBs, the source of contamination must be located and eliminated. If the contamination occurred during the extraction procedure and more sample is available the samples will require re-extraction and re-analysis. If the contamination occurred after this step, then re-extraction may not be required and the existing extracts will be reanalyzed. Any aliquots of the extracts (i.e., injection vials) which could have become contaminated will be discarded.

8.1.3 Laboratory Control Spike: A Laboratory Control Spike sample is analyzed with each extraction batch. An Aroclor is spiked into either granular sodium sulfate for solids or organic free water for liquids. This Control Spike must achieve a percent recovery of 70 to 130 percent. If the Control Spike recovery is not within limits, the cause must be tracked down and corrected. If there is sufficient sample, the samples associated with the Laboratory Control Spike that failed must be re-extracted and re-analyzed. If no more sample is available, the data must be flagged to indicate low or high Control Spike recovery.

8.1.4 Continuing Check Standard: As outlined in section 7.2, a Continuing Check Standard will be analyzed on each working day prior to sample analysis and at an interval of one Continuing Check Standard per 10 samples. The Continuing Check Standard must meet the acceptance criteria established in Section 7.2. If the Continuing Check Standard fails to meet the acceptance criteria, the calibration standard must be re-analyzed and new response factors generated.

8.1.4.1 The Continuing Check Standard must be analyzed again and compared to the acceptance criteria. If the Continuing Check Standard fails to meet the acceptance criteria after re-calibration, sample analysis must not proceed until the problem is corrected.

8.1.4.2 All samples that were analyzed after the continuing check standard exceeded established criteria must be re-analyzed.

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8.1.5 Duplicate Analysis: Duplicate analysis of the same sample is performed to assess method precision. A duplicate can also be performed as a blind duplicate, with the sample split in the field. The analysis of a duplicate sample precludes that PCBs are to be found at appreciable levels in samples. If this is not known the analysis of matrix spike / matrix spike duplicates provide more consistent quality control information. The relative percent difference of the two measurements on the sample is calculated on total PCB concentration by the following equation:

$$RPD = (DUP1 - DUP2)/AVG \times 100$$

Where: RPD = Relative Percent Difference.
DUP1 = The greater of the measured values.
DUP2 = The lesser of the measured values.
AVG = Average of the two analysis.

8.1.5.1 The relative percent difference must be less than or equal to 25% if the concentration of PCB in the sample is greater than or equal to 0.5ug/g. The percent relative standard deviation must be less than or equal to 50% if the concentration of the PCB in the sample is less than 0.5ug/g.

8.1.6 Matrix Spike and Matrix Spike Duplicate: Spiked sample matrix data are analyzed to assess analytical accuracy and recovery of analytes of interest. Thus the sample is spiked and carried through sample analytical procedures including extraction, clean up, and GC analysis.

8.1.6.1 There must be sufficient sample for analysis of matrix spike/matrix spike duplicate samples and the sample must be homogeneous in PCB distribution for valid data to be produced. Preferably a sample of low level should be used in this case so that the spike level is of sufficient concentration over the background level of the chosen sample. Spike the two samples with the Aroclor matrix spike standard at a concentration approximately five times the sample concentration. Extract and analyze the two spiked samples following procedures used for actual sample analysis. Calculate the percent recovery of the matrix spike/matrix spike duplicate by the following equation:

$$P = A-B/T \times 100$$

Where: P = Percent recovery, %.
A = concentration of analyte in the spike sample aliquot
T = Known true value of the spike concentration
B = Background concentration of PCB in the unspiked sample aliquot

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8.1.6.2 Matrix spike recovery information is used to assess the long-term precision and accuracy of the method for each encountered matrix. Matrix spike/matrix spike duplicate results are not used alone to qualify an extraction batch. Generally, percent recovery for MS/MSD samples should be greater than or equal to 70% and less than or equal to 130% based on the total PCB concentration. If the percent recovery is outside the limits, all calculations should be checked and the data should be narrated to describe possible matrix interference.

8.1.7 Surrogates: Surrogate spiking compounds monitor the extraction efficiency and sample processing procedures for each sample. Surrogate compounds are chosen which do not chromatographically interfere with the PCB target congeners and which behave similarly to the target PCB congeners during extraction and sample processing.

8.1.7.1 Surrogate spike additions are made to each field sample, method blank, and extracted QC check sample prior to extraction. Congener IUPAC 207 (2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl) or Tetra-Chloro-Meta-xylene and Decachlorobiphenyl surrogate compounds are used.

8.1.7.2 Calculate the surrogate percent recovery as follows:

% Recovery = (Surr. Amount spiked / Theoretical Spike conc.) * 100

8.1.7.3 The percent recovery limits for surrogates are:

IUPAC 207 70% - 130%

Tetra-Chloro-Meta-Xylene 70% - 130%

Decachlorobiphenyl 70% - 130%

8.1.8 Retention Time Windows:

8.1.8.1 The Continuing Check Standards establish the retention time window for each analyte from the retention time windows determined during the 72-hour retention time window study. The retention time window equals the absolute retention time of the Initial Continuing Check Standard for a given batch of samples plus or minus three times the standard deviation determined in Section 7.1.2.5

8.1.8.2 Besides using the retention time window to assign peaks for quantification, the analyst should also rely on their experience in pattern recognition of multi-residue sample analysis.

8.1.9 Analytical Sequence Queue: A typical analytical sequence is as follows:

- 1) Continuing check standard (high or low)
- 2) Method Blank
- 3) Lab Control Spike
- 4 to 10) Samples (including Duplicates, MS/MSD)
- 11) Continuing Check Standard
- 12 to 20) Samples (including Duplicates, MS/MSD)
- 21) Continuing Check Standard; repeat 12 to 20

9.0 References

9.1 US EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants," July, 1988.

9.2 Standard Methods for the Examination of Water and Wastewater, 19th Edition, Published by: American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1995.

9.3 US EPA SW-846, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," Office of Solid Waste and Emergency Response, 3rd Edition, 1986 and its updates.

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

9.5 Mullin, M.D. 1985. PCB Workshop, US EPA Large Lakes Research Station, Grosse Ile, MI, June

9.6 M. Zell, K. Ballschmiter, Baseline Studies of the Global Pollution, III. Trace Analysis of Polychlorinated Biphenyls (PCB) by ECD Glass Capillary Gas Chromatography in Environmental Samples of Different Trophic Levels, Fresenius Z. Anal. Chem., 304, 337-349, 1980.

9.7 M.D. Mullin, C.M. Pochini, S. McCrindle, M. Romkes, S.H. Save, "High-Resolution PCB Analysis: Synthesis and Chromatographic Properties of All 209 PCB Congeners," Environ. Sci. Technol., Vol 18, No. 6, pp 468-476, 1984.

9.8 D.L. Swackhamer, "Quality Assurance Plan, Green Bay Mass Balance Study, 1. PCBs and Dieldrin, US EPA Great Lakes National Program Office" Quality Assurance Coordinator, Field and Analytical Methods Committees, University of Minnesota, December 11, 1987.

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9.9 George M. Frame, Robert E. Wagner, James C. Carnahan, John F. Brown, Jr., Ralph J. May, Lynn A. Smullen, and Donna L. Bedard, "Comprehensive, Quantitative, Congener-Specific Analyses of Eight Aroclors and Complete PCB Congener Assignments on DB-1 Capillary GC Columns", Chemosphere, Vol. 33, No. 4, pp. 603-623, 1996.

9.10 "Standard Operating Procedure for the Gas Chromatographic Analysis of Hydrophobic Organic Contaminant Extracts from Great Lakes Water Samples", USEPA Great Lakes National Program Office, 77 West Jackson Boulevard, Chicago, IL 60604-2590, GLNPO Organics SOP - 10, 6/1/94:Revision 2

9.11 "Biphenyls and Halogenated Pesticides by High Resolution Gas Chromatography", M.D. Mullin, Large Lakes Research Station, LLRS-SOP-ORG-013, revision 2, August 3, 1990, p 1-10.

9.12 Contract Laboratory Program - Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration. Document OLM3.2, 1996.

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APPENDIX A

Congener Composition of Multi-Aroclor Calibration Standard (6.27 ug/mL)

**Congener Composition of Aroclor Calibration Mixture (6.27 ppm)
(Aroclors 1232, 1248, 1262 in a ratio of 25:18:18)**

file S:\TEXT\SOP\CSSOP01.DOC

DB-1 Peak Number ¹	IUPAC # ²	Relative Retention Time	Amount ng/mL
2	001	0.3582	445.14
3	002	0.3867	-
4	003	0.3890	259.67
5	004 010	0.4041	126.12
6	007 009	0.4239	44.51
7	006	0.4307	70.48
8	005 008	0.4346	519.33
9	014	0.4468	-
10	019	0.4476	10.39
11	030	0.4574	-
12	011	0.4586	-
13	012 013	0.4619	9.90
14	015 018	0.4646	137.25
15	017	0.4662	137.25
16	024 027	0.4718	9.64
17	016 032	0.4770	144.67
19	023 034 054	0.4848	-
20	029	0.4880	1.97
21	026	0.4903	26.71
22	025	0.4917	11.87
23	031	0.4953	152.94
24	028 050	0.4960	195.75
25	020 021 033 053	0.5021	147.34
26	022 051	0.5060	107.58
27	045	0.5099	33.01
28	036	0.5127	-
29	046	0.5145	14.84
30	039	0.5171	-
31	052 069 073	0.5196	176.94
32	043 049	0.5223	85.32
33	038 047	0.5242	37.10
34	048 075	0.5252	37.10
35	062 065	0.5278	-
36	035	0.5287	-
37	104 044	0.5316	159.51
38	037 042 059	0.5336	96.45
39	041 064 071 072	0.5392	152.09
41	068 096	0.5418	-
42	040	0.5433	34.87
43	057 103	0.5473	-
44	058 067 100	0.5501	4.08
45	063	0.5525	7.79
46	074 094 061	0.5552	70.48
47	070	0.5573	126.12
48	066 076 098 080 093 095 102 088	0.5590	267.08

DB-1 Peak Number ¹	IUPAC # ²	Relative Retention Time	Amount ng/mL
49	055 091 121	0.5637	18.92
50	056 060	0.5684	129.83
51	084 092 155	0.5720	66.77
52	089	0.5736	3.71
53	090 101	0.5760	66.77
54	079 099 113	0.5790	27.45
55	119 150	0.5832	1.04
56	078 083 112 108	0.5847	5.56
57	097 152 086	0.5879	20.77
58	081 087 117 125 111 115 145	0.5905	43.03
59	116 085	0.5929	25.97
60	120 136	0.5948	27.82
61	077 110 148	0.5968	79.01
62	154	0.6012	-
63	082	0.6027	16.32
64	151	0.6077	63.06
65	124 135	0.6100	10.76
66	144	0.6111	22.26
67	107 109 147	0.6121	4.82
68	123	0.6138	-
69	106 118 139 149	0.6155	148.38
70	140	0.6173	-
71	114 134 143	0.6228	7.49
72	122 131 133 142	0.6262	1.08
73	146 165 188	0.6313	14.47
74	105 132 161	0.6335	50.26
75	153	0.6363	109.25
76	127 168 184	0.6379	-
77	141	0.6458	63.06
78	179	0.6470	54.16
79	137	0.6508	2.78
80	130 176	0.6535	9.64
82	138 163 164	0.6576	100.16
83	158 160 186	0.6609	9.27
84	126 129	0.6645	0.48
85	166 178	0.6707	40.8
87	175 159	0.6762	7.42
88	182 187	0.6789	133.54
89	128 162	0.6809	3.71
90	183	0.6844	63.06
91	167	0.6892	1.82
92	185	0.6954	17.43
93	174 181	0.7022	118.7
94	177	0.7069	63.06
95	156 171	0.7125	29.31
96	157 202	0.7172	2.45
98	173	0.7204	1.41
99	201	0.7272	14.47
100	172 204	0.7317	20.77
101	192 197	0.7370	4.08

DB-1 Peak Number ¹	IUPAC # ²	Relative Retention Time	Amount ng/mL
102	180	0.7405	226.28
103	193	0.7450	15.58
104	191	0.7507	4.45
105	200 <i>169</i>	0.7570	15.95
106	170	0.7783	47.48
107	190	0.7832	15.58
108	198	0.7992	4.45
109	199	0.8036	155.8
110	196 203	0.8135	159.51
111	189	0.8351	1.48
112	195	0.8636	20.51
113	208	0.8733	9.16
114	207	0.8908	3.45
115	194	0.9172	66.77
116	205	0.9337	4.08
117	206	1.0295	25.22
118	209	1.1419	0.45

1 - Note that 5 DB-1 peaks (PK18, PK40, PK81, PK86, PK97) have been removed from the DB-1 peak numbering scheme. The following low level congeners that were designated as separately eluting peaks have been determined to co-elute with another congener. The DB-1 peak numbers are no longer required for these congeners, but the original DB-1 numbering system has remained intact for all other peaks.

PK 18 (23) now elutes in PK 19 (23,34,54)

PK 40 (68) now elutes in PK 41 (68,96)

PK 81 (176) now elutes in PK 80 (130,176)

PK 86 (166) now elutes in PK 85 (166,178)

PK 97 (157) now elutes in PK 96 (157,202)

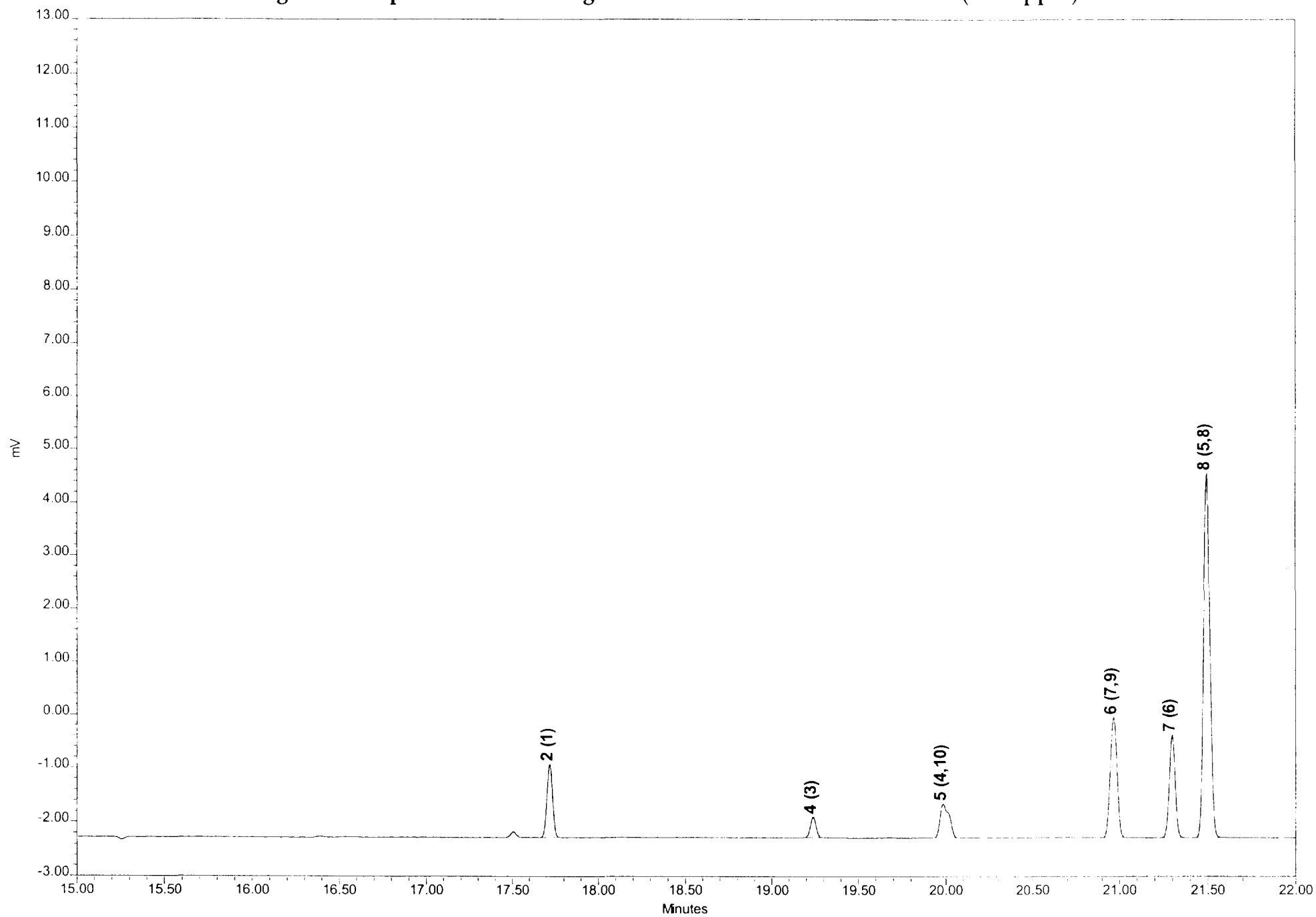
2 - IUPAC congener numbers listed in boldface font were found to be present in at least one of the Aroclors at or above 0.05 weight percent. These congeners should be considered the primary congeners existing in a peak composed of co-eluting congeners. IUPAC congener numbers listed in italic font were absent or present below 0.05 weight percent.

APPENDIX B

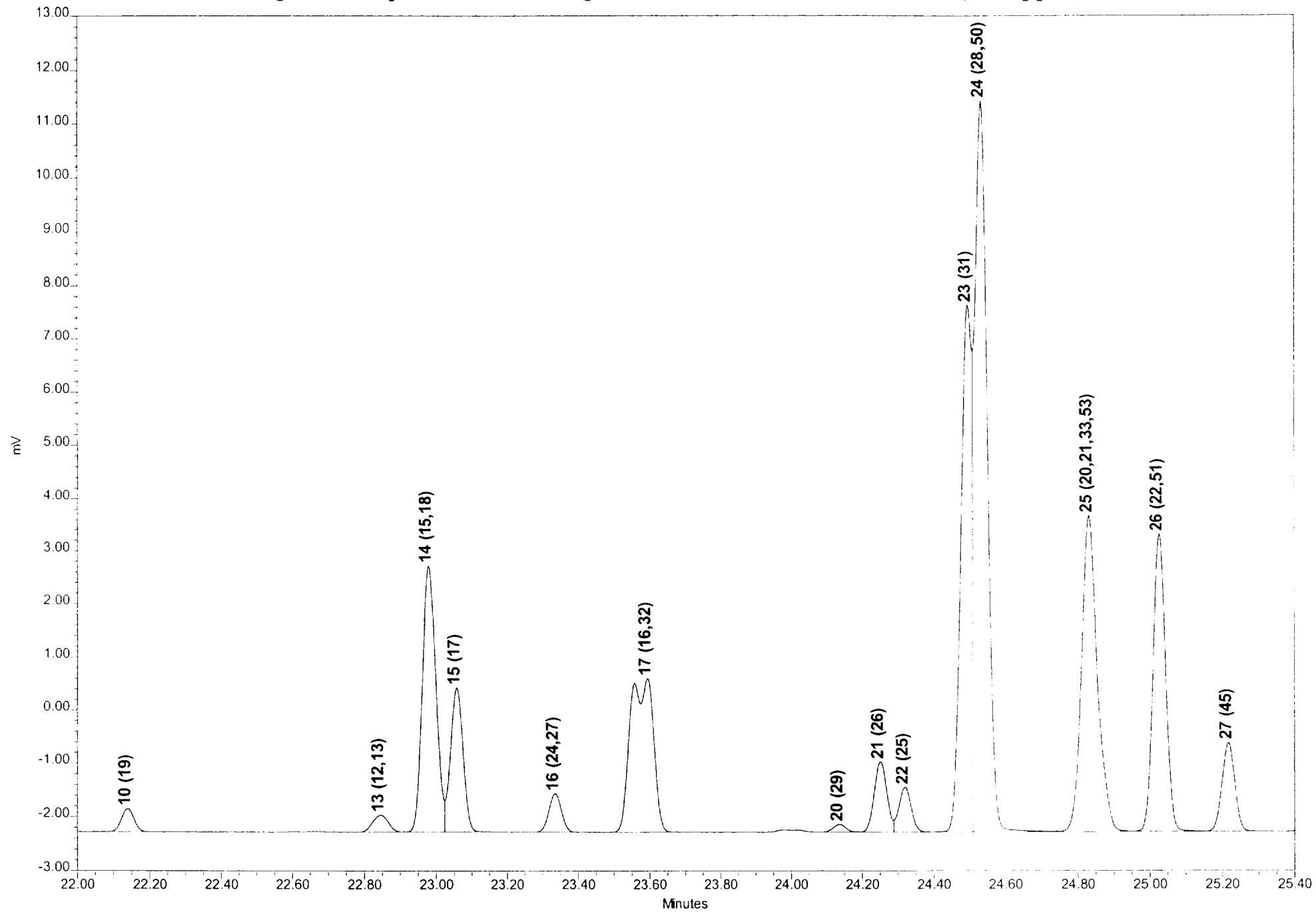
DB-1 Capillary GC Chromatograms:

- 1.) 6.27 ug/mL Calibration Standard
- 2.) Supplemental Congener Standard

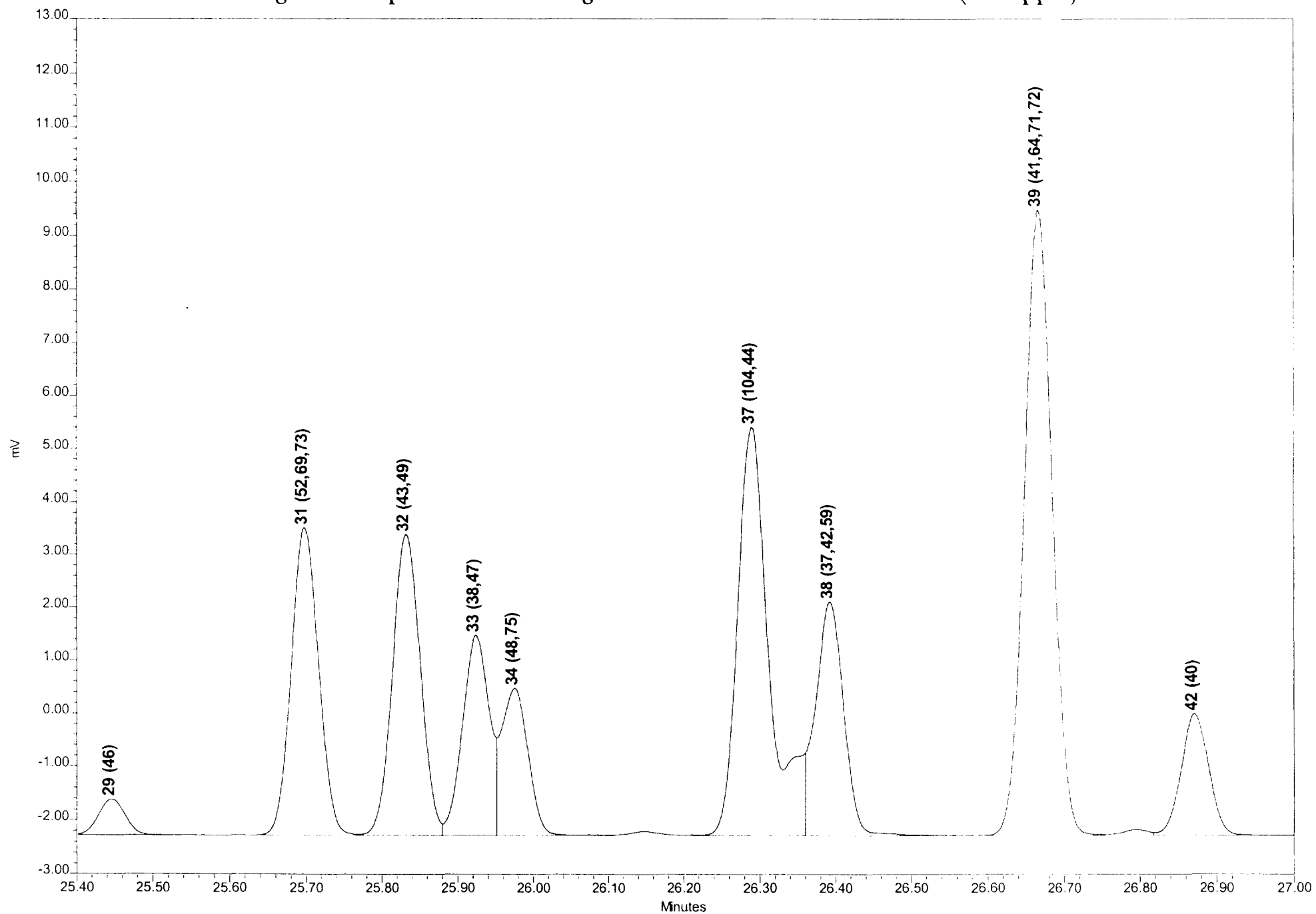
Congener Composition Chromatogram of Aroclor Calibration Mixture (6.2/ ppm)



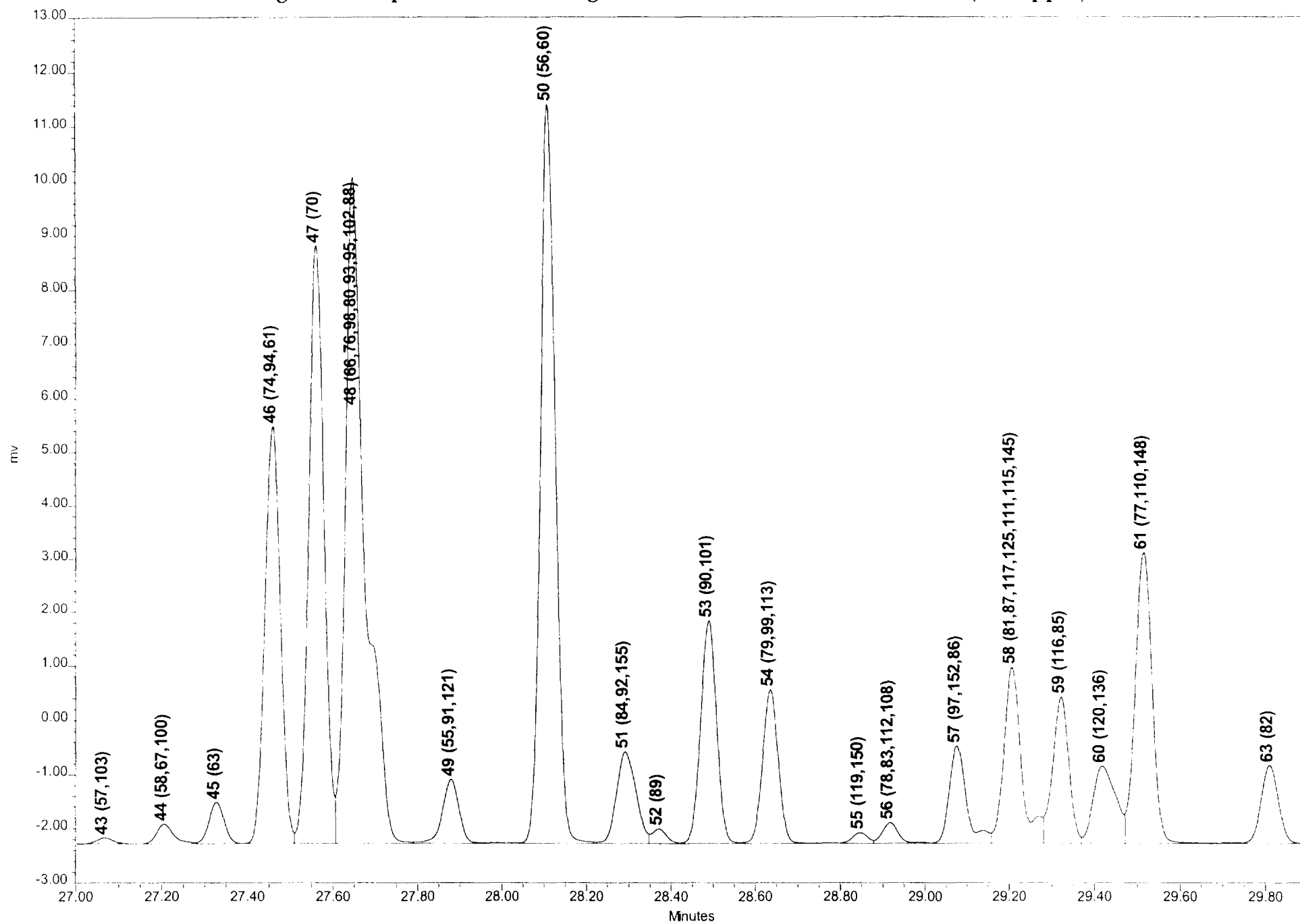
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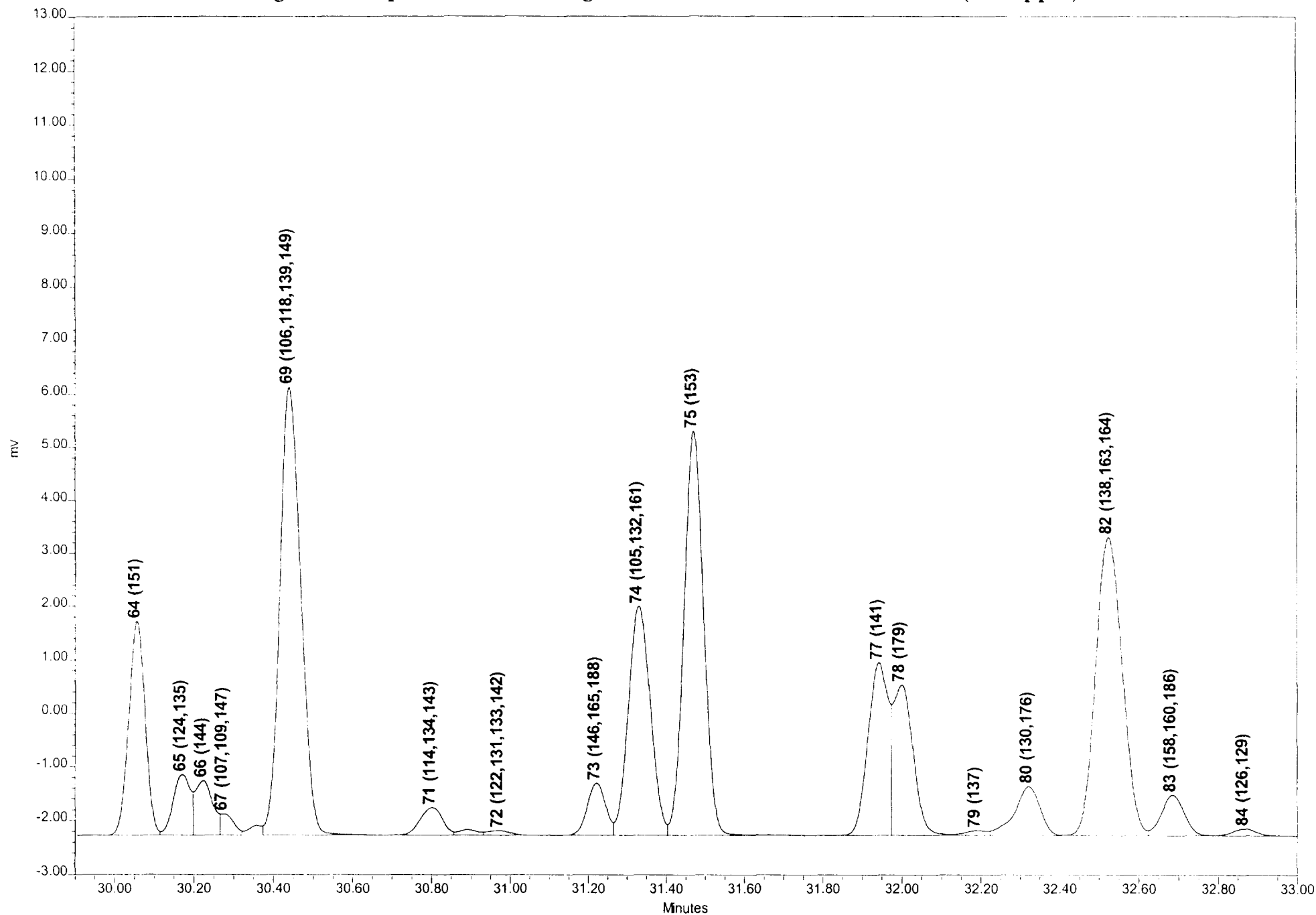
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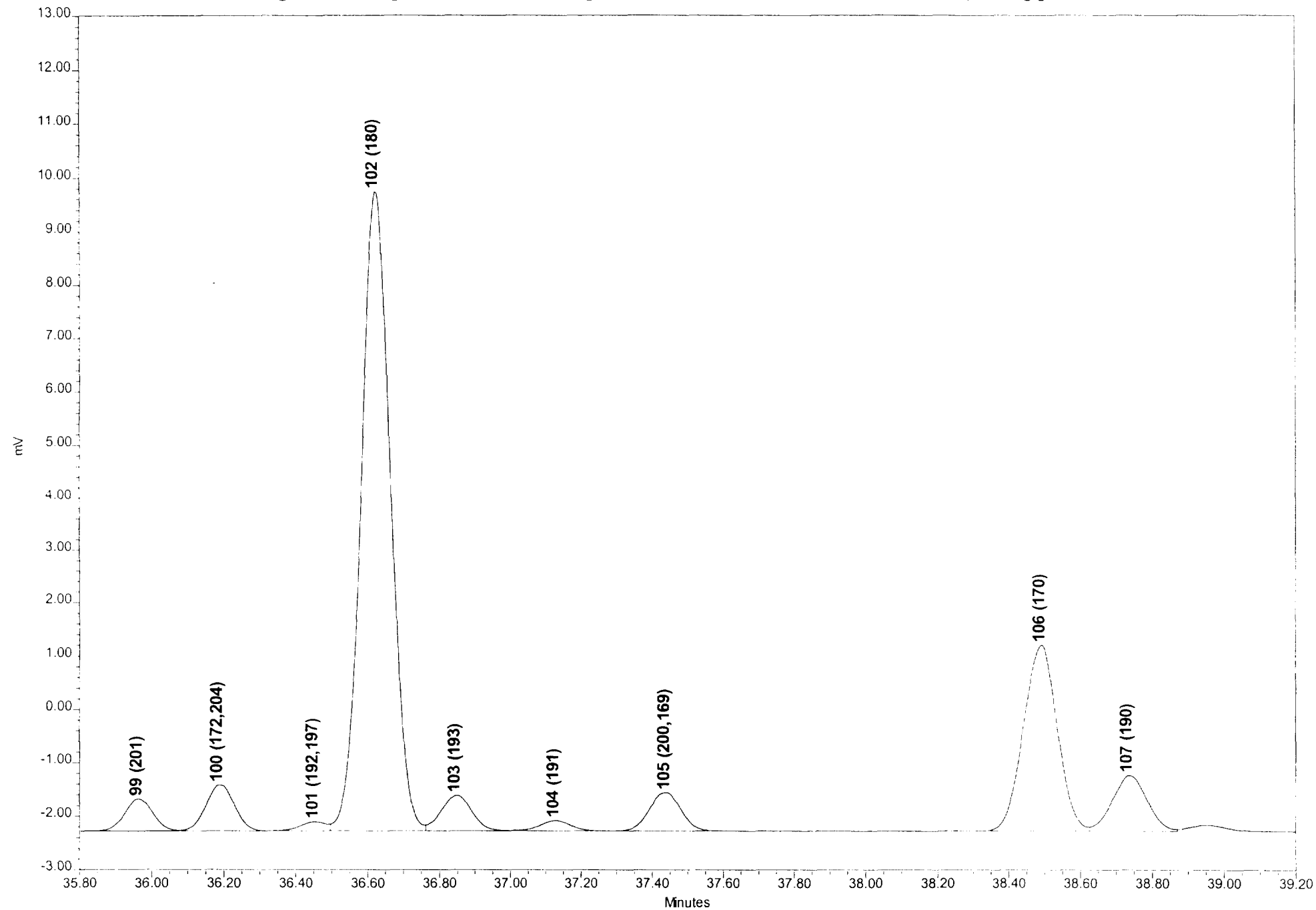
Congener Composition Chromatogram of Aroclor Calibration Mixture (6.27 ppm)



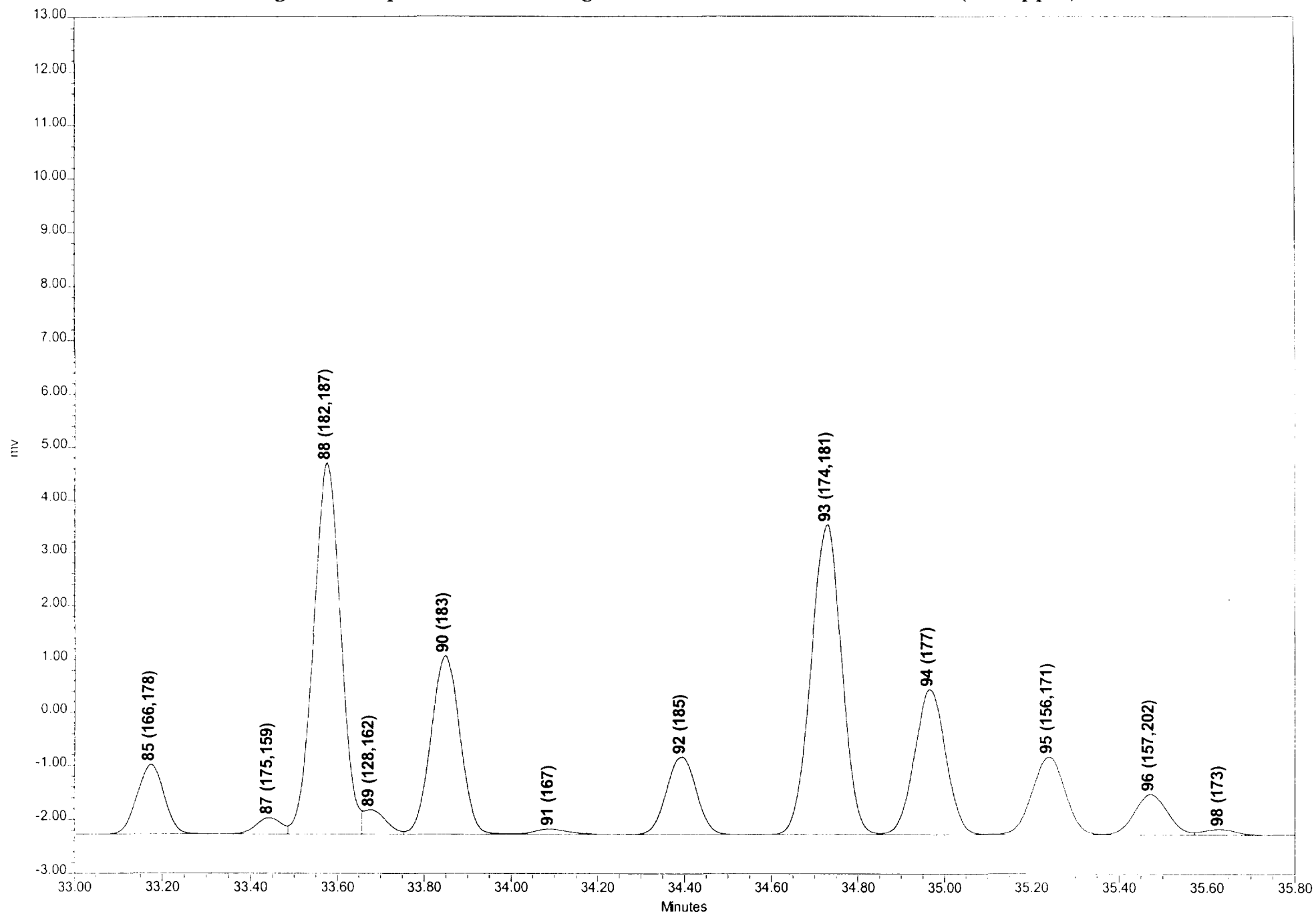
Congener Composition Chromatogram of Aroclor Calibration Mixture (6.27 ppm)



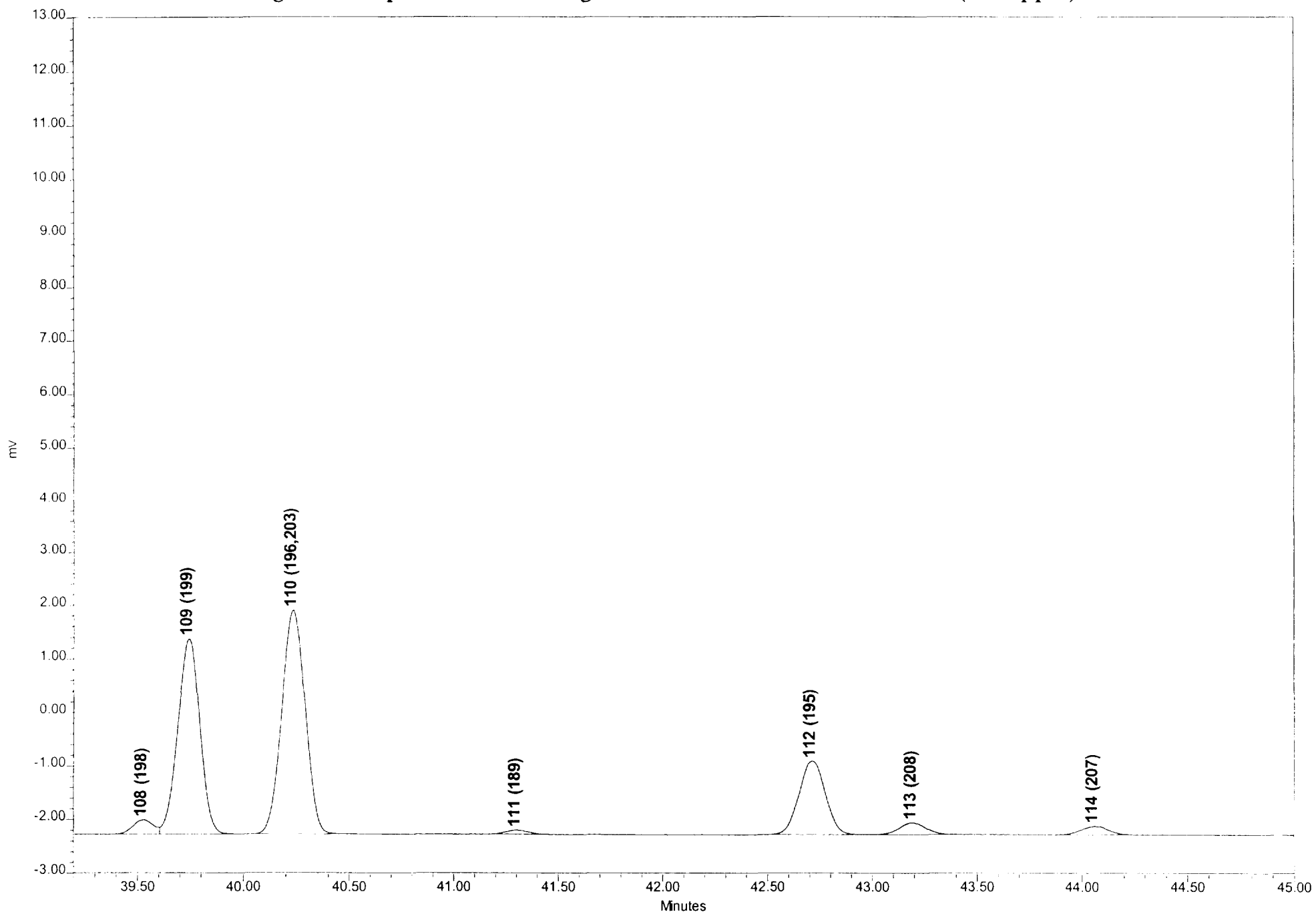
Congener Composition Chromatogram of Aroclor Calibration Mixture (6.27 ppm)



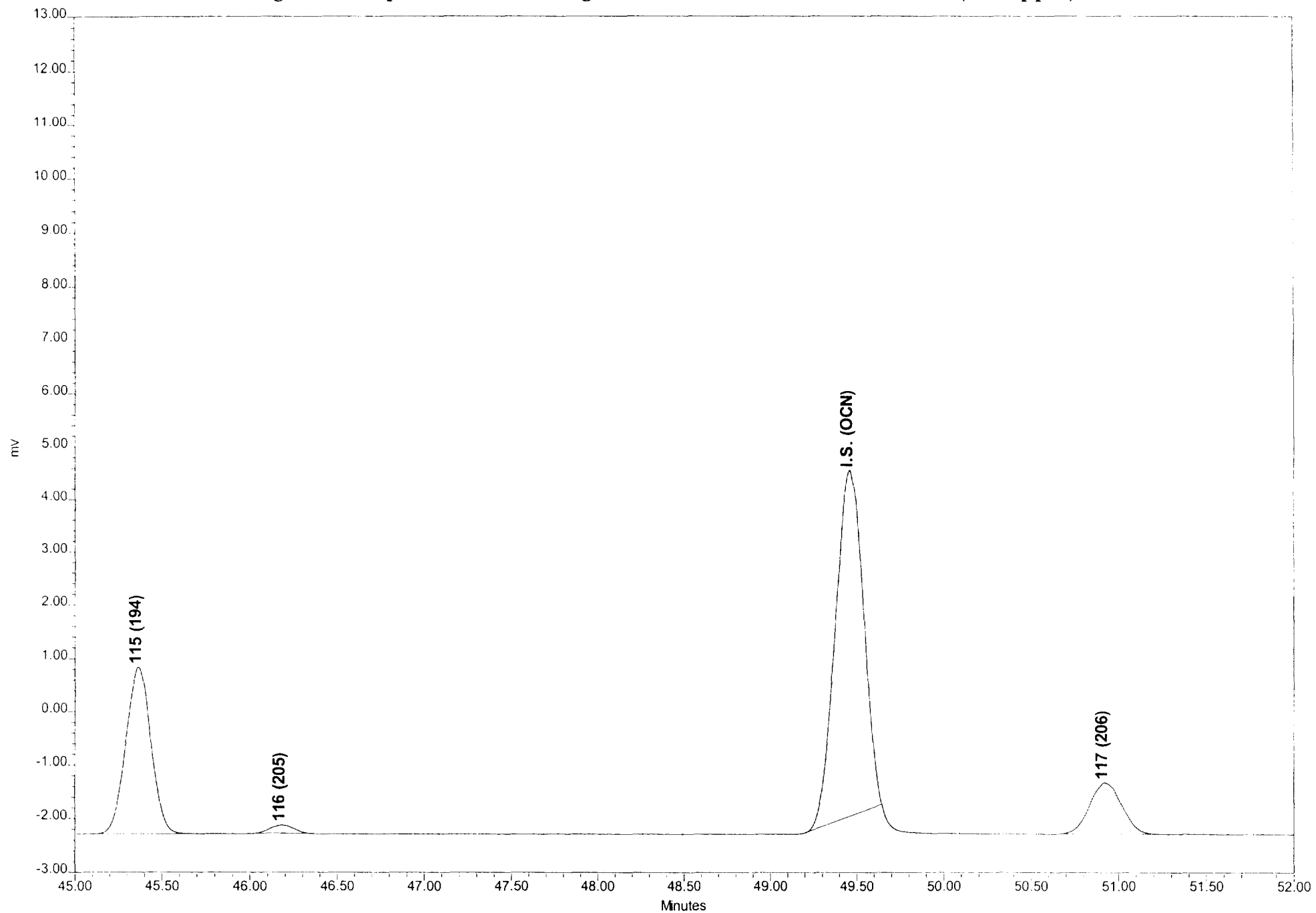
Congener Composition Chromatogram of Aroclor Calibration Mixture (6.27 ppm)



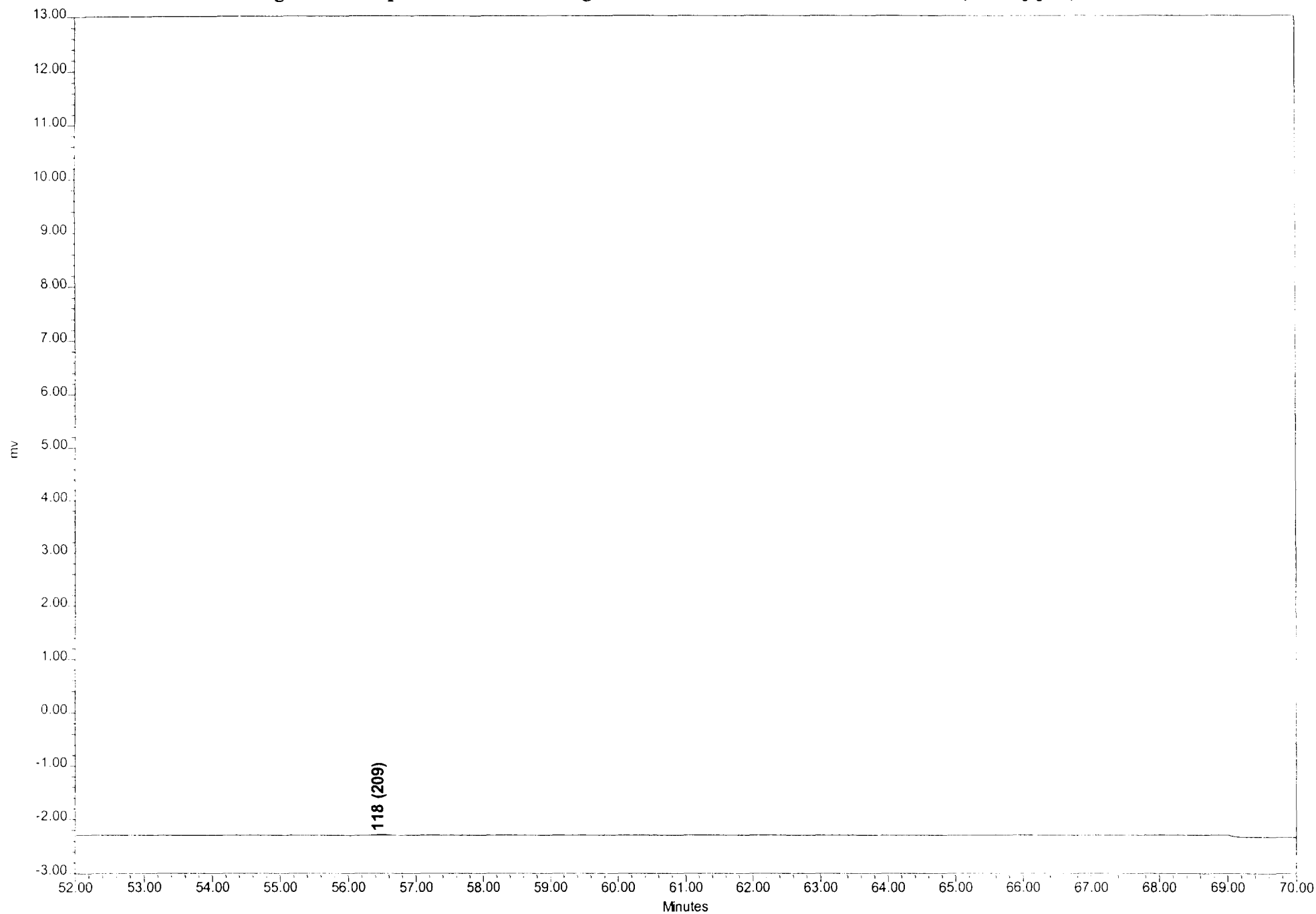
Congener Composition Chromatogram of Aroclor Calibration Mixture (6.27 ppm)



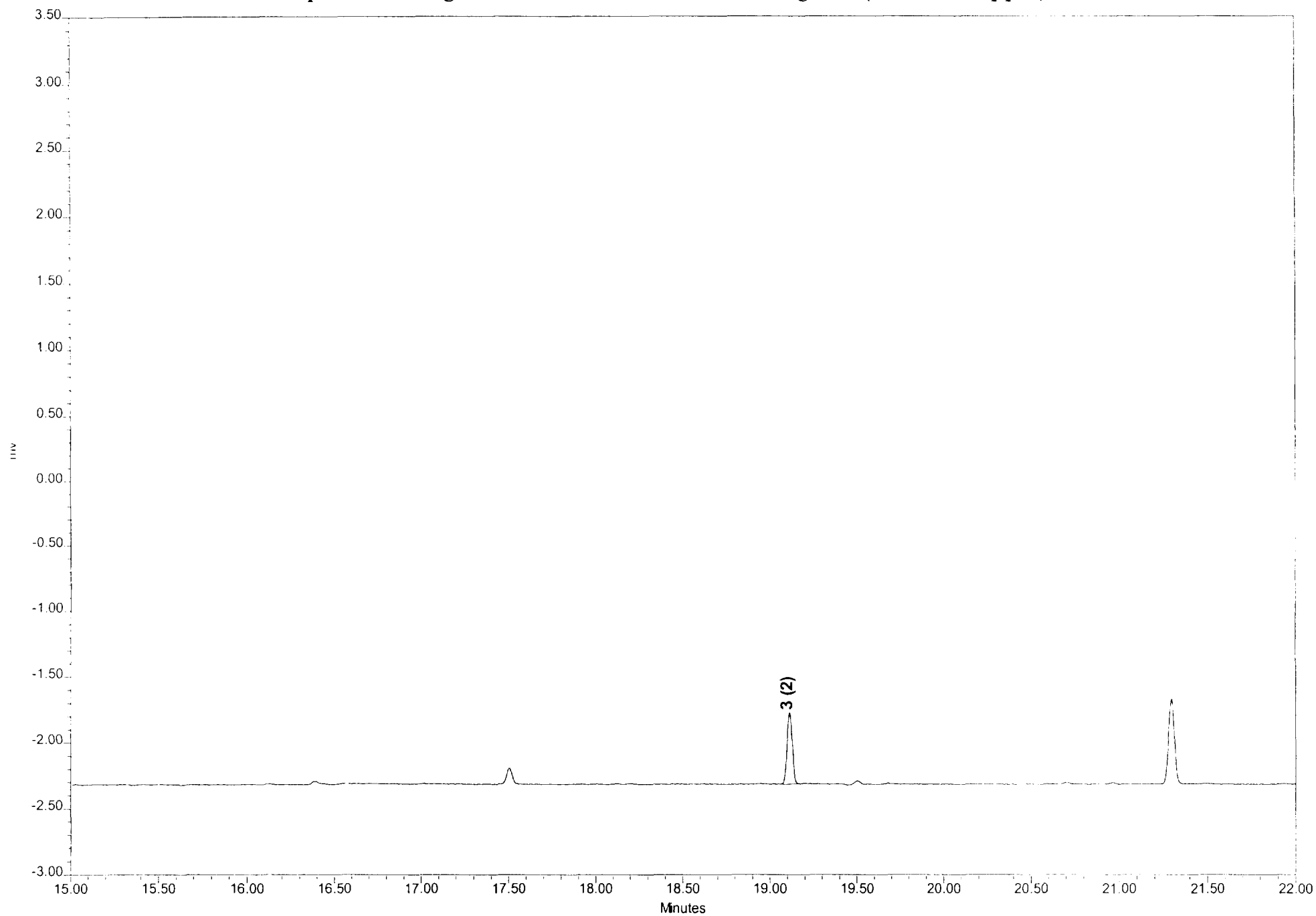
Congener Composition Chromatogram of Aroclor Calibration Mixture (6.27 ppm)



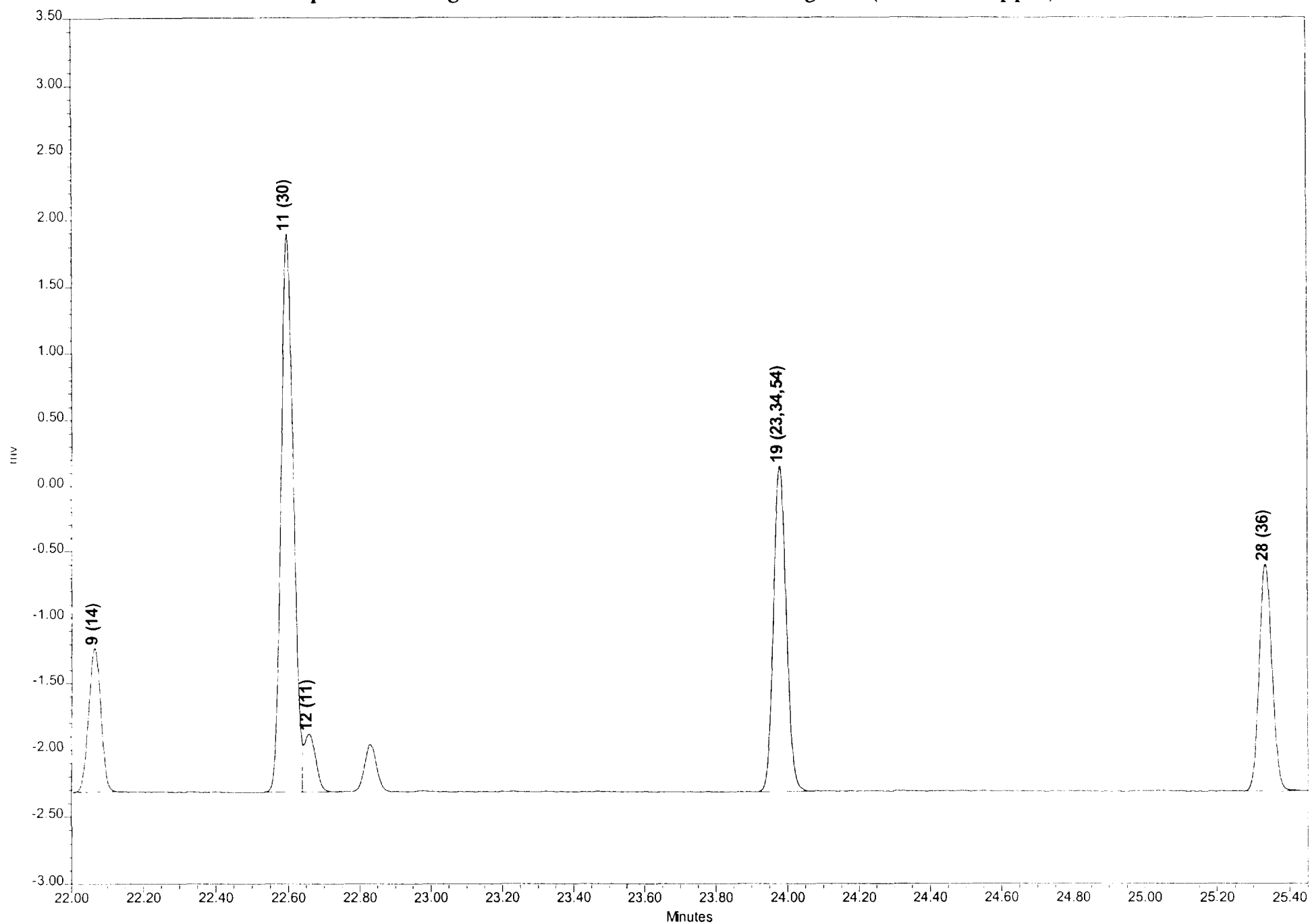
Congener Composition Chromatogram of Aroclor Calibration Mixture (6.27 ppm)



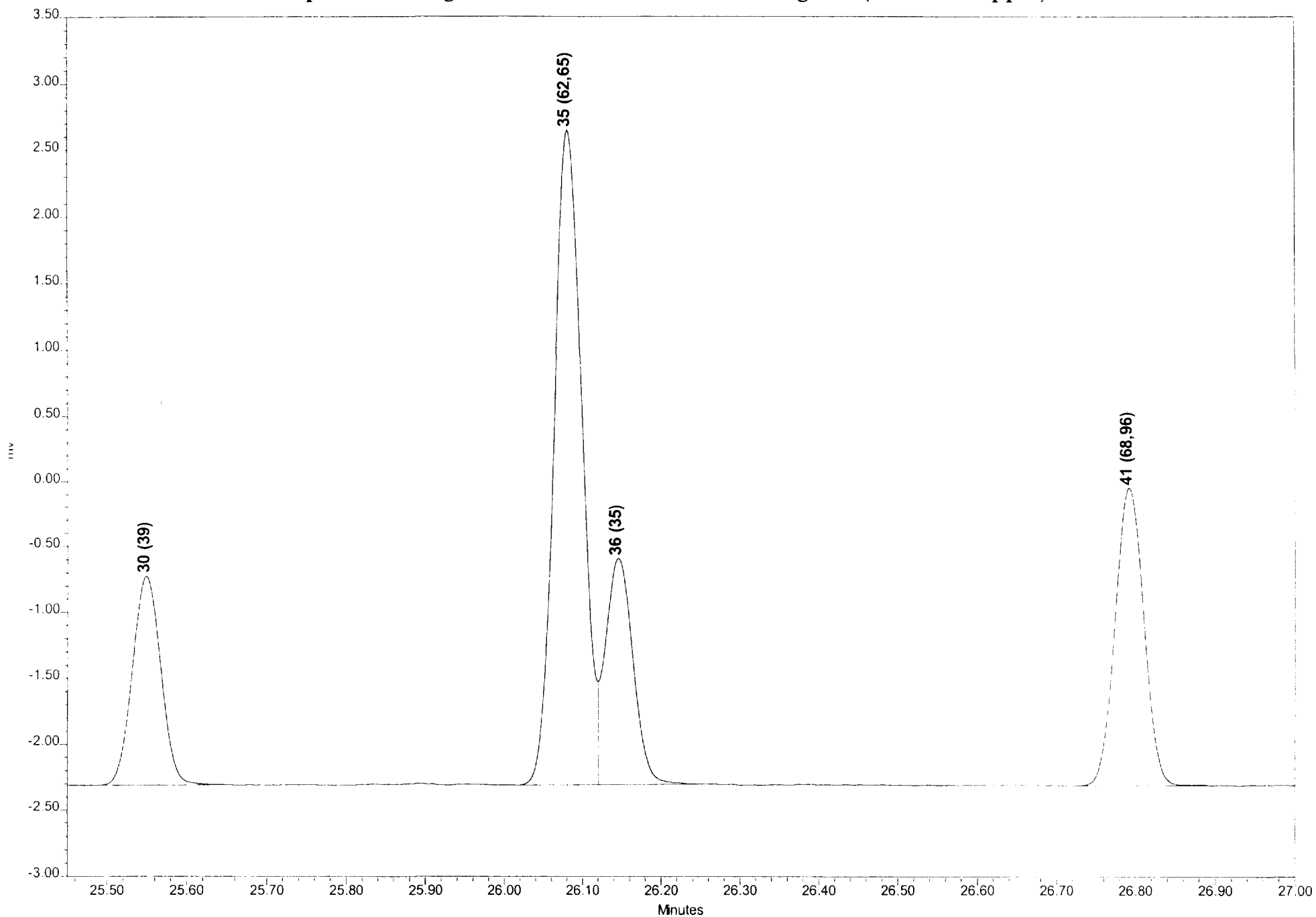
Independent Congener Calibration Mix I Chromatogram (2.00/0.050 ppm)



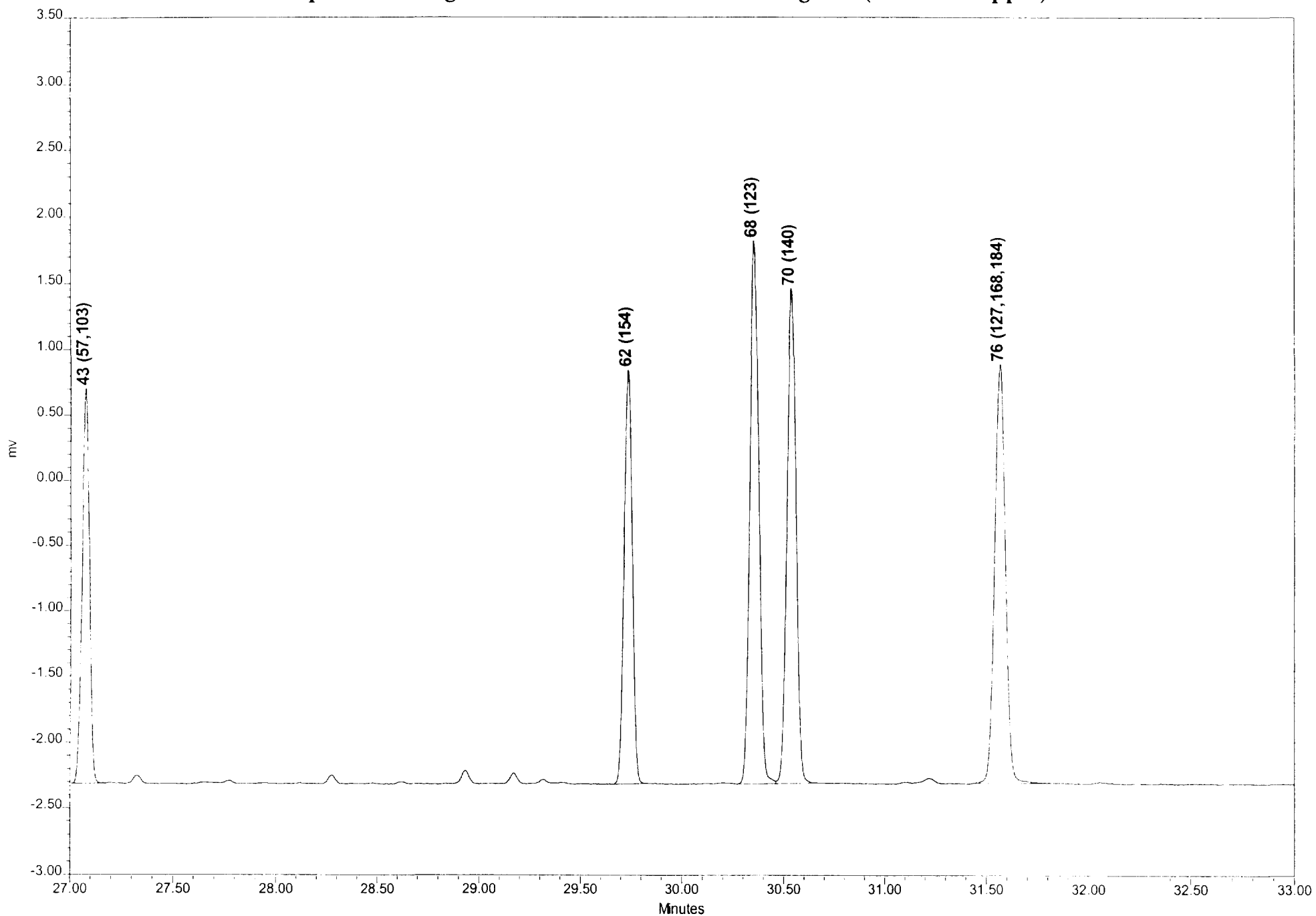
Independent Congener Calibration Mix I Chromatogram (2.00/0.050 ppm)



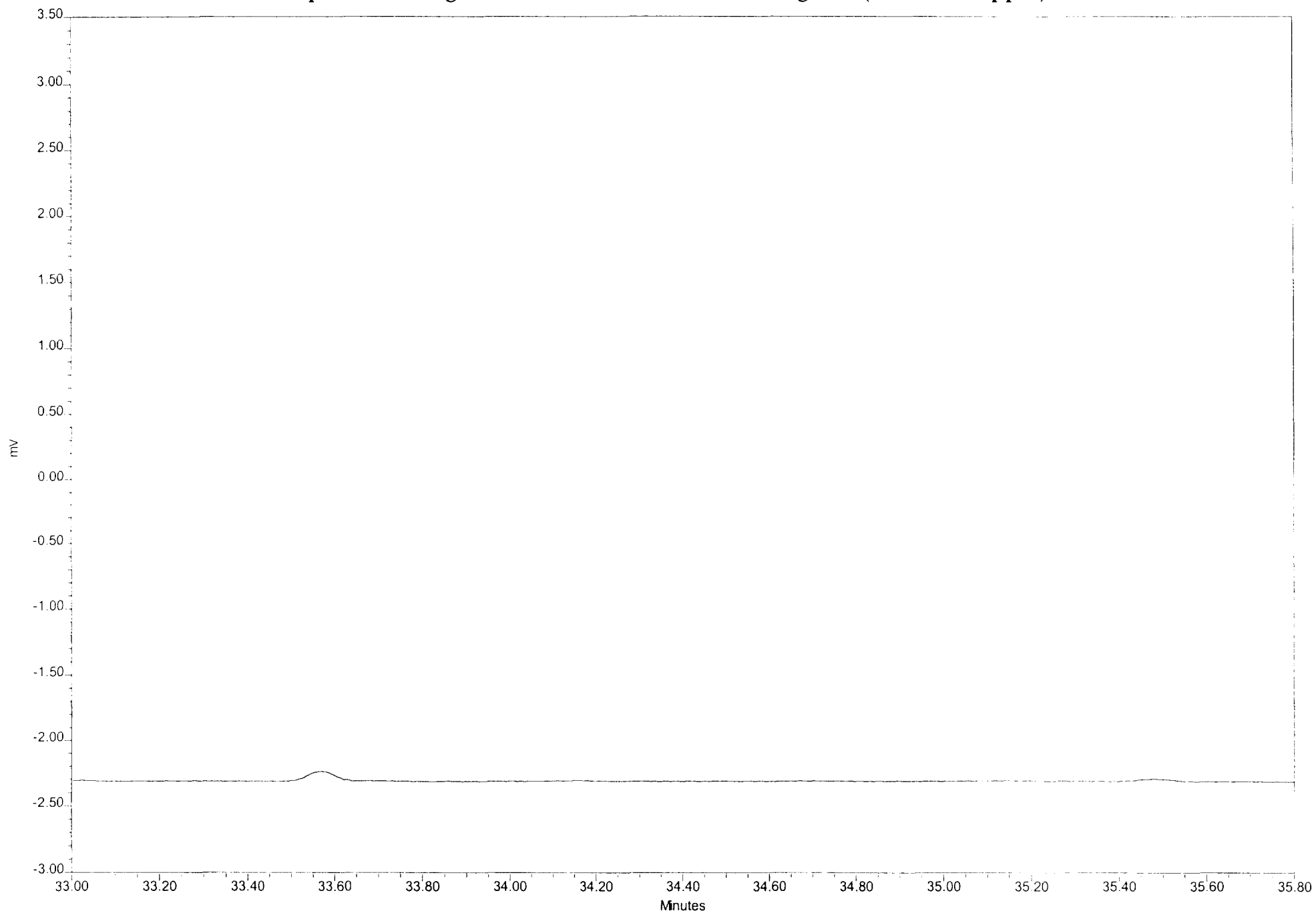
Independent Congener Calibration Mix I Chromatogram (2.00/0.050 ppm)



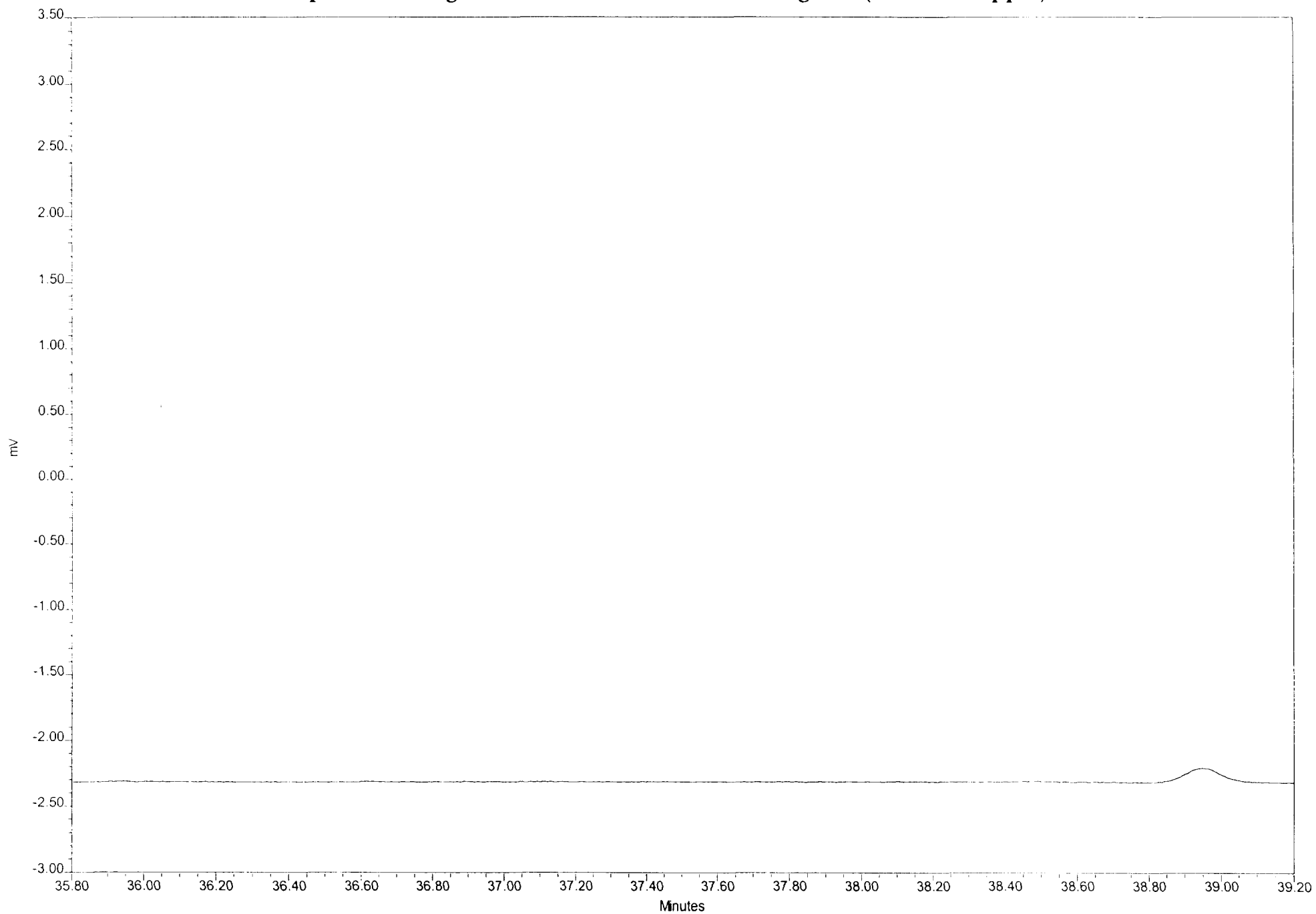
Independent Congener Calibration Mix I Chromatogram (2.00/0.050 ppm)



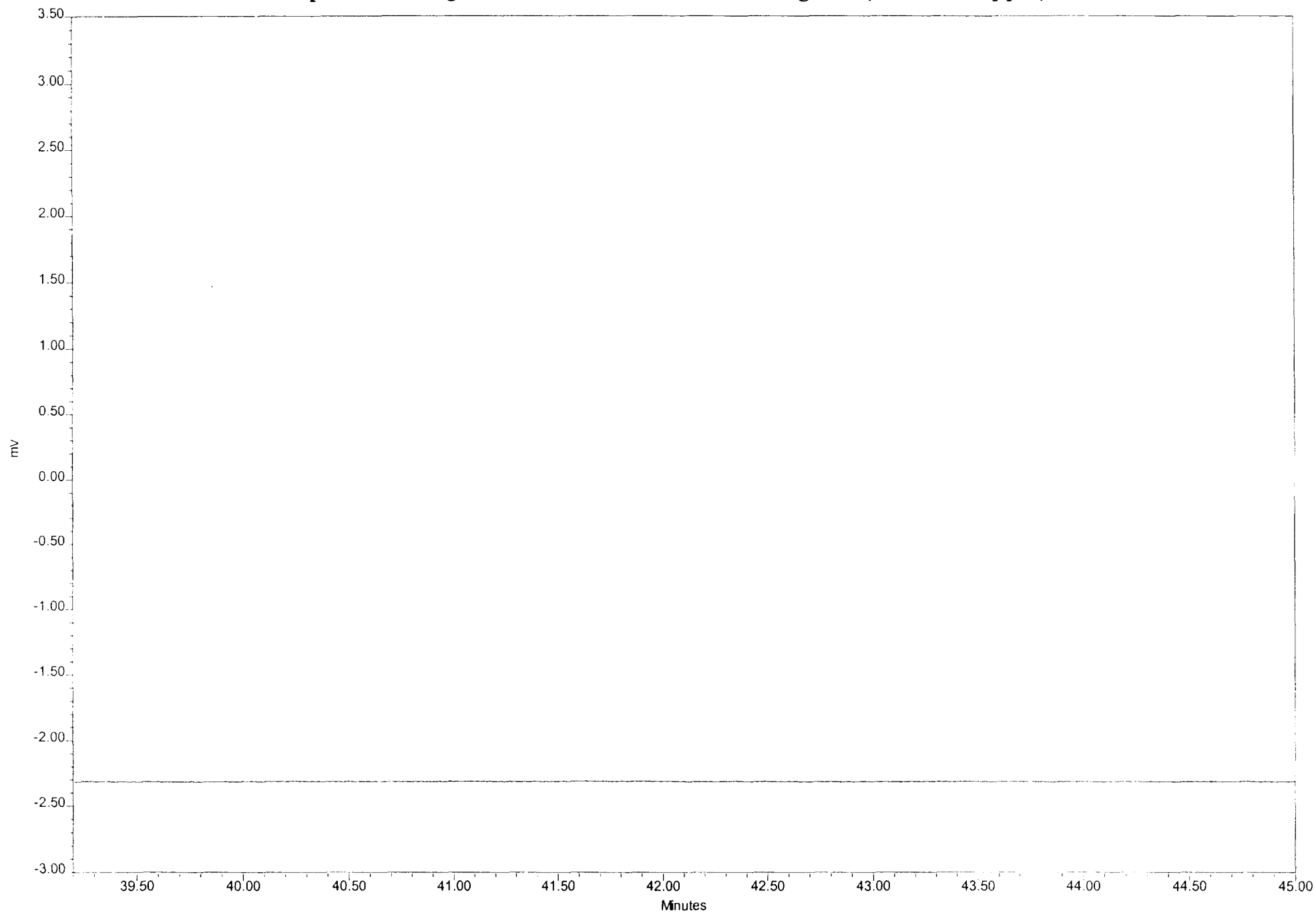
Independent Congener Calibration Mix I Chromatogram (2.00/0.050 ppm)



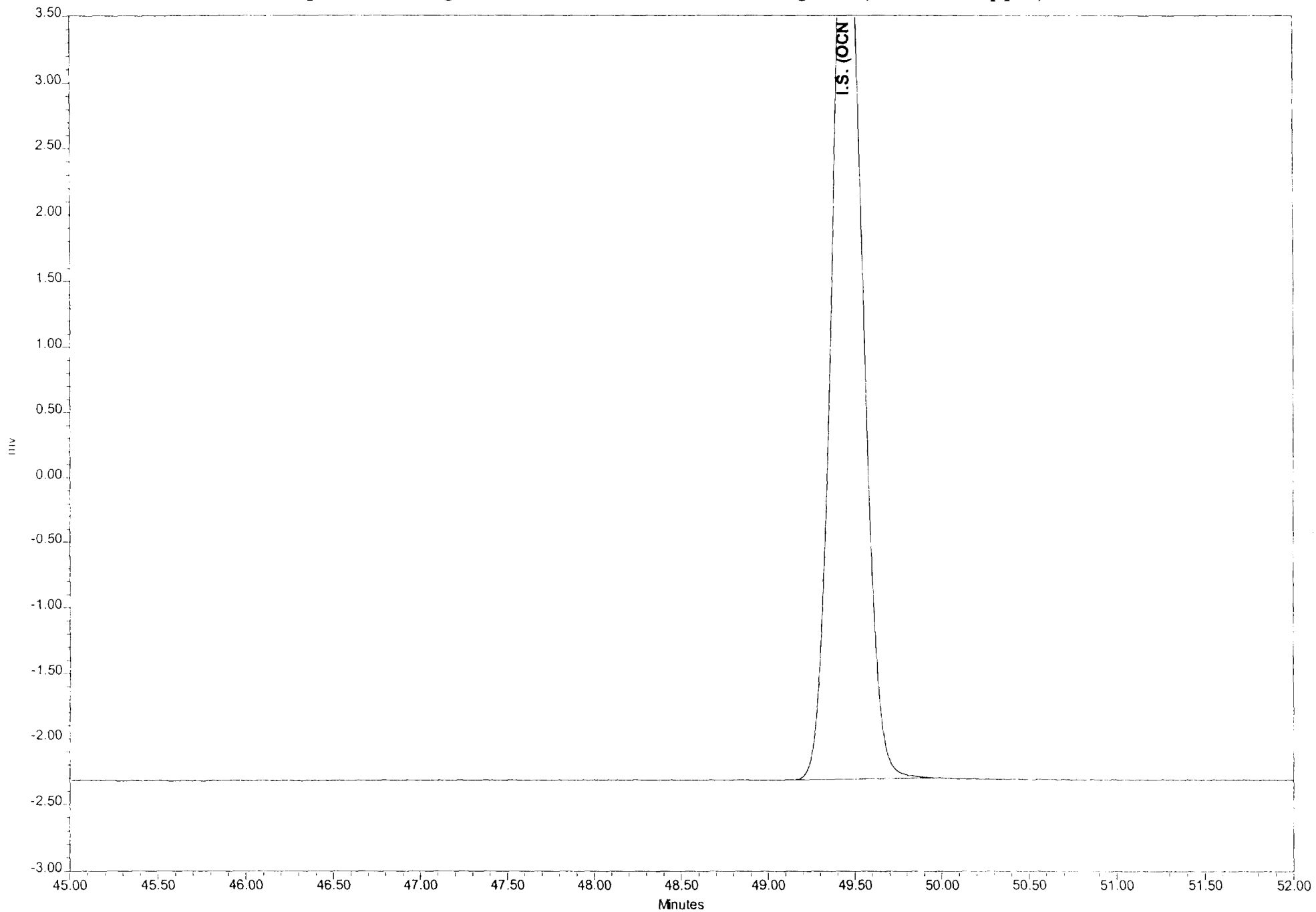
Independent Congener Calibration Mix I Chromatogram (2.00/0.050 ppm)



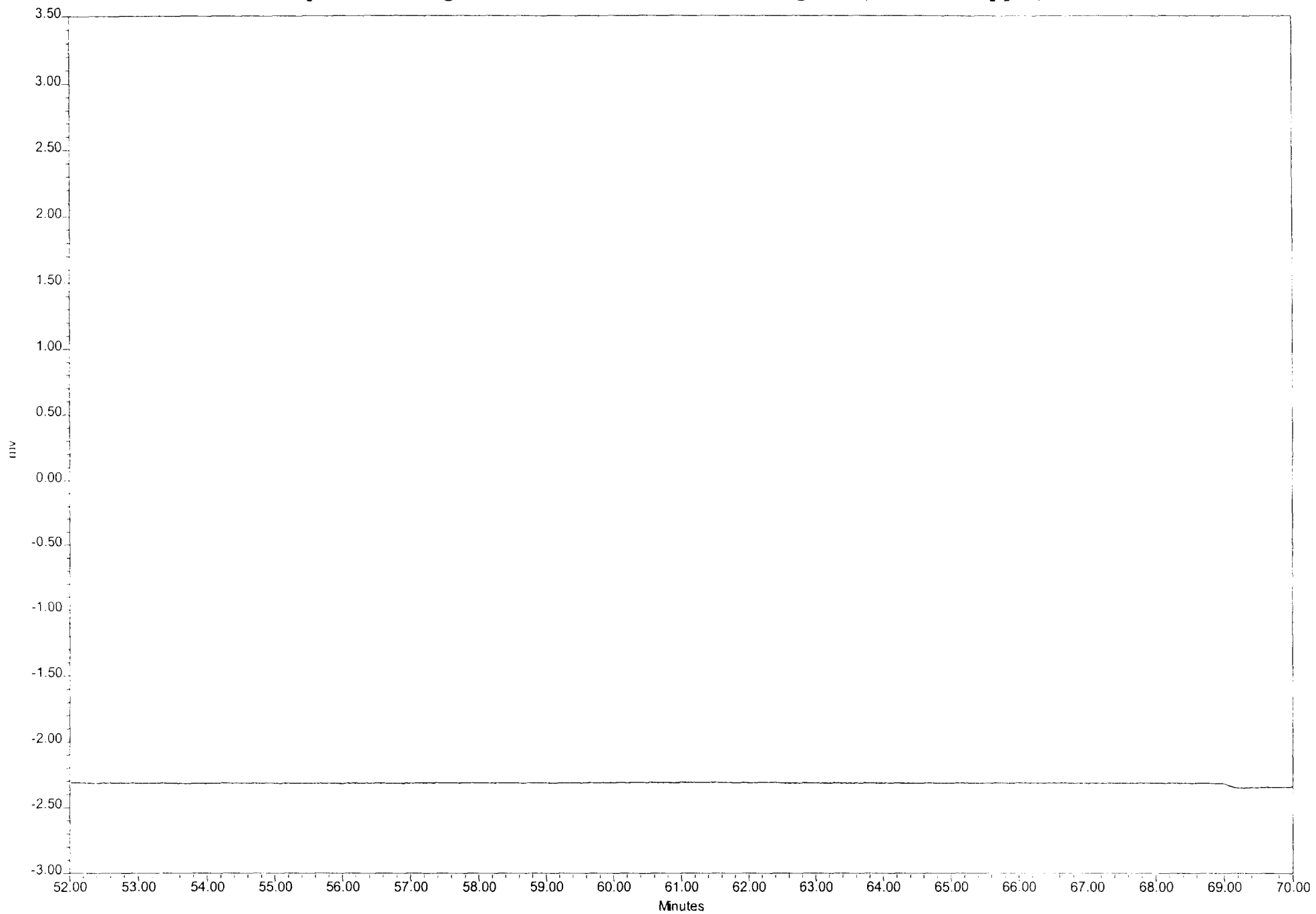
Independent Congener Calibration Mix I Chromatogram (2.00/0.050 ppm)



Independent Congener Calibration Mix I Chromatogram (2.00/0.050 ppm)



Independent Congener Calibration Mix I Chromatogram (2.00/0.050 ppm)



APPENDIX C

Example of Reporting Format

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

NEA File Name : M:\MIL32EXP\LIMSGC9\QEAH2OS\HC0720E.ARS

Customer : Customer Name

Sample Description : PER CHK STD 1.27 PPM

Comment : This is the comment line.

Date Acquired : 07/25/1999 6:02:11 AM

Total PCBs in sample = 1,280 PPB

PCB Homolog Distribution

Homologs	Weight %	Mole %
Mono	10.06	15.07
Di	13.44	16.94
Tri	16.70	18.30
Tetra	21.41	20.77
Penta	8.87	7.67
Hexa	8.32	6.57
Hepta	13.31	9.53
Octa	7.29	4.79
Nona	0.60	0.37
Deca	0.01	0.00

Nominal 'Aroclor' Distribution

Aroclor	Indicator Peak (PK # / IUPAC #)	Amount PPB	Percent Sediment	Biota
A1221	2/001	78.2433	37.9	30.2
A1242	23+24/31+28	56.6203	27.4	21.9
A1254SED	61/110	17.4922	8.5	
A1254BIO	***/###	69.6382		26.9
A1260	102/180	40.9458	19.8	15.8
A1268	115/194	13.3999	6.5	5.2
***/### : 69+75+82/149+153+138				

Ortho CI / biphenyl Residue = 1.59

Meta + Para CI / biphenyl Residue = 2.14

TOTAL CI / biphenyl Residue = 3.73

PCB Congener Amount Report

NEA File Name : M:\MIL32EXP\LIMSGC9\QEAH2OS\HC0720E.ARS

Customer : Customer Name
Sample Description : PER CHK STD 1.27 PPM

Comment : This is the comment line.

Date Acquired : 07/25/1999 6:02:11 AM

Type for Mixed Peak Deconvolution = S

DB-1 Peak Number ¹	Retention Time	Molecular Weight	Peak Area	Amount PPB	Picomoles/ml Sample
2	17.7	188.7	883.0	78.24330	414.64388
3	19.1	188.7			
4	19.2	188.7	281.0	50.62299	268.27234
5	20.0	223.1	692.0	23.14727	103.75289
6	21.0	223.1	2116.0	9.27642	41.57965
7	21.3	223.1	1616.0	15.12691	67.80327
8	21.5	223.1	6354.0	113.42413	508.40040
9	22.1	223.1			
10	22.2	257.5	348.0	2.03863	7.91701
11	22.6	257.5			
12	22.7	223.1			
13	22.9	223.1	504.0	3.00498	13.46921
14	23.0	249.0	5134.0	32.42612	130.22538
15	23.1	257.5	2250.0	27.67384	107.47122
16	23.4	257.5	595.0	1.87339	7.27530
17	23.6	257.5	4549.0	29.71349	115.39219
19	24.0	267.9	33.0	0.18177	0.67850
20	24.2	257.5	86.0	0.28132	1.09250
21	24.3	257.5	1218.0	5.82654	22.62734
22	24.4	257.5	714.0	2.43861	9.47033
23	24.5	257.5	7292.0	30.42972	118.17367
24	24.6	257.5	8573.0	26.19055	101.71087
25	24.9	259.5	6331.0	30.02271	115.69445
26	25.1	258.7	4528.0	20.82710	80.50676
27	25.3	292.0	1615.0	7.64903	26.19531
28	25.4	257.5			
29	25.5	292.0	632.0	3.30965	11.33442
30	25.6	257.5			
31	25.8	292.0	6006.0	42.80030	146.57637
32	25.9	292.0	5284.0	18.07799	61.91092
33	26.0	292.0	2414.0	5.68864	19.48164
34	26.0	292.0	2385.0	8.01321	27.44250
35	26.2	292.0			

DB-1 Peak Number ¹	Retention Time	Molecular Weight	Peak Area	Amount PPB	Picomoles/ml Sample
36	26.2	257.5	60.0	0.47238	1.83449
37	26.4	292.0	7938.0	33.25945	113.90223
38	26.5	272.4	4898.0	24.33481	89.33484
39	26.7	292.0	9773.0	27.35511	93.68188
41	26.9	326.4	91.0	0.53300	1.63297
42	27.0	292.0	2261.0	8.20674	28.10527
43	27.2	298.9	81.0	0.35617	1.19160
44	27.3	298.9	365.0	0.86579	2.89659
45	27.4	292.0	600.0	1.42025	4.86387
46	27.5	292.0	5391.0	11.03331	37.78531
47	27.7	292.0	9486.0	24.20346	82.88856
48	27.7	293.5	12749.0	48.52383	165.32821
49	28.0	324.7	1245.0	4.37350	13.46936
50	28.2	292.0	9977.0	20.88990	71.54075
51	28.4	326.4	2440.0	17.82949	54.62466
52	28.5	326.4	223.0	0.80736	2.47353
53	28.6	326.4	4235.0	15.61744	47.84755
54	28.7	326.4	2236.0	4.99636	15.30748
55	28.9	326.4	149.0	0.17855	0.54703
56	29.0	326.4	470.0	1.50917	4.62368
57	29.2	326.4	2132.0	5.04432	15.45441
58	29.3	326.4	3749.0	10.04761	30.78312
59	29.4	326.4	2214.0	4.74721	14.54415
60	29.5	360.9	1763.0	5.45281	15.10892
61	29.6	315.8	5542.0	17.49220	55.39012
62	29.8	360.9			
63	29.9	326.4	1668.0	3.91069	11.98128
64	30.2	360.9	4301.0	13.71831	38.01139
65	30.3	350.5	1303.0	2.41984	6.90397
66	30.3	360.9	1137.0	4.87926	13.51970
67	30.4	336.8	393.0	0.96651	2.86969
68	30.5	326.4	128.0	0.33579	1.02877
69	30.6	337.5	10477.0	29.00927	85.95339
70	30.7	360.9			
71	30.9	347.8	683.0	1.39061	3.99830
72	31.1	336.8	112.0	0.18198	0.54032
73	31.3	360.9	1170.0	2.99337	8.29418
74	31.4	347.8	5845.0	10.17993	29.26949
75	31.6	360.9	8883.0	21.00865	58.21183
76	31.7	360.9			
77	32.1	360.9	4088.0	12.19844	33.80006
78	32.1	395.3	3706.0	13.08926	33.11222
79	32.3	360.9	164.0	0.81556	2.25979
80	32.4	360.9	1748.0	2.28914	6.34287
82	32.6	360.9	8875.0	19.62024	54.36475
83	32.8	360.9	1013.0	1.75607	4.86581
84	33.0	360.9	176.0	0.09053	0.25085
85	33.3	395.3	2110.0	8.79149	22.24005
87	33.5	395.3	429.0	1.36234	3.44634
88	33.7	395.3	11168.0	27.57678	69.76165

DB-1 Peak Number ¹	Retention Time	Molecular Weight	Peak Area	Amount PPB	Picomoles/ml Sample
89	33.7	360.9	569.0	0.76971	2.13275
90	33.9	395.3	5472.0	12.81202	32.41088
91	34.2	360.9	115.0	0.23802	0.65952
92	34.5	394.3	2588.0	3.61858	9.17723
93	34.8	394.3	10229.0	24.31356	61.66259
94	35.0	394.3	4951.0	13.21653	33.51897
95	35.3	382.2	2712.0	5.91989	15.48898
96	35.5	429.8	1633.0	0.56220	1.30805
98	35.7	395.3	195.0	0.27090	0.68530
99	36.0	429.8	1260.0	3.10479	7.22380
100	36.2	395.3	1786.0	4.24207	10.73127
101	36.5	429.8	282.0	0.73293	1.70528
102	36.6	395.3	23213.0	40.94582	103.58163
103	36.9	395.3	1454.0	3.11639	7.88361
104	37.1	395.3	373.0	0.76914	1.94571
105	37.4	429.8	1633.0	3.21654	7.48381
106	38.5	395.3	7971.0	9.27838	23.47174
107	38.7	395.3	2511.0	3.00916	7.61235
108	39.5	429.8	700.0	0.87465	2.03502
109	39.7	429.8	10344.0	33.83315	78.71836
110	40.2	429.8	12022.0	32.89431	76.53399
111	41.2	395.3	249.0	0.31388	0.79403
112	42.6	429.8	4226.0	3.87268	9.01042
113	43.1	464.2	828.0	1.97978	4.26493
114	43.9	464.2	512.0	0.59306	1.27760
115	45.2	429.8	11600.0	13.39986	31.17697
116	46.0	429.8	573.0	0.80195	1.86587
117	50.7	464.2	4939.0	5.11337	11.01545
118	56.2	498.6	96.0	0.09025	0.18101

Concentration = 1,280 PPB

Total Nanomoles = 4.53

Average Molecular Weight = 282.6

Number of Calibrated Peaks Found = 102

Internal Standard Retention Time = 49.26 Minutes

Internal Standard Peak Area = 136033.0

Congener Weight and Mole Report

NEA File Name : M:\MIL32EXP\LIMSGC9\QEAH2OS\HC0720E.ARS

Customer : Customer Name

Sample Description : PER CHK STD 1.27 PPM

Comment : This is the comment line.

Date Acquired : 07/25/1999 6:02:11 AM

Type for Mixed Peak Deconvolution = S

DB-1 Peak Number ¹	Retention Time	T-CL:O-CL	IUPAC # ²	RRT	Congeners ³	Weight Percent	Mole Percent	DB-1 Peak Number ¹
2	17.69	1:1	001	.1544	2	6.111	9.152	2
3	19.10	1:0	002	.1937	3	-	-	3
4	19.23	1:0	003	.1975	4	3.954	5.921	4
5	19.98	2:2	004 010	.2245	2-2 ; 26	1.808	2.290	5
6	20.98	2:1	007 009	.2566	24 ; 25	0.725	0.918	6
7	21.31	2:1	006	.2709	2-3	1.181	1.496	7
8	21.51	2:1	005 008	.2785	23 ; 2-4	8.859	11.221	8
9	22.09	2:0	014	.2973	35	-	-	9
10	22.16	3:3	019	.3045	26-2	0.159	0.175	10
11	22.63	3:2	030	.3165	246	-	-	11
12	22.69	2:0	011	.3238	3-3	-	-	12
13	22.88	2:0	012 013	.3297	34 ; 3-4	0.235	0.297	13
14	23.01	2:0 3:2	015 018	.3387	4-4 ; 25-2	2.533	2.874	14
15	23.09	3:2	017	.3398	24-2	2.161	2.372	15
16	23.37	3:2	024 027	.3508	236 ; 26-3	0.146	0.161	16
17	23.60	3:2	016 032	.3625	23-2 ; 26-4	2.321	2.547	17
19	24.02	3:1 4:4	023 034 054	.3800	235 ; 35-2 ; 26-26	0.014	0.015	19
20	24.19	3:1	029	.3820	245	0.022	0.024	20
21	24.30	3:1	026	.3911	25-3	0.455	0.499	21
22	24.37	3:1	025	.3937	24-3	0.190	0.209	22
23	24.55	3:1	031	.4024	25-4	2.377	2.608	23
24	24.59	3:1 4:3	028 050	.4031	24-4 ; 246-2	2.046	2.245	24
25	24.89	3:1 4:3	020 021 033 053	.4170	23-3 ; 234 ; 34-2 ; 25-26	2.345	2.553	25
26	25.09	3:1 4:3	022 051	.4267	23-4 ; 24-26	1.627	1.777	26
27	25.28	4:3	045	.4334	236-2	0.597	0.578	27
28	25.40	3:0	036	.4379	35-3	-	-	28
29	25.51	4:3	046	.4450	23-26	0.258	0.250	29
30	25.62	3:0	039	.4488	35-4	-	-	30
31	25.76	4:2	052 069 073	.4554	25-25 ; 246-3 ; 26-35	3.343	3.235	31
32	25.90	4:2	043 049	.4610	235-2 ; 24-25	1.412	1.366	32
33	25.99	4:2	038 047	.4639	345 ; 24-24	0.444	0.430	33
34	26.04	4:2	048 075	.4651	245-2 ; 246-4	0.626	0.606	34
35	26.15	4:2	062 065	.4865	2346 ; 2356	-	-	35
36	26.22	3:0	035	.4738	34-3	0.037	0.040	36
37	26.36	5:4 4:2	104 044	.4832	246-26 ; 23-25	2.598	2.514	37
38	26.47	3:0 4:2	037 042 059	.4870	34-4 ; 23-24 ; 236-3	1.901	1.972	38
39	26.75	4:2	041 064 071 072	.4990	234-2 ; 236-4 ; 26-34 ; 25-35	2.137	2.068	39
41	26.88	5:4	068 096	.5057	24-35 ; 236-26	0.042	0.036	41
42	26.95	4:2	040	.5102	23-23	0.641	0.620	42
43	27.15	4:1 5:3	057 103	.5155	235-3 ; 246-25	0.028	0.026	43

DB-1 Peak Number ¹	Retention Time	T-CL:O-CL	IUPAC # ²	RRT	Congeners ³	Weight Percent	Mole Percent	DB-1 Peak Number ¹
44	27.29	4:1 5:3	058 067 100	.5212	23-35 ; 245-3 ; 246-24	0.068	0.064	44
45	27.41	4:1	063	.5267	235-4	0.111	0.107	45
46	27.55	4:1 5:3	074 094 061	.5340	245-4 ; 235-26 ; 2345	0.862	0.834	46
47	27.65	4:1	070	.5407	25-34	1.890	1.829	47
48	27.74	4:1 5:3	066 076 098 080 093 095 102 088	.5447	24-34 ; 345-2 ; 246-23 ; 35-35 ; 2356-2 ; 236-25 ; 245-26 ; 2346-2	3.790	3.649	48
49	27.97	4:1 5:3	055 091 121	.5549	234-3 ; 236-24 ; 246-35	0.342	0.297	49
50	28.21	4:1	056 060	.5676	23-34 ; 234-4	1.632	1.579	50
51	28.39	5:3 6:4	084 092 155	.5666	236-23 ; 235-25 ; 246-246	1.393	1.206	51
52	28.47	5:3	089	.5779	234-26	0.063	0.055	52
53	28.59	5:2	090 101	.5814	235-24 ; 245-25	1.220	1.056	53
54	28.74	5:2	079 099 113	.5880	34-35 ; 245-24 ; 236-35	0.390	0.338	54
55	28.94	5:2 6:4	119 150	.5969	246-34 ; 236-246	0.014	0.012	55
56	29.02	5:2	078 083 112 108	.6029	345-3 ; 235-23 ; 2356-3 ; 2346-3	0.118	0.102	56
57	29.18	5:2 6:4	097 152 086	.6062	245-23 ; 2356-26 ; 2345-2	0.394	0.341	57
58	29.31	5:2	081 087 117 125 111 115 145	.6175	345-4 ; 234-25 ; 2356-4 ; 345-26 ; 235-35 ; 2346-4 ; 2346-26	0.785	0.679	58
59	29.42	5:2	116 085	.6224	23456 ; 234-24	0.371	0.321	59
60	29.52	6:4	120 136	.6257	245-35 ; 236-236	0.426	0.333	60
61	29.62	4:0 5:2	077 110 148	.6295	34-34 ; 236-34 ; 235-246	1.366	1.223	61
62	29.84	6:3	154	.6349	245-246	-	-	62
63	29.92	5:2	082	.6453	234-23	0.305	0.264	63
64	30.17	6:3	151	.6499	2356-25	1.071	0.839	64
65	30.28	5:1 6:3	124 135	.6563	345-25 ; 235-236	0.189	0.152	65
66	30.34	6:3	144	.6584	2346-25	0.381	0.298	66
67	30.39	5:1 6:3	107 109 147	.6628	234-35 ; 235-34 ; 2356-24	0.075	0.063	67
68	30.47	5:1	123	.6658	345-24	0.026	0.023	68
69	30.55	5:1 6:3	106 118 139 149	.6672	2345-3 ; 245-34 ; 2346-24 ; 236-245	2.266	1.897	69
70	30.65	6:3	140	.6707	234-246	-	-	70
71	30.91	5:1 6:3	114 134 143	.6796	2345-4 ; 2356-23 ; 2345-26	0.109	0.088	71
72	31.07	5:1 6:3	122 131 133 142	.6871	345-23 ; 2346-23 ; 235-235 ; 23456-2	0.014	0.012	72
73	31.33	6:2	146 165 188	.6955	235-245 ; 2356-35 ; 2356-246	0.234	0.183	73
74	31.44	5:1 6:3	105 132 161	.7035	234-34 ; 234-236 ; 2346-35	0.795	0.646	74
75	31.58	6:2	153	.7036	245-245	1.641	1.285	75
76	31.68	6:2	127 168 184	.7068	345-35 ; 246-345 ; 2346-246	-	-	76
77	32.05	6:2	141	.7203	2345-25	0.953	0.746	77
78	32.11	7:4	179	.7205	2356-236	1.022	0.731	78
79	32.30	6:2	137	.7284	2345-24	0.064	0.050	79
80	32.43	6:2 7:4	130 176	.7329	234-235 ; 2346-236	0.179	0.140	80
82	32.63	6:2	138 163 164	.7403	234-245 ; 2356-34 ; 236-345	1.532	1.200	82
83	32.79	6:2	158 160 186	.7429	2346-34 ; 23456-3 ; 23456-26	0.137	0.107	83
84	32.96	6:2	126 129	.7501	345-34 ; 2345-23	0.007	0.006	84
85	33.27	7:3	166 178	.7537	23456-4 ; 2356-235	0.687	0.491	85
87	33.53	7:3	175 159	.7611	2346-235 ; 2345-35	0.106	0.076	87
88	33.66	7:3	182 187	.7653	2345-246 ; 2356-245	2.154	1.540	88
89	33.74	6:2	128 162	.7761	234-234 ; 235-345	0.060	0.047	89
90	33.93	7:3	183	.7720	2346-245	1.001	0.715	90
91	34.16	6:1	167	.7814	245-345	0.019	0.015	91
92	34.46	7:3	185	.7848	23456-25	0.283	0.203	92
93	34.79	7:3	174 181	.7965	2345-236 ; 23456-24	1.899	1.361	93
94	35.02	7:3	177	.8031	2356-234	1.032	0.740	94
95	35.29	6:1 7:3	156 171	.8105	2345-34 ; 2346-234	0.462	0.342	95
96	35.52	8:4	157 202	.8089	234-345 ; 2356-2356	0.044	0.029	96
98	35.66	7:3	173	.8152	23456-23	0.021	0.015	98
99	36.00	8:4	201	.8197	2346-2356	0.242	0.159	99
100	36.22	7:2	172 204	.8278	2345-235 ; 23456-246	0.331	0.237	100
101	36.48	8:4	192 197	.8293	23456-35 ; 2346-2346	0.057	0.038	101
102	36.65	7:2	180	.8362	2345-245	3.198	2.286	102

DB-1 Peak Number ¹	Retention Time	T-CL:O-CL	IUPAC # ²	RRT	Congeners ³	Weight Percent	Mole Percent	DB-1 Peak Number ¹
103	36.87	7:2	193	.8397	2356-345	0.243	0.174	103
104	37.13	7:2	191	.8447	2346-345	0.060	0.043	104
105	37.44	8:4	200 <i>169</i>	.8494	23456-236 ; 345-345	0.251	0.165	105
106	38.47	7:2	170	.8740	2345-234	0.725	0.518	106
107	38.72	7:2	190	.8740	23456-34	0.235	0.168	107
108	39.49	8:3	198	.8845	23456-235	0.068	0.045	108
109	39.70	8:3	199	.8875	2345-2356	2.642	1.737	109
110	40.18	8:3	196 203	.8935	2345-2346 ; 23456-245	2.569	1.689	110
111	41.23	7:1	189	.9142	2345-345	0.025	0.018	111
112	42.62	8:3	195	.9321	23456-234	0.302	0.199	112
113	43.10	9:4	208	.9320	23456-2356	0.155	0.094	113
114	43.95	9:4	<i>207</i>	.9423	23456-2346	0.046	0.028	114
115	45.23	8:2	194	.9620	2345-2345	1.047	0.688	115
116	46.03	8:2	205	.9678	23456-345	0.063	0.041	116
117	50.68	9:3	206	1.010	23456-2345	0.399	0.243	117
118	56.15	10:4	<i>209</i>	1.050	23456-23456	0.007	0.004	118

Concentration = 1,280 PPB

Total Nanomoles = 4.53

Average Molecular Weight = 282.6

Number of Calibrated Peaks Found = 102

¹ - Note that 5 DB-1 peaks (PK18, PK40, PK81, PK86, PK97) have been removed from the DB-1 peak numbering scheme. The following low level congeners that were designated as separately eluting peaks have been determined to co-elute with another congener. The DB-1 peak numbers are no longer required for these congeners, but the original DB-1 numbering system has remained intact for all other peaks.

PK 18 (23) now elutes in PK 19 (23,34,54)

PK 40 (68) now elutes in PK 41 (68,96)

PK 81 (176) now elutes in PK 80 (130,176)

PK 86 (166) now elutes in PK 85 (166,178)

PK 97 (157) now elutes in PK 96 (157,202)

² - IUPAC congener numbers listed in **boldface** font were found to be present in at least one of the Aroclors at or above 0.05 weight percent. These congeners should be considered the primary congeners existing in a peak composed of co-eluting congeners. IUPAC congener numbers listed in *italic* font were absent or present below 0.05 weight percent.

³ - PCB congener identification is denoted by position of the chlorine atoms on each ring of the biphenyl molecule. Designation used in this report has unprimed chlorines separated from prime chlorines by a hyphen that represents separation of the biphenyl rings.

Attachment I-2

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ANSP Protocols for Biota Sampling/Analyses

SAMPLING TECHNIQUES

As in past years, fish will be collected from the Cornwall reach in August or September (1992) by the Connecticut Department of Fisheries, with assistance by staff of the Academy of Natural Sciences. Fish collected in this period will include brown trout and smallmouth bass.

Collections of trout in the other three periods (May-June 1992, July 1992, October 1992) will be obtained principally by backpack electroshocking by Academy staff. Fish may also be collected by angling by Academy staff or local volunteer fishermen under special permit from the Connecticut Department of Fisheries.

Bass, perch, sunfish, and eel from Bulls Bridge Station, Lillinonah, and Zoar will be collected principally by boat electroshocking. Other gear (e.g., gill nets, trotlines) will be used if needed. These fish will be collected during a single trip in August or September (1992) and, if necessary, in a second trip in October.

Benthic insects will be collected from the Cornwall reach during May or June. Collecting techniques and species sampled will be consistent with the 1990 survey conducted by the State of Connecticut.

Crayfish will be collected from available habitats at the three sampling sites. If possible, sampling will be performed on the same trip that benthic insects are collected. At the present time, however, it appears that schedule conflicts will require crayfish sampling to be done in July. Specimens will be captured from shallow water areas using a Wildco dip net (#484-D82) with a 3-mm (1/8 in) ace mesh. Animals will be held alive in a container with a small amount of fresh water. The holding water will be changed at frequent intervals, depending upon air temperature, container size, and the number of crayfish collected. Specimens will be separated in the field according to species, maturity, sex, and reproductive form. Every effort will be made to obtain a series of adults of the same species, sex, and (if males) reproductive form. Specimens will then be preserved on ice while still in the field.

Crayfish Sample Preparation

Crayfish will be sorted in the field and preserved on ice. They will be transferred to dry ice within 24 hours of collection. Specimens will be transported to the laboratory and maintained in a freezer. Samples will be prepared by thawing specimens and removing tissue from the tails. Sufficient crayfish will be processed to provide a minimum of 20 g dry weight of tissue per sample.

SAMPLING TECHNIQUES

As in past surveys, fish will be collected from the Cornwall reach in August (or possibly September) 1994 by the Connecticut Department of Fisheries, with assistance by staff of The Academy. Fish collected in this period will include brown trout and smallmouth bass.

Academy staff will collect brown trout in October 1994, primarily by electroshocking. Fish may also be collected by angling, either by Academy staff or by local volunteer fishermen under special permit from the Connecticut Department of Fisheries.

Smallmouth bass from Bulls Bridge, Lillinonah, and Zoar will be collected principally by boat electroshocking. Other gear (e.g., gill nets, trotlines) will be used if needed. These fish will be collected during a single trip in August (or September, depending on trout sampling; see above) 1994 and, if necessary, in a second trip in October.

Benthic insects will be collected from the Cornwall reach during May or June. Collecting techniques and species sampled will be consistent with the 1990 survey conducted by the State of Connecticut and the 1992 survey conducted by The Academy. The collecting techniques will include kick-net samples, kick-screen samples, and direct sampling of individual stones from the river bottom.

Aging of Fish

Ages of the specimens analyzed were estimated using otoliths and tags (brown trout). Examination of otoliths from 3 of the 44 brown trout was needed to verify their ages. The remainder of brown trout could be aged using fin clips (Table 3). All of the remaining specimens were aged using otoliths.

Table 3. Fin clips of the 44 brown trout, analyzed for PCBs, which were collected from the Cornwall station during June to October 1992. See table notes for key to fin clips. Fin abbreviations are ADP (adipose), LM (left maxillary), RM (right maxillary), LV (left ventral), RV (right ventral) and UM (no fin clips).

	CLIP								Total
	ADP-90	LM-91	LV-92	RM-90	RM-92	LM-92	RV-91	UM	
6/16-17/92	0	0	0	0	7	0	0	0	7
7/13-14/92	0	0	0	0	2	0	0	0	2
8/6-7/92	1	1	3	1	12	0	1	0	19
9/22/92	0	0	0	1	6	0	0	0	7
10/20/92	0	0	1	2	5	0	1	0	9
ALL	1	1	4	4	32	0	2	0	44

KEY:
ADP-90=Stocked in Spring 1990 as 2+ Burlington adult, age 4+ when collected
LM-91 =Stocked in 1991 as 1+ Survivor yearling, age 2+ when collected
LV-92 =Stocked on May 14, 1992 as 2+ adult, age 2+ when collected
RM-90 =Stocked in Spring 1990 as 1+ yearling, age 3+ when collected
RM-92 =Stocked on May 28, 1992 as 1+ yearling, age 1+ when collected
LM-92 =Stocked on May 28, 1992 as 1+ yearling, age 1+ when collected
(assume RM should have been clipped, same size as RM-92 fish)
RV-91 =Stocked in Spring 1991 as 2+ Burlington adult, age 3+ when collected
UM =Unmarked, or wild fish

The largest pair of otoliths (sagitta) were dissected from the fish in the lab during the filleting procedure and placed in small vials of 95% ethanol. One of these sagitta was embedded with fast-cure epoxy resin and dried. Thin sections were cut transversely through the otolith with a Buehler Isomet low-speed saw. Three to five of these thin sections per fish were affixed to a microscope slide with emersion oil. Sections were examined under a dissecting microscope at 50x magnification, with either direct viewing or viewing through a video image on a monitor. Specimens which were more difficult to age (e.g., eels, white perch) were examined under a

compound microscope (100-200x magnification) when it was necessary to examine the edge or kernel of the otolith more carefully for annuli.

When viewing the sectioned otoliths, annuli (annual marks) are visible as narrow, dark bands. Annuli are typically well-pronounced, with thin, faint bands representing other cycles of growth. Age was estimated using these pronounced bands, with the innermost band assumed to represent the first winter-spring (transition between age 0+ and 1+).

Ages were determined independently by two fisheries biologists who read the otoliths and then compared results. For 96% of the otoliths, age discrepancies between the two biologists were less than or equal to one year; exact agreement occurred for 65% of the otoliths. As in past ANSP studies, eels were responsible for major discrepancies in aging (3 of the 5 had to be re-examined with the compound microscope). A mutually agreed upon determination was accomplished for these discrepancies after re-examination of the otoliths and discussion.

Brown Trout

Most of the brown trout analyzed from the Cornwall station were identifiable by fin clips applied prior to stocking. These brown trout were stocked at ages 1+ or 2+ in the spring of 1990, 1991 or 1992 (Table 3). The oldest trout was a 4+ that was stocked in 1990 as a 2+ adult (based upon the adipose fin clip). Information regarding fin clips, stocking dates and age at stocking were provided by the State of Connecticut Inland Fisheries Division.

Yearling trout were identified by a right maxillary clip (51 collected of which 32 were analyzed) and were stocked at age 1+ on 28 May 1992. Trout with a left ventral clip (12 collected, of which 4 were analyzed) were stocked on 14 May 1992 as age 2+ adults.

Like the 1992 yearling fish, three larger, apparently holdover brown trout had right maxillary clips. Examination of otoliths of these three trout confirmed that they were from the Spring 1990 stockings which also used a right maxillary clip.

Total age of each specimen was assigned, adding river-age (time between stocking and collection) to age at stocking. Age at stocking was assigned assuming a hatching date of 1 January. For example, a yearling fish stocked in April was assigned an age at stocking of 1.4 years. River-age was the primary variable used for analysis of PCB uptake rates.

- *From ANSP, 1993*

SAMPLE HANDLING AND PREPARATION

Fish

After capture, fish specimens were held in water or on wet ice until the field processing site was reached. Processing was done typically within 1 to 6 h of the time of capture. Fish not needed for chemical analysis were measured (total length in cm) and released or discarded.

At the field processing site, fish needed for chemical analysis were measured for total length in cm. Specimens were wrapped in clean aluminum foil. Specimens of the same species from the same locality were sometimes wrapped together. An aluminum tag was inserted inside each package, but without contacting the specimens. The inner label contained the date, species, number of specimens, locality and serial number. The foil packs were secured with freezer tape, and a wax pencil was used to label the outer tape with the same information that was on the inner tag. The foil packs containing the specimens were placed in coolers with dry ice and frozen.

Upon arrival at ANSP, fish data were logged into a computer database and the specimens placed into the ANSP fisheries section freezer until laboratory processing. All samples were accounted for in this way. Chain-of-custody forms were used to verify transfer of specimens from state collecting crews to ANSP field personnel and from ANSP field personnel to laboratory personnel for processing or storage.

Handling of fish followed the Academy's Standard Operating Procedure P-14-04 (Fish Preservation, Fixation, and Curation, Rev. 2). Quality control procedures were followed during field sampling and laboratory work, all efforts being made to minimize contamination of the specimens. In particular, sample material was prepared using clean equipment and on clean surfaces, avoiding contact between sample material from different specimens or contact with uncleaned laboratory surfaces.

Fish to be analyzed for PCBs were thawed in the laboratory, measured for total length (± 0.1 cm) and weight (± 0.1 g). The field identification of each specimen was verified. For the brown trout from Cornwall, fin clips, if present, were observed carefully and recorded. During sample preparation, any external or internal anomalies such as fin wear, trematodes, nematodes, etc., were noted and recorded in the database. The laboratory methods followed the Academy's Standard Operating Procedure P-14-12 (Preparation of Tissue Samples for Contaminant Analysis, Rev. 1 Draft).

Comparisons of field and laboratory lengths indicate that laboratory lengths are usually similar but slightly less than field lengths, indicating slight shrinkage of some fish, presumably due to freezing.

Each fish was given a four digit analysis number used for tracking through chemical analysis. Specimen data were entered directly into a computer database, with hard copy backup each day.

A clean glass plate and stainless steel fillet knife or scalpel blade were used for each specimen. Sterile scalpel blades were placed onto a scalpel handle and rinsed with dichloromethane prior to use in filleting and mincing. If necessary, debris and mucus were removed from the fish by rinsing in distilled water. The left fillet was taken unless both fillets were necessary to obtain sufficient sample material. Following standard practice based on typical human food-preparation customs, eels were processed with the skin and scales removed, whereas the skin and scales were left on the trout fillets. The fillets of the other species (yellow perch, white perch, smallmouth bass and sunfish species) had the skin on but with the scales removed. Fillets were taken to include the flesh covering the abdominal cavity. The entire fillet was minced and placed into clean glass jars with a clean piece of aluminum foil between the jar and the lid. Fillet weight and sex of the specimen were recorded. Otoliths of most target specimens (except for brown trout of known age; see brown trout section) were dissected and preserved separately in small vials with 95% ethanol. The fillets were delivered to the ANSP Chemistry Section along with a Chain-of-Custody form. The remains were wrapped in aluminum foil, labeled, and refrozen, permitting examination or analysis of additional material, if necessary.

Cleaning of the glass plates and fillet knives at the end of each lab session included the following steps:

1. wash with Alconox detergent and thoroughly rinse in tap water;
2. either: a) rinse in 50% nitric acid, rinse with distilled and double distilled water, and solvent-rinse with acetone and dichloromethane, or b) muffle overnight in a muffle furnace; and
3. cover with muffled aluminum foil to avoid contamination prior to use.

Insects

Aquatic insects were placed in I-Chem Superfund Analyzed glass jars bearing a duct tape label on the outside. The label bore a unique code number with the project name, site identification, date, and name of the taxon. Samples were placed on wet ice, then transferred to the sample cooler in a walk-in freezer at the end of the day.

Samples were packed in the freezer cooler with frozen blocks of "blue ice" and taken by vehicle to ANSP. Specimens were ground in a Tissuemizer and processed as described above for fish samples.

ANALYSES OF PCBS

The laboratory method used for treatment of fish is based on Academy Standard Operating Procedure P-16-83, "Soxhlet Extraction and Cleanup of Tissue for PCB Congener Analysis."

Fish tissues received from the Academy's fisheries section were ground using a Tissuemizer. Each entire ground sample was desiccated by addition of three parts sodium sulphate to fish tissue by mass.

An aliquot of about 70 g of this material was placed into a glass extraction thimble and extracted over approximately 20 h using 1:1 acetone:hexane in a Soxhlet extraction tube. Surrogate standards (PCBs 14, 65, 166) [NOTE: We now use only PCBs 14 and 166 as surrogate standards; because of apparent matrix effects, PCB 65 showed depressed recoveries in all samples analyzed in the 1992 study] were added to the sample before extraction began. Following extraction, the sample volume was exchanged to hexane and reduced to about 1 ml using rotary evaporation, with final volume adjusted to 10 ml. An aliquot of 1 ml was removed from this volume for gravimetric analysis of lipids. The remaining extract was concentrated to about 2 ml, and washed with an equal volume of sulfuric acid. This volume was cooled overnight to separate the hexane and acid phases. The hexane phase was then transferred to another vial. Additional hexane washes were added, then all were combined. The hexane phase volume was reduced to about 2 ml.

The extract was cleaned by passing it through fully activated Florisil in a Sep-Pak cartridge, followed by three volumes of hexane. Internal standards (PCBs 30 and 204) were then added to each sample.

Each sample was analyzed using capillary gas chromatography, employing electron capture detectors and DB-5 columns. The temperature program was slower than in previous years with the intent of separating some PCB congeners from each other or from non-PCB compounds such as hexachlorobenzene, *para, para'*-DDE, and pentachloroanisole. (Both hexachlorobenzene and *para, para'*-DDE were in fact found in the samples.) The oven temperature at injection was 50°C, ramped to 130°C at 5.0°C/min, to 203°C at 0.3°C/min, and finally to 280°C at 10.0°C/min. The compounds of interest were found in the range between 130°C and 203°C.

PCB identification was congener-specific, based on Academy Standard Operating Procedure P-16-84, "Congener-specific Quantitation of Polychlorinated Biphenyls (PCBs) by Capillary Column Gas Chromatography." The method uses Aroclors 1232, 1248 and 1262 mixed in a ratio of 250:180:180. The identification and mass of individual congeners in this mixture was generated by USEPA's Large Lakes Research Station at Grosse Ile, Michigan, and were enumerated by Mullin (1985). Up to 121 PCB congeners are found in this mixture, containing nearly all environmentally significant PCB. The three surrogate standards [NOTE:

now *two* surrogate standards] and two internal standards are included in this mixture in order to make a calibration standard.

Two methods were used for quantitating PCB concentrations based on the congener-specific data. The first was the same method used in previous Housatonic River projects. This method is based on estimating the concentrations of selected congeners which are essentially unique to Aroclor 1254 or 1260, extrapolating to Aroclor concentrations from the relative proportions of these congeners in each Aroclor, and then summing the two Aroclor estimates. The resulting estimate of total PCB concentration is called TPCB and assumes that all PCB congeners found in the original Aroclor mixture are found in the same relative proportions in fish tissue, which may not be the case in environmental samples.

The second method, used only in 1992, is simply to sum all of the 121 individual congeners analyzed to derive a total PCB value. These estimates are called CTPCB. This method does not require assumptions about the relative proportions of congeners in Aroclors, which are central to the first method. The effect of computing PCB concentrations by these two different methods is discussed later in this report in the section entitled "Relationship Between Congener and Aroclor Quantitation Methods".

There were also certain minor differences between the 1990 and 1992 analytical methods. These consist of the following: (1) The 1990 method employs an external standard, whereas the 1992 method uses the internal standard method for quantitation, adding standards (PCB congeners 30 and 204) to the sample before injection into the instrument. (2) The 1990 method uses a non-PCB surrogate (TCMX), whereas the 1992 method measures recovery using three PCB surrogates (PCB congeners 14, 65, and 166) [NOTE: now *two* surrogate standards - PCBs 14 and 166] added to the sample immediately before beginning extraction. (3) The 1992 method uses a slower temperature ramp to increase resolution of the chromatogram.

Of these differences, the only one likely to bias TPCB estimates is the faster temperature ramp used in the 1990 (and previous) studies. A faster ramp decreases chromatogram resolution, possibly preventing closely spaced peaks from being distinguished. The result is a potential bias toward elevated PCB estimates in pre-1992 studies. Such unresolved peaks were probably sufficiently rare that the resulting bias was small. TPCB estimates should therefore be comparable across all studies.

REPORTING LIMIT

Values for all QA/QC analyses are included, as are detection and quantitation limits (LOD and LOQ). The LOD is calculated as the mean value of blanks, plus 3 standard deviations, while the LOQ is the mean plus 10 standard deviations.

QUALITY ASSURANCE PROCEDURES

The Academy's Division of Environmental Research will follow the general provisions established by the Division's Quality Assurance Implementation Plan (November, 1988). A QA/QC Officer will be assigned to ensure that the project complies with this Plan, as well as specific requirements set forth in the project protocol.

QA/QC elements established for the proposed study include field and study audits, data validation, and final report review. The anticipated field audit will consist of a pre-field check to ensure that the appropriate data forms, field logbooks, chain-of-custody forms, and so forth are taken, plus a full day observation of field procedures to ensure that the associated logs are adequately completed, the protocol and Standard Operating Procedures (SOPs) are followed, and all samples are shipped under Chain-of-Custody.

A percentage of data generated throughout the course of this project will be validated by QA to ensure against errors in data recording, data entry, and computing.

The study audit will consist of a laboratory review prior to final study completion to ensure that laboratory and personnel records are complete and adequately maintained, the study protocol and laboratory SOPs were followed, and performance records are complete and adequate.

The final report will be reviewed by QA as a final check to ensure adherence to SOPs and study protocol, and that all required QA elements of the project have been addressed.

QA/QC samples are analyzed with samples at a minimum rate of 5 percent. Each of four different types of QA/QC samples are analyzed with each group of 20 samples. They are blanks, duplicates, standard reference materials, and matrix or sample spikes. Blanks are used to calculate detection and quantitation limits. Duplicates are used to measure precision, which is calculated as relative percent difference (RPD). The RPD target is less than 50 percent. RPD is calculated using

$$RPD = 100 * (\text{dup 1} - \text{dup 2}) / 0.5(\text{dup 1} + \text{dup 2}).$$

Standard reference materials and matrix/sample spikes are used to measure recovery, or accuracy. Our target for reference materials is to be within the 95 percent confidence limit of the certified value. Our target for recovery of spikes is greater than 50 percent and less than 120 percent. In addition, accuracy is measured in each sample by recovery of surrogate standards. Our target recovery for these is also between 50 and 120 percent.

Appendix J

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Soil Gas Sampling Procedures

Appendix J

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 six 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 quadrupole 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 T. The augers will be advanced to the water table depth or one foot above the water table if the depth to water is known. A one inch inside diameter (ID) polyvinyl chloride (PVC) well casing with a one 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 one 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. 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.
- OVA photoionization detector.
- 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" 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-foot 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 OVA photoionization detector to the teflon tubing. Record the OVA reading in the field log book and on the sample chain-of-custody 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 chain-of-custody 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 OVA photoionization meter, 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 M.

IV. Soil Gas Analysis

Soil gas sample analysis will be performed using EPA TO-14 methodology. This method incorporates the use of a quadrupole or ion trap GC/MS with a capillary column to provide optimum detection limits. The GC/MS system requires a one liter gas sample (which can easily be recovered from a 6 liter canister) to provide a 0.5 parts per billion volume (ppbv) detection limit. The six liter canister also provides several additional one 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 ground water investigation.

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Appendix K

Air Monitoring Procedures

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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 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 (QA) objectives, sampling procedures, sample custody, analytical procedures, calibration procedures, data validation and reduction, internal quality control checks, preventative maintenance, routine QA 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 Climatronics Electronic Weather Station (EWS) operated at the GE Pittsfield facility will be used. The EWS has been operating continuously since 1991 at the GE facility in East Street Area 2-South, providing data to support GE activities. The EWS measures and continuously records wind speed, wind direction, precipitation, temperature, relative humidity and integrated solar radiation. The siting of the meteorological station was established with the approval of the Massachusetts Department of Environmental Protection (MDEP). The station was installed and continues to operate in accordance with EPA On-Site Meteorological Program Guidance for Regulatory Modeling Applications and a Site Specific Meteorological Monitoring Quality Assurance Project Plan. The operation of the EWS has been successfully audited by the MDEP.

Barometric pressure is measured and recorded manually on each sampling day. In addition, a portable relativity humidity indicator is used for field verification of humidity conditions.

The collected meteorological data is 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 am to 5:00 pm, 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 MIE dataRAM real-time airborne particulate monitor, Model pDR-1000 or equivalent. The dataRAM uses a passive sampling technique and light scattering photometer to determine particulate concentrations. The dataRAM has a measurement range of 0.001 to 400 mg/m³. Particulate data will be logged by the instrument's datalogger and averaged and recorded for each hour and for each sampling day.

Calibrations and maintenance will be conducted at the frequency and in accordance with the procedures recommended by the manufacturer. All calibrations will be recorded.

The dataRAM has an inherent sensitivity to moisture and readings taken under very high humidity conditions are unreliable. GE may, at times, use the professional engineering judgement of its environmental consultants to determine the reliability and usability 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 to indicate what data is included in the average and reasons for excluding specific data.

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 one location. 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 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 exceeds the notification levels described below will be reported to the GE Project Manager and/or to MDEP and/or EPA (as appropriate) within 24 hours of collection. 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 FSP/QAPP. A written report summarizing the results will be provided to GE within 4 weeks of 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
Signature of Individual Responsible For Monitoring Program

2.4 Action Level

For each day of monitoring, the particulate data from the downwind monitor will initially be compared with the data from the background monitor. If the average 10-hour PM10 concentration at the downwind monitor exceeds the average concentration at the background monitor, the downwind concentrations will then be compared with a notification level of 120 µg/m3 - which represents 80 percent of the current 24-hour National Ambient Air Quality Standard (NAAQS) for PM10 (150 µg/m3). This level has been selected to allow notice to GE before concentrations reach the level of the 24-hour NAAQS. Any exceedances of the notification level or the NAAQS will be immediately reported to the GE Project Manager and the regulating agency (MDEP and/or EPA).

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, upwind or background sites, and a co-located site. The samplers will typically operate for 24-hours from 7 a.m. to 7 a.m. during the monitoring period. The specific number of sampling sites and days will be determined on a project to project basis.

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 (Appendix I). This method employs a high volume sampler and a sampling cartridge consisting of a glass fiber filter with a polyurethane foam (PUF) absorbent to sample ambient air at a rate of 0.20 - 0.28 m³/minute. The filter and PUF cartridge are placed in clean, sealed containers and returned to the laboratory for analysis. The PCBs are recovered by Soxhlet extraction with 10% diethyl ether/hexane. The extracts are reduced in volume using Kuderna-Danish (K-D) concentration techniques and subjected to column chromatographic cleanup. The extracts are analyzed for PCBs using capillary gas chromatography with electron capture detection (GC/ECD), as described in Method TO-4A.

Analytical laboratories are required to follow quality assurance measures and performance criteria as described in Method TO-4A.

4. PCB Data Quality Assurance Objectives

4.1 Quality Assurance

The objective of this quality assurance plan is to ensure that the data collected on ambient levels of PCB are adequate to meet the objectives of the specific monitoring program and the intended uses of the data. The following objectives were used as guidelines to assuring quality in the design and implementation of the monitoring program.

- The sampling and analytical procedures should follow the EPA Compendium Method TO-4A and EPA recommended guidelines, where applicable. The EPA Compendium Methods are not standard EPA reference methods. Pursuant to conversations with the TO-4A method 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. A field book 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, $\pm$ 60 minutes, from approximately 7 a.m. to 7 a.m., at a target rate of 8 standard cubic feet per minute (scfm). The minimum target total sample volume is 300 scm. 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 - Results for total PCB will be reported in $\mu\text{g}/\text{m}^3$ corrected to EPA standard conditions of 25°C and 760 mm Hg.

Completeness - The project completeness requirements are as follows:

- 90% validity of the total proposed samples;
- 90% validity at each sampling site; and
- No sampler may have two or more consecutive invalid samples.

Precision - Sampling precision for the TO-4A Method is undefined. Sampling precision, however, will be measured during each sampling event at one sampling site with a co-located sampler. Precision will be measured by calculating

the percentage difference between the readings at the two sites, the average percentage difference, and the standard deviation and upper and lower 95% probability limits for precisions.

Analytical precision for PCB compounds is determined to be in the range of ± 5 to $\pm 30\%$ measured as the relative standard deviation.

Accuracy - Sampling accuracy is measured by auditing the flow rate of the samplers every six months using a flow transfer standard. The differences between the audit flow measurement and the flow indicated by the sampler magnehelic is 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 65-125% are considered acceptable.

4.3 Detection Limits

The target PCB project detection limit (DL) is 0.0005 µg/m³. The DL was established in consideration of the following:

- Massachusetts Allowable Ambient Levels (AAL); and
- Analytical detection capabilities as limited by sampling duration and sampling rate.

4.4 Allowable PCB Ambient Air Level (AAL)

The Massachusetts AALs for total PCBs used in the Air Toxics Program are specified below.

- PCB Annual Average 0.0005 µg/m³ (0.5 ng/m³)
- PCB 24-Hour Average 0.003 µg/m³ (3.0 ng/m³)

4.5 Analytical Detection Capabilities

The TO-4A specified analytical detection limit is 100 ng/PUF or 0.1 µg/PUF.

4.6 Sampling Flow Rate and Total Volume

Method TO-4A (Appendix I) recommends a sampling rate of 0.20 - 0.28 m³/min with an acceptable flow rate range within + 10% and a recommended minimum total volume of 300 scm. The anticipated operating rate is 0.225 m³/min. At this sampling rate, a total flow volume of 324 m³/air sample will be achieved over a 24-hour sampling period.

4.7 Summary of Project Detection Limits

24 Hour Average Mass. PCB AAL	0.003 µg/m ³
Annual Average Mass. PCB AAL	0.0005 µg/m ³
Target Sampling Rate	0.225 m ³ /min
Target Minimum Sample Volume	300 m ³ /PUF
Lab Detection Limit	0.1 µg/PUF
Allowable Project DL	0.0005 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 standard operating procedures (SOP) 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 with acetone for 14-24 hours in a Soxhlet extractor. The PUFs and filters will be dried in a vacuum oven at room temperature for 2-4 hours. 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 analyzed with gas chromatograph-electron capture detection for PCBs to serve as a blank check.

Glass cartridges will be cleaned and reused after each sampling event. New PUFs will be used for each event. The PUFs will be cleaned in accordance with procedures specified in Methods TO-4A. The solvent used for cleanup will be a 95/5 hexane/ethyl ether. 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 an 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.

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 Sampling 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, the PUF cartridges will be sorted by date of extract. The PUFs with filters will be stored in ice chests. The batches will be used in order of extraction date. As each batch is used the date of use 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 and assigned to a specific sampler. The sampler and PUF cartridge numbers will be recorded on the field data sheet.

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.

6.1.3 Sample Shipment

Samples will be shipped in sealed ice chests on 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 records for the field 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 final sample extract will be concentrated to a 2 mL final volume to meet the required analytical detection limits.

7.2 Test Method

The analytical procedure to determine PCBs will be by electron capture detection as described in TO-4A. Any deviations from TO-4A, if required, will be proposed in the project-specific work plans.

To corroborate the results using GC/ECD, samples may be analyzed with high resolution GC/MS. The results of both methods will be reported.

8. PCB Calibration Procedures

Calibrations 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	EPA 600/4-77-027a, Sec. 2.5; 40 CFR 50, Appendix B; Method TO-4A; (Orifice Type Flow Transfer Standard)
Variations From Reference	None
Accuracy	± 2 percent actual flow rate
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 percent.
Reference Procedure	40 CFR 50, Appendix B (Conventional Orifice Type Flow Standard)
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 will be recorded on a worksheet and maintained in the calibration log.

9. PCB Data Validation and Reduction

9.1 Data Reduction

The PCB sampling data and analytical results will be combined to report an ambient concentration of PCBs in $\mu\text{g}/\text{m}^3$ 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 $P_1 \dots P_n$ to $Q_1 \dots Q_n$

where: $P_1 \dots P_n$ = magnehelic readings from sampling event recorded on the field data sheet

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

2. Determine the average flow rate:

$$Q_{\text{STD}} = \frac{Q_1 + Q_2 + \dots + Q_n}{N}$$

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

N = number of flow readings

3. Calculate the total elapsed time:

$$\text{ETM}_{\text{Finish}} - \text{ETM}_{\text{Start}} = \text{ET}$$

where: $\text{ETM}_{\text{Finish}}$ = elapsed time meter reading at the end of sampling

$\text{ETM}_{\text{Start}}$ = elapsed time meter reading at the start of sampling

ET = total elapsed time (hours)

4. Calculate the standard volume of air sampled:

$$V_m = Q_{\text{STD}} \times 1000 \text{ l}/\text{m}^3 \times 60 \text{ min}/\text{hr} \times \text{ET}$$

where: V_m = total standard volume sampled (m^3)

Q_{STD} = flow rate (m^3/min)

ET = elapsed time (hours)

Analytical Data

The laboratory will reduce the analytical results for each sample to total PCBs measured in $\mu\text{g}/\text{PUF}$. The procedures for this determination and calculation are defined in the Compendium Method (Appendix I).

Sample Concentrations Conversions

The analytical data provided by the laboratory will be reduced for comparison with standards.

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

where: CA = concentration of PCBs in sample ($\mu\text{g}/\text{m}^3$)

V_m = total standard volume of air

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	$\pm 10\%$ Deviation from Calculated Value	Note sample as suspect; Recalibrate
Magnehelic Zero Check	Before and After Each Sampling Event	$\pm 2"$ H ₂ O	Adjust
Leak Check	Before and After Each Sampling Event; at Each Calibration	No Leaks	Repair Leak
Magnehelic Readings	Every 6 Hours During Sampling	$\pm 10"$ H ₂ O	Note Reading; Adjust Instrument

10.2 Field Sampling Precision Check

As a precision check on field sampling for PCBs, two samplers will be co-located at one sampling site. The samplers are located 2-4 meters apart. One sampler will be identified as the normal routine 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 15%.

10.3 Laboratory, Travel and Solvent Blanks

10.3.1 Laboratory Blank

One PUF cartridge and filter from each batch of approximately 20 extracted PUFs and prepared filters will be analyzed, without shipment to the field, for PCBs. In order for the PUF batch to be considered acceptable for use, the blank level must be below the limit of detection.

10.3.2 Travel or Trip Blank

For each sampling event, one PUF cartridge and filter will be carried to the field and returned in a clean sample holder. No air is drawn through the cartridge. The PUF and filter will remain in an ice chest during sampling and will be packaged and shipped, along with the remaining samples, to the laboratory for analysis.

10.3.3 Solvent Blank

During the analysis of each batch (approximately 20) of samples, one process blank will be analyzed to assure that the extraction solvent is free from PCB contamination.

10.3.4 Analytical Collection Efficiency and Spike Recovery

The procedures for analytical efficiency and spike recovery are identified in the TO-4 Method. Collection and recovery efficiencies of 65-125 percent are considered adequate for this method.

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, timer wheel, magnehelic gauge and ETM will be all 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 before 500 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

An inventory of spare parts consisting of the following will be maintained for the high volume samplers:

- 1 Dual Sampling Module
- 2 Filter Gaskets
 - 1 Silicone Gasket for Upper Module
 - 1 Silicone Gasket for Lower Module
- 2 Glass Cartridges with Support Screens and PUFs
- 1 Replacement Motor
- 12 Replacement Motor Brushes
- 1 Calibration Orifice

12. Specific Routine Procedures to Assess PCB Data Precision, Accuracy and Completeness

12.1 Accuracy

The results from the routine calibrations and one-point audits will be used to calculate the accuracy of the sampler flow rate. The percentage differences between the audit and measured points will be calculated. Variations greater than $\pm 15\%$ warrant recalibration.

12.2 Precision

The calculation of sampler precision is based on the operation of the co-located sampler. The percentage difference between the two readings is recorded. The average percentage difference will be calculated for each project.

12.3 Completeness

Data completeness will be calculated for each project. Completeness is calculated as a percentage of valid data compared to the amount of data expected.

13. Corrective Action for PCB Sampling

13.1 Responsibility

Corrective action may be initiated as a result of disruptions in PCB sampling or problems associated with the quality control checks, calibrations and performance, system and QA 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.

13.2 Internal Quality Control Checks

Corrective actions for internal quality control checks are described in Section 13.1 of this plan.

13.3 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.

13.4 Performance and System Audits

Any data 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.

13.5 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 this 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.

13.6 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.

14. 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 will 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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Appendix L

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

Appendix L

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 "horizon" 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 "dating" 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 G;
- Section cores into samples as specified in Appendix G;
- 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 kiloelectron 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 and 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 "dead-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:

$$A = \frac{(N/dt-B)}{2.22 (E)(V)(D)(I)}$$

$$E = \frac{2 [(N/dt+B)/dt]^{1/2}}{2.22 (E)(V)(D)(I)}$$

$$L.T. = \frac{4.66 (B/dt)^{1/2}}{2.22(E)(V)(D)(I)}$$

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 "not 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 M

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Handling, Packing, and Shipping Procedures

Appendix M

Handling, Packing and Shipping Procedures

I. 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 comments.

Step 2 - Fill in sample label (Attachment M-1) 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 M-2) 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.

Step 7 - Initiate chain-of-custody (Attachment M-3) by designated sampling personnel responsible for sample custody (after sampling or prior to sample packing). Note: If the designated sampling person relinquishes the samples to other sampling or field personnel for packing or other purposes, the samplers will complete the chain-of-custody prior to this transfer. The appropriate personnel will sign and date the chain-of-custody form to document the sample custody transfer.

II. 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 and package 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 temperature of approximately 4°C.
- Step 6 - Fill the remaining space in the cooler with cushioning material.
- Step 7 - Place the completed chain-of-custody forms (Attachment M-3) in a large resealable-type bag and tape the forms 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 M-4) on the top and on one side, and arrows indicating "This Side Up" (Attachment M-4) on two adjacent sides.
- Step 11 - Place custody seal evidence tape (Attachment M-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.

III. 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 chain-of-custody 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 lab 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 chain-of-custody forms.
- When the samples are received by the laboratory, the lab personnel shall complete the chain-of custody by recording 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 chain-of-custody forms.

Attachment M-1

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Sample Label

		PROJECT #	
SAMPLE I.D.			DATE
SAMPLE TYPE ☐ Soil/Sediment ☐ Water		COLLECTION MODE ☐ Composite ☐ Grab	
TIME			
ANALYSIS			
SAMPLER(S)		PRESERVATIVE	

Attachment M-2

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Custody Seal Label

CUSTODY SEAL

BBL

BLASLAND, BOUCK & LEE, INC.
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6723 Towpath Road, Box 66, Syracuse, N.Y. 13214-0066
TEL (315) 446-9120

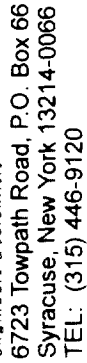
SEALED BY

DATE _____ TIME _____

Attachment M-3

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Chain-of-Custody Form

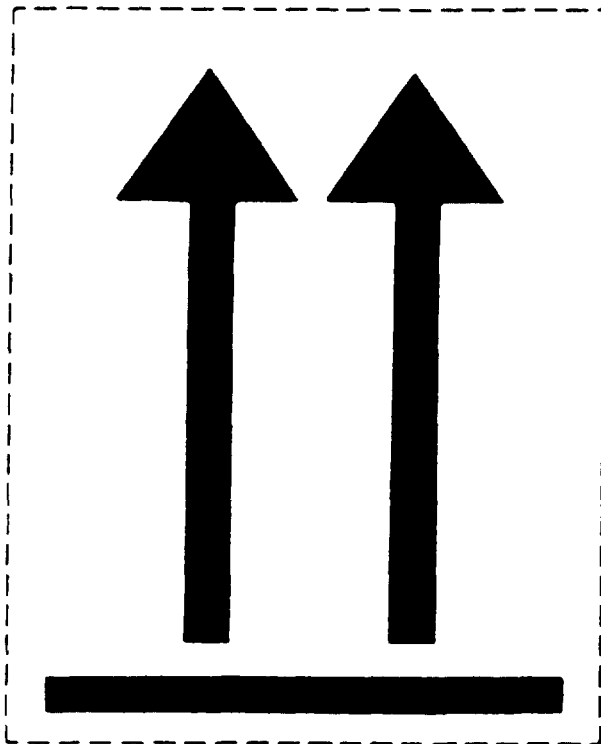
[illegible]

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

Attachment M-4

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Shippers Certification for Restricted Articles



Appendix N

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Standard Operating Procedures for Shipment of Department of Transportation Hazardous Materials

Appendix N

Standard Operating Procedures for 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 eleven 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 SOP

Shipment procedures consist of 11 steps. A summary of the eleven 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 modal-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) provide 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 of dangerous goods/Hazardous Shipping Papers and/or carrier-specific air bill (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 O

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Photoionization Detector Field Screening Procedures

Appendix O

Photoionization Detector Field Screening Procedures

I. Introduction

Field screening with a photoionizer 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 O-1.

II. Materials

- MiniRAE 2000 or equivalent
- Photionization detector (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 Photionization Detector (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 (approx. 500 ml) soil or "mason" type jars are preferred; jars less than eight ounces (approx. 250 ml) total capacity may not be used;
- Step 2 - Allow headspace development for at least ten minutes. Vigorously shake jars for 15 seconds both at the beginning and end of the headspace development period. 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 two and five 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 percent. 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 ($\pm$) lamp source. Operation, maintenance, and calibration shall be performed in accordance with the manufacturer's specifications presented in Attachment O-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; and
- 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 O-1

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MiniRAE 2000 Operation and Maintenance Manual

MiniRAE 2000

Portable VOC Monitor
PGM-7600

OPERATION AND MAINTENANCE MANUAL

Document No. 114001-1
Rev. A



RAE SYSTEMS INC.
680 West Maude Avenue, #1
Sunnyvale, CA 94086

December 1998



2. OPERATION OF MINIRAE 2000

The MiniRAE 2000 Portable VOC Monitor is a compact Monitor designed as a broadband VOC gas monitor and datalogger for work in a hazardous environment. It gives real time measurements and activates alarm signals whenever the exposure exceeds preset limits. Prior to factory shipment the MiniRAE 2000 is preset with default alarm limits and the sensor is pre-calibrated with standard calibration gas. After the monitor is fully charged, it is ready for immediate operation.



Figure 2-1 MiniRAE 2000

2.1 Physical Description

The main components of the MiniRAE 2000 Portable VOC monitor include:

- Three keys for user to interact with the monitor: 1 operation key and 2 programming keys for normal operation or programming of the monitor
- LCD display with back light for direct readout and calculated measurements
- Buzzer and red LED's for alarm signaling whenever the exposures exceed preset limits
- Wrist strap
- Charge contact for plugging directly to the charging station
- Gas entry and exit ports
- Serial communication port for PC interface
- External alarm and analog output port
- Protective rubber cover

2.2 Keys and Display

Figure 2.2 shows the LCD display and the keypad on the front panel of the monitor. The function of the 3 keys during normal operation are summarized below:

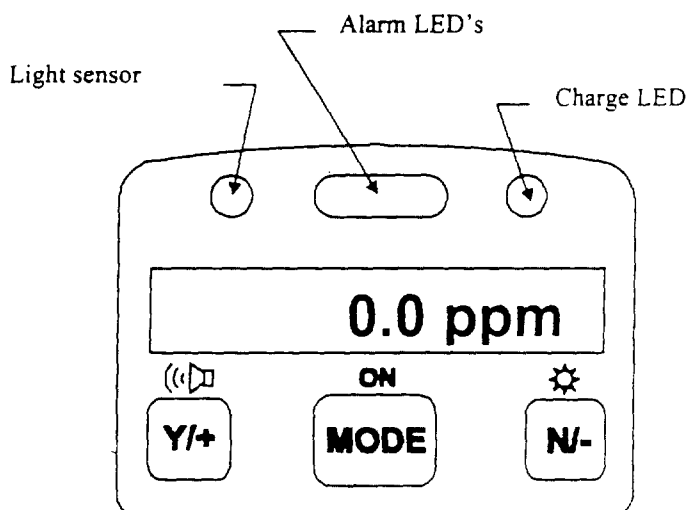


Figure 2.2 LCD display and Keypad

Key Function in Normal Operation	
[MODE]	-Turn on/off the power* and step through menu items
[N/-]	-Toggle on/off the back light, negative acknowledge/decrement value
[Y/+]	-Start measurement, positive acknowledge/increment value

* Pressing and holding **[MODE]** key for 5 seconds turns off the power to the monitor. Monitor will beep once per

OPERATION OF MINIRAE 2000

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

OPERATION OF MINIRAE 2000

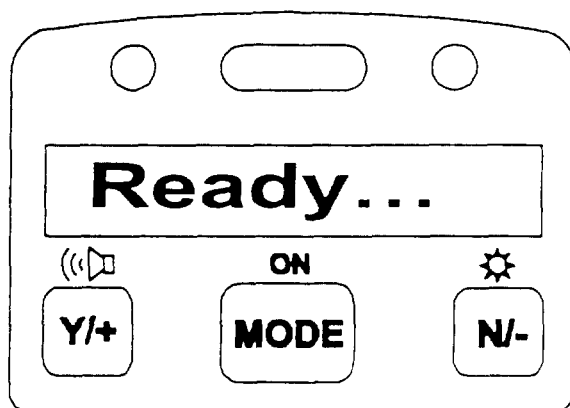
is completely drained or is disconnected from the monitor for more than 30 minutes, the real time clock will be lost. In this case, the user needs to enter the real time clock information again, as described in Section 4, or send the PC clock during configuration through the PC communication.

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.



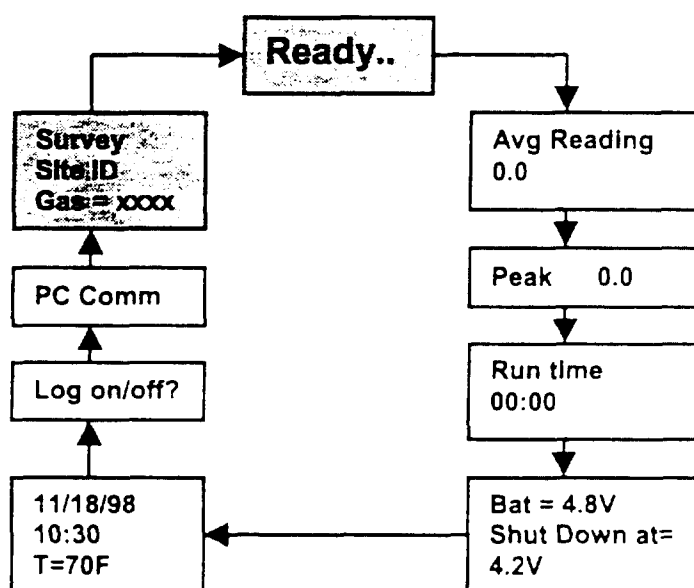
Press the [**MODE**] key to cycle through the idle operation menu. The PID sensor and pump are turned off during this idle operation.

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)

OPERATION OF MINIRAE 2000

- **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

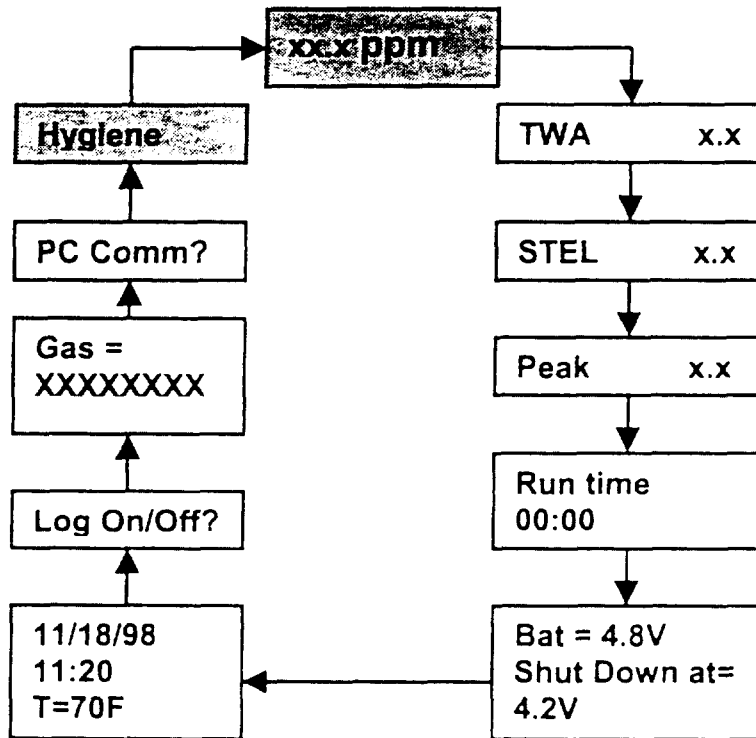
In Hygiene Mode, the unit will continuously taking measurements, once the power is turned on. After the initial start-up sequence displaying the current monitor settings, the LCD displays the instantaneous readings.

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.

OPERATION OF MINIRAE 2000



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.

PROGRAMMING OF MINIRAE 2000

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.

PROGRAMMING OF MINIRAE 2000

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 P

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Temperature, Turbidity, Conductivity, pH, and Dissolved Oxygen Field Measurement Procedures

Appendix P

Temperature, Turbidity, Conductivity, pH, and Dissolved Oxygen Field Measurement Procedures

I. Introduction

Hydrochemical parameters such as specific conductance, pH, temperature, turbidity, 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, conductivity, and dissolved oxygen 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. Calibration will exceed twice per day as conditions and/or manufacturer's specifications dictate.

II. Materials

The following materials shall be available, as required, during measurement of hydrochemical parameters:

- Health and safety equipment (as required by the Health and Safety Plan);
- Two Horiba U-10 or equivalent water quality meters (one for back up);
- pH and conductivity calibration standards;
- Thermometer;
- 500 ml glass container;
- Cleaning equipment (as required in Appendix X);
- Fine screw driver;
- Extra batteries;
- Field notebook; and
- Appropriate log forms.

III. Procedures

The detailed procedure for obtaining the temperature, turbidity, conductivity, pH, and DO of a 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/conductivity/DO meters will be field calibrated in accordance with manufacturer's instructions during each day of use as specified in Attachment P-1;
- Step 2 - Obtain a small quantity of the water sample, place it in a clean glass container, agitate, then discard. Refill the container. Rinse the pH, conductivity, DO, turbidity, and temperature probes with distilled water. Submerge probes into the container containing the water. Allow approximately one minute for readings to stabilize; then record the measurements on the appropriate forms; and

Step 3 - Clean the probe and cable with a non-phosphate soap and water wash followed by a distilled/deionized water rinse. Store in a clean container.

Attachment P-1

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Horiba Instruction Manual

HORIBA

**INSTRUCTION
MANUAL**

**WATER
QUALITY
CHECKER
U-10**

CODE: 040801000HK-5

Section 2

Making Measurements

Making a measurement with the U-10 Water Checker is extremely simple. Just turn on the power and place the probe in the sample of water you wish to measure.

All six parameters are measured simultaneously. These parameters may be stored in memory, printed out, or viewed one-by-one on the LCD readout. For printing and data storage, see the appropriate sections following this one. To view the parameters one-by-one on the readout, use the SELECT Key to toggle the upper cursor through them.

While the U-10 is both rugged and precise, the key to accurate measurements is cleanliness and frequent calibration. It is essential to clean the U-10 thoroughly after each measurement, and it is recommended that you re-calibrate your U-10 as frequently as possible. For best results, you should recalibrate it before each measurement session. Cleaning and calibration procedures are described below in this section and in the following one.

How to make a measurement	12
Initial readout	13
Select the parameter you want shown on the readout	14
Expanded readout	15
Measuring fresh water	16
Measuring salt water	17
After measurement: Cleaning and storing the U-10	18

How to make a measurement



1 Turn the power on.

2

Gently place the probe into the water sample.

Basically, that's all there is to it: just turn it on and put the probe in the sample. Of course, the U-10 can do many sophisticated things with the sample data, and for best results, you should be careful about calibrating the unit and maintaining it in good condition. This is explained in detail below and in the next section.

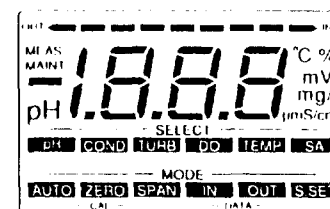
Be careful!

Never drop or throw the probe into the water. It is a precision instrument containing five delicate sensors and five pre-amps; you can damage it beyond repair by unnecessary rough handling.

Initial readout



When you first turn the power on, the U-10 will be in the MEAS mode, the readout will look like this, with all the LCD segments activated.



After about two seconds, the readout will change to show that a new measurement is being made. The readout will show the last parameter that the upper cursor was on when the previous measurement was made, i.e., pH as illustrated here.



(Expanded readout shown)

The display of the decimal point in the readout mode will also be in the same format as was selected with the EXP Key in the previous measurement, i.e., standard or expanded (as illustrated here).

Select the parameter you want shown on the readout of the measured data



All six parameters are automatically measured at once. Use the SELECT Key to toggle the upper cursor to the parameter you want.

pH : pH
COND : Conductivity
TURB : Turbidity
DO : Dissolved oxygen
TEMP : Temperature
SAL : Salinity

To get a uniform reading, slowly move the probe up and down to circulate the water through it. (Move it 1 foot (30 cm) per sec.) Then wait for the readout to stabilize while doing this.

Expanded readout



Use the EXP readout mode when you wish to see the results with one additional decimal place of accuracy. The EXP Key toggles the readout back and forth between standard to expanded display. The table below shows the result of using the EXP readout mode for each of the six parameters.

Table 1 Accuracy of expanded readout

Parameter	Range of measurement	Accuracy	
		Standard readout	Expanded readout
pH	0-14 pH	0.1 pH	0.01 pH
COND	0-1 mS/cm	0.01 mS/cm	0.001 mS/cm
	1-10 mS/cm	0.1 mS/cm	0.01 mS/cm
	10-100 mS/cm	1 mS/cm	0.1 mS/cm
TURB	0-800 NTU	10 NTU	1 NTU
DO	0-19.9 mg/l	0.1 mg/l	0.01 mg/l
TEMP	0-50 °C	1 °C	0.1 °C
SAL	0-4‰	0.1‰	0.01‰

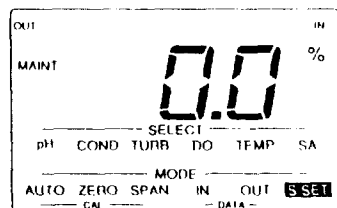
Note that the salinity parameter is the only value not measured directly with its own sensor. The U-10 obtains salinity by converting the conductivity value. If large amounts of conductive ions other than salt water components are present in the sample, an error may occur. Be cautious when interpreting the salinity results.

Measuring fresh water or salt water?

The U-10 can be set to the salinity for either fresh water or salt water when measuring DO. This is done by using the S.SET Sub-Mode.

Measuring fresh water

1. First, use the MODE Key to put the U-10 in the MAINT mode. Keep pressing the MODE Key to toggle the lower cursor to the S.SET Sub-Mode.
2. Once you are in the S.SET Sub-Mode, use the UP/DOWN Keys to select the salinity value. For fresh water, set the salinity to 0.0%.



3. Finally, press the ENT Key to complete the salinity setting while in the S.SET Sub-Mode.
4. When the salinity setting has been made, switch back to the MEAS mode by pressing the the MODE Key.

Measuring salt water

1. First, use the MODE Key to put the U-10 in the MAINT mode. Keep pressing the MODE Key to toggle the lower cursor to the S.SET Sub-Mode.
2. For salt water, set it to *R* i.e., for auto-salinity. The *R* setting should be sufficient for measurements of normal sea water with a salinity value close to 3.3%. For sea water of an unusual salinity, however, and where the value is otherwise known, you may wish set the value manually to any salinity within the range of 0.0%-4.0%. (You may also possibly want to use a manual setting if, for example, the COND sensor is malfunctioning but it is still desirable to take readings of the other parameters.)
3. Finally, press the ENT Key to complete the salinity setting while in the S.SET Sub-Mode.
4. When the salinity setting has been made, switch back to the MEAS mode by pressing the the MODE Key.

After measurement: Cleaning and storing the U-10



1. Turn OFF the power.
2. Wash the probe thoroughly with tap water. Be sure to flush off all of sample solution from the probe.

Storing the U-10 for brief periods, i.e., about 1 week or less:

Fill the calibration beaker with tap water and fit the probe over it.

For longer storage

The pH sensor must always be kept moist. Fill the small rubber cap with water and use it to cover the pH sensor.

The KCl internal solution in the reference sensor may seep out over time. Place vinyl tape around the O-ring portion to prevent this.

If you are going to store the U-10 for a prolonged period without using it, remove the battery from the main unit.

3

Section Calibrating the U-10

The U-10 Water Checker may be calibrated either manually or automatically. The 4-parameter auto-calibration procedure is quite handy and should be sufficient for most measurement operations.

Manual calibration for each of the four parameters is more accurate but, of course, also more time-consuming. This method should be used for more precise measurement. The manual calibration procedure is explained below in detail, following the description of the auto-calibration procedure.

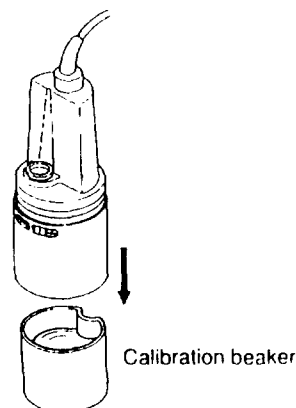
The auto-calibration procedure is extremely simple. The U-10 Water Checker uses just a single solution to do a simultaneous calibration of four parameters: pH, COND, TURB, and DO. Your U-10 comes with a bottle of standard phthalate pH solution and a calibration beaker for this purpose.

Auto-calibration procedure	20
Manual (2-point) calibration procedures	23
pH Calibration	24
1. Zero calibration	24
2. Span calibration	25
COND Calibration	26
1. Zero calibration	28
2. Span calibration	29
TURB Calibration	30
1. Zero calibration	31
2. Span calibration	31
DO Calibration	32
1. Zero calibration	33
2. Span calibration	33

Auto-calibration procedure

Fill the calibration beaker to about 2/3 with the standard solution. Note the line on the beaker.

Fit the probe over the beaker, as illustrated. Note that the beaker is specially shaped to prevent the DO sensor from being immersed in the standard solution. This is because the DO auto-calibration is done using atmospheric air.

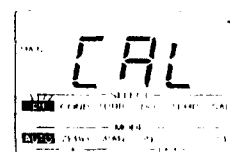


With the power on, press the MODE Key to put the unit into the MAINT mode. The lower cursor should be on the AUTO Sub-Mode; if it is not, use the MODE Key to move the lower cursor to AUTO.

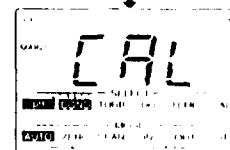


With the lower cursor on AUTO, press the ENT Key. The readout will show *CAL*. Wait a moment, and the upper cursor will gradually move across the four auto-calibration parameters one-by-one: *pH*, *COND*, *TURB*, and *DO*. When the calibration is complete, the readout will briefly show *End* and then will switch to the MEAS mode.

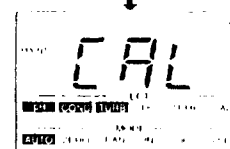
The upper cursor will blink while the auto-calibration is being made. When the auto-calibration has stabilized, the upper cursor will stop blinking.



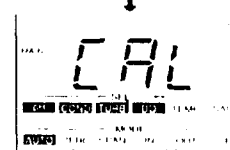
First, pH is being auto-calibrated



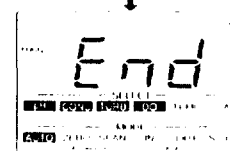
Then, COND is being auto-calibrated



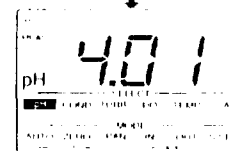
Next, TURB is being auto-calibrated



Finally, DO is being auto-calibrated



Auto-calibration now ends

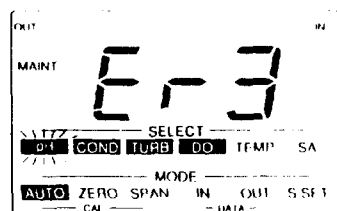


And the readout switches to the MEAS mode

Note: If you wish to abort the auto-calibration for any reason, press the CLR Key. The parameters auto-calibrated so far will be stored in memory.

Auto-calibration error

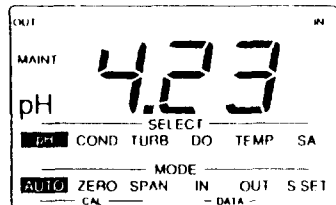
After the DO auto-calibration, if the unit does not switch to the MEAS mode as it should, and the readout shows either *E-3* or *E-4*, an auto-calibration error has occurred. Parameters will blink where an error occurred.



pH auto-calibration error

CLR

If this happens, re-do the auto-calibration. First, press the CLR Key to cancel the error code.



ENT

Then press the ENT Key to re-start the auto-calibration. Restart the auto-calibration beginning again with pH.

Manual (2-point) calibration procedures

For normal measurements, the 4-parameter auto-calibration described above is sufficiently accurate. However, you may wish to do a parameter-by-parameter, 2-point manual calibration of one or more of the four parameters. This is recommended either for high-accuracy measurements, especially when using the expanded readout mode. It is necessary if a new probe is being used for the *first time*.

Parameters to be calibrated manually.

pH	• Zero (see page 24.)
	• Span (see page 25.)
COND	• Zero (see page 28.)
	• Span (see page 29.)
TURB	• Zero (see page 31.)
	• Span (see page 31.)
DO	• Zero (see page 32.)
	• Span (see page 33.)

Parameters not to be calibrated

Sample temperature
Salinity

pH calibration

pH calibration on the U-10 is done using two commercially-available standard solutions of different pH values, one for the zero calibration, the other for the span calibration. Note that the temperature characteristics of the various standard solutions that are available may differ; therefore, before using these two solutions to make the pH calibration, carefully measure the temperature and determine the temperature characteristics of each.

Preparation

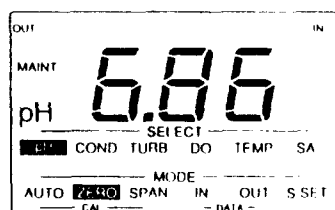
Wash the probe 2-3 times, using de-ionized or distilled water. Place it in a beaker of each standard solution.

1. Zero calibration

Use a pH7 standard solution for the zero calibration.

Operation

1. With the power on, press the MODE Key to put the unit into the MAINT mode.
2. Press the MODE Key again to move the lower cursor to ZERO.
3. Use the SELECT Key to move the upper cursor to pH.
4. When the readout has stabilized, use the UP/DOWN Keys to select the value of the pH 7 standard solution at the temperature of the sample. Refer to Table 2 for pH values of standard solutions at various temperatures.



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5. Press the ENT Key to complete the zero calibration for pH.

Section 3

2. Span calibration

Use either a pH4 or a pH9(10) standard solution for the span calibration.

Operation

1. Use the MODE Key to move the lower cursor to SPAN.
2. As in Step 4. above in zero calibration, when the readout has stabilized, use the UP/DOWN Keys to select the value of the standard solution (i.e., either pH4 or pH9) at the temperature of the sample. Again, refer to Table 2 for pH values of standard solutions at various temperatures.
3. Press the ENT Key to complete the span calibration for pH.

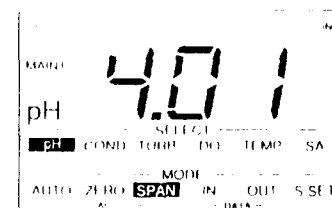


Table 2 pH values of standard solutions at various temperatures*

Temperature C / F	pH2 ^a	pH4 ^b	pH7 ^c	pH9 ^d	pH10 ^e	pH12 ^f
0 / 32	1.67	4.01	6.98	9.46	10.32	13.43
5 / 41	1.67	4.01	6.95	9.39	10.25	13.21
10 / 50	1.67	4.00	6.92	9.33	10.18	13.00
15 / 59	1.67	4.00	6.90	9.27	10.12	12.81
20 / 68	1.68	4.00	6.88	9.22	10.06	12.63
25 / 77	1.68	4.01	6.86	9.18	10.01	12.45
30 / 86	1.69	4.01	6.85	9.14	9.97	12.30
35 / 95	1.69	4.02	6.84	9.10	9.93	12.14
40 / 104	1.70	4.03	6.84	9.07	9.89	11.99
45 / 113	1.70	4.04	6.83	9.04	9.86	11.84
	1.71	4.06	6.83	9.01	9.83	11.70

a. oxalate, b. phthalate, c. neutral phosphate, d. borax
e. carbonate, f. Sat calcium hydroxide solution

* These pH values are for Japanese standard solutions. Should you prefer to use different standard solutions, be sure to make the proper adjustments in calibration.

Section 3

COND calibration

The U-10 can measure conductivity in the range of 0-100 mS/cm. Depending on the sample concentration, however, the U-10 automatically selects the proper range out of its three possible ranges of 0-1 mS/cm, 1-10 mS/cm, and 10-100 mS/cm.

Therefore, if you are doing a manual calibration for COND, this must be done for each of the three ranges. However, since the zero point is common for all three ranges, only the three one-point span calibrations need be done separately.

Preparing the standard solution for COND span calibration

This solution uses a potassium chloride as a reagent. For greater accuracy, the solution should be freshly prepared each time. If it is unavoidable to use a stored solution, be sure to keep it tightly capped in a polyethylene or hard glass bottle. The shelf life of this solution is six months. Date-stamp the bottle for reference. Never use a KCl standard solution that has been stored for more than six months; the calibration accuracy may be adversely affected.

Use potassium chloride powder of the best quality commercially available. Dry the powder for two hours at 105°C, and cool it down, in a desiccator. Weigh out an appropriate amount of dried and cooled potassium chloride powder according to the table below. Make the potassium chloride standard solution as shown.

Table 3 Making the potassium chloride standard solution

KCl standard solution	KCl weight g	Conductivity* mS/cm	Range to be calibrated mS/cm
0.005N	0.373	0.718	0-1
0.05N	3.73	6.67	1-10
0.5N	37.28	58.7	10-100

* Value at the temperature, 25°C

To prepare the standard solution, use a 1-liter volumetric flask. First, dissolve the KCl in a small amount of de-ionized or distilled water. Then fill the flask with de-ionized or distilled water up to the 1-liter line. Finally, shake the solution to mix it thoroughly.

1. Zero calibration

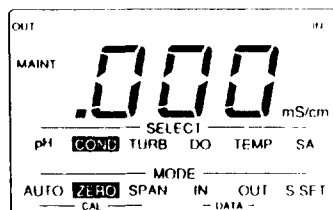
This calibration is carried out in atmospheric air; no solution is needed.

Preparation

Wash the probe 2-3 times, using de-ionized or distilled water. Shake the probe to remove any water droplets from the COND sensor. Then allow it to dry by exposing it to fresh air.

Operation

1. Use the MODE Key to move the lower cursor to ZERO.
2. Use the SELECT Key to move the upper cursor to COND.
3. Use the UP/DOWN Keys to set the readout to zero.



4. Press the ENT Key. This completes the zero calibration for COND.

2. Span calibration

This procedure uses a standard solution of potassium chloride. For best results, a fresh batch of the solution should be prepared each time. See page 27 for details.

Preparation

Wash the probe 2-3 times using de-ionized or distilled water. Following this, wash it 2-3 times in the KCl standard solution you have prepared. Then place the probe in a beaker of the KCl solution maintained at a temperature of $25 \pm 5^\circ\text{C}$.

Operation

1. Use the MODE Key to move the lower cursor to SPAN.
2. After the readout stabilizes, as you did for the pH calibration, use the UP/DOWN Keys to select set the value of the KCl standard solution, referring to the KCl table.
3. Press the ENT Key to complete the span calibration for this COND range.
4. Repeat this procedure for the three ranges, using each of three values of KCl standard solutions.

TURB calibration

Use good-quality de-ionized water, which may be considered as having a turbidity of zero. If that is not readily available, distilled water may be used instead. When doing the turbidity zero calibration, it is particularly crucial that you clean the probe thoroughly. Never use a dirty probe; otherwise the calibration will be unreliable.

Preparing the standard solution for TURB span calibration

1. Weigh out 5.0 g of hydrazine sulfate.
2. Dissolve this in 400 ml of de-ionized or distilled water.
3. Then weigh out 50 g of hexamethylenetetramine, and dissolve it in 400 ml of de-ionized or distilled water.
4. Mix these two solutions, add enough de-ionized or distilled water to make 1,000 ml, and stir the mixed solution thoroughly.
5. Allow this solution to stand for 24 hours at a temperature of $25 \pm 3^\circ\text{C}$.

The turbidity of this solution is equivalent to 4000 NTUs. The shelf-life of this solution is six months; i.e., this 4,000-NTU value will remain accurate for a maximum of six months.

Each time you carry out this calibration, it is necessary to dilute the 4,000-NTU standard solution to prepare an 800-NTU standard solution for calibration. To do this, measure out 50 ml of the 4,000-NTU solution into a 250-ml measuring flask.

It is recommended that you use a rubber pipette aspirator for this. Then add de-ionized or distilled water up to the 250-ml line.

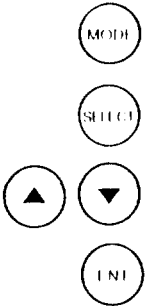
The standard solution used here for the turbidity calibration will precipitate easily. Therefore, be sure to stir the solution thoroughly before use.

1. Zero calibration

Preparation

Wash the probe thoroughly 2-3 times using de-ionized or distilled water. Shake off excess water droplets, and then place it in a beaker of de-ionized or distilled water.

Operation

- 
1. Use the MODE Key to move the lower cursor to ZERO.
 2. Use the SELECT Key to move the upper cursor to TURB.
 3. After the readout has stabilized, set it to 0.0, using the UP/DOWN Keys.
 4. Press the ENT Key to complete the zero calibration for TURB.

2. Span calibration

Preparation

Wash the probe thoroughly, using de-ionized or distilled water. Shake off excess water droplets. Then place it in a beaker of the 800-NTU solution you have prepared for this purpose.

Operation

- 
1. Stir this 800-NTU span standard solution thoroughly.
 2. Use the MODE Key to move the lower cursor to SPAN.
 3. After readout has stabilized, i.e., about 60 to 90 seconds, set the readout to "800" NTU, which is the value for this standard solution.
 4. Press the ENT Key to complete the span calibration for TURB.

DO calibration

Unlike the other calibration procedures, the solution for the DO calibration cannot be stored for use; because the amount of dissolved oxygen in the solution is crucial, a fresh batch must be prepared each time, just before it is used in the DO calibration.

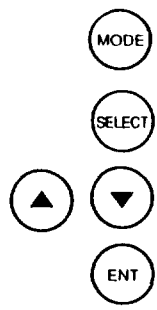
1. Zero calibration

Use a solution of sodium sulfite dissolved in either de-ionized water or tap water.

Preparation

1. Add about 50g of sodium sulfite to 1,000 ml of water (either de-ionized water or tap water will do). Stir this mixture to dissolve.
2. Wash the probe 2-3 times in tap water, and place it in the zero standard solution.

Operation

- 
1. Use the MODE Key to move the lower cursor to ZERO.
 2. Use the SELECT Key to move the upper cursor to DO.
 3. After the readout has stabilized, set it to 0.0, using the UP/DOWN Keys.
 4. Press the ENT Key. This completes the zero calibration for DO.

2. Span calibration

Use either de-ionized water or tap water that has been saturated with oxygen in air.

Preparation

1. Put 1 or 2 liters of water in a container (either de-ionized water or tap water will do). Use an air pump to bubble air through the solution until it is oxygen-saturated.
2. Wash the probe 2-3 times in tap water, and put it in the span calibration solution.

Operation

- 
1. First, be sure the U-10 is set for fresh water readings. To do this, set the S.SET Sub-Mode to 0.0%.
 2. Then, use the MODE Key to move the lower cursor to SPAN.
 3. After the readout has stabilized, while slowly moving the probe up and down in the solution, set the readout value to the appropriate DO value for the temperature of this solution. For DO values at various temperatures, refer to Table 4.
 4. Press the ENT Key to complete the span calibration for DO.

Table 4 Amounts of saturated dissolved oxygen in water at various temperatures, salinity = 0.0‰

Temperature	DO	Temperature	DO
0 °C	14.16 mg/l	21 °C	8.68 mg/l
1	13.77	22	8.53
2	13.40	23	8.39
3	13.04	24	8.25
4	12.70	25	8.11
5	12.37	26	7.99
6	12.06	27	7.87
7	11.75	28	7.75
8	11.47	29	7.64
9	11.19	30	7.53
10	10.92	31	7.42
11	10.67	32	7.32
12	10.43	33	7.22
13	10.20	34	7.13
14	9.97	35	7.04
15	9.76	36	6.94
16	9.56	37	6.86
17	9.37	38	6.76
18	9.18	39	6.68
19	9.01	40	6.59
20	8.84		

Appendix Q

BLASLAND, BOUCK & LEE, INC.
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In-Situ Hydraulic Conductivity Test Procedures

Appendix Q

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 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 X);
- 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 Q-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 photoionization detector 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 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 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 "S" 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 X) 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 X.

* steps 12-14 may be modified if a pressure transducer and automatic data logger are utilized.

Attachment Q-1

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In-Situ Hydraulic Conductivity Test Log

DATE STARTED _____
DATE FINISHED _____
SHEET _____ OF _____
PROJECT _____
JOB NO. _____
LOCATION _____

[illegible]

Appendix R

BLASLAND, BOUCK & LEE, INC.
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Water Level/Oil Thickness Measurement Procedures

Appendix R

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 to measure headspace vapors;
- Health and safety equipment (as required by the Health and Safety Plan);
- Cleaning equipment (as required in Appendix X);
- 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 R-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 X.
- 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 photoionization detector 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 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 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 downhole 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 R-1

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Water Level/Oil Thickness Monitoring Field Log

WATER LEVEL/OIL THICKNESS
MONITORING FIELD LOG
WELL No. ____

Date: _____ Time: _____

Project: _____

Project No.: _____

Weather Conditions: _____

Temperature: _____

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)	
Reference Mark to Bottom of Well (if applicable)	

Appendix S

BLASLAND, BOUCK & LEE, INC.
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Passive Oil Recovery Procedures

Appendix S

Passive Oil Recovery Procedures

I. Introduction

Oil encountered while performing water level/oil thickness measurement procedures will be recovered when quantities exceed amounts determined by GE and approved by USEPA.

II. Materials

The following equipment and materials will be available, as required, during passive oil recovery efforts:

- Photoionization detector;
- Health and safety equipment (as required by the Health and Safety Plan);
- Cleaning equipment (as required in Appendix X);
- Plastic sheeting;
- Field book or appropriate log forms;
- Absorbent pads;
- Peristaltic pump and tubing;
- Bailer;
- Non-absorbent cord (polypropylene);
- Graduated cylinder; and
- Container for recovered oil.

III. Procedures

- Step 1 - Notify GE representatives that the Passive Oil Recovery Program will be initiated so GE can initiate manifesting procedures, if necessary.
- Step 2 - Perform Water Level/Oil Thickness Measurement procedures in accordance with Appendix R.
- Step 3 - If LNAPL is present in well and exceeds action level quantity, it must be recovered for disposal by GE.
- Step 4 - Remove LNAPL utilizing a bailer or peristaltic pump, transfer material into a container supplied by GE and record volume.
- Step 5 - If DNAPL is present in well and exceeds action level quantities, it must be removed for disposal by GE.
- Step 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 original thickness of the DNAPL layer. Record the estimated quantity onto the field log (Attachment S-1).

Step 7 - Do not transport liquid in vehicle along public roadways. Notify GE that LNAPL/DNAPL has been collected and is ready to be picked up for disposal.

Step 8 - Collect all 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.

Attachment S-1

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Passive Oil Recovery Field Log

General Electric
Pittsfield, Massachusetts
Passive Oil Recovery
Field Log

Personnel:

Date:

Time:

Temperature:

Weather Conditions:

GE Well ID	Depth to LNAPL	Depth to Water	LNAPL Thickness	Depth to DNAPL	Depth to Well Bottom	DNAPL Thickness	Est. LNAPL Removed	Est. DNAPL Removed

Notes:

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Appendix T

Monitoring Well Installation and Development Procedures

Appendix T

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's (MDEP's) *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;
- Personal protective equipment (as required by the Health and Safety Plan);
- Drilling equipment required by ASTM D-1586;
- Appropriate sampling equipment (e.g., spatulas);
- Equipment cleaning materials (specified in Appendix X);
- Appropriate sample containers, labels, and Chain-of-custody 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) D1586 - *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 B, C, D, and O 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 six-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 two-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 one-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 one to two 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 two 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 two 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 2.0 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 T-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 T-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 upon the equipment used, the hammer delivers 140 to 350 foot pounds of energy with each blow. The hammer, operated at 1200 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 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 B, C, D, and O 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 outlines in Section IX.

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 upon 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 X. Well materials and well development equipment will also be cleaned in accordance with the procedures outlined in Appendix X.

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 feet. 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 Health and Safety Plan);

- Cleaning equipment (as required in Appendix X);
- Photoionization detector (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:

- Personnel Protective Equipment (as required by the Health and Safety Plan);
- Cleaning equipment (as required in Appendix X);
- Photoionization detector 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 personal protective equipment (as required by the Health and Safety Plan).
- Step 2 - Place plastic sheeting around the well.
- Step 3 - All equipment entering each monitoring well will be cleaned as specified in Appendix X.
- 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 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 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 - When complete, secure the lid back on the well.

Step 9 - Place plastic sheeting and tubing in plastic bags for appropriate disposal and clean pump as specified in Appendix X.

The procedure for developing a well using the bailer method is outlined below:

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

Step 2 - Place plastic sheeting around the well.

Step 3 - Bailers and new rope will be cleaned as specified in Appendix X.

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 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 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 ten 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.

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 X.

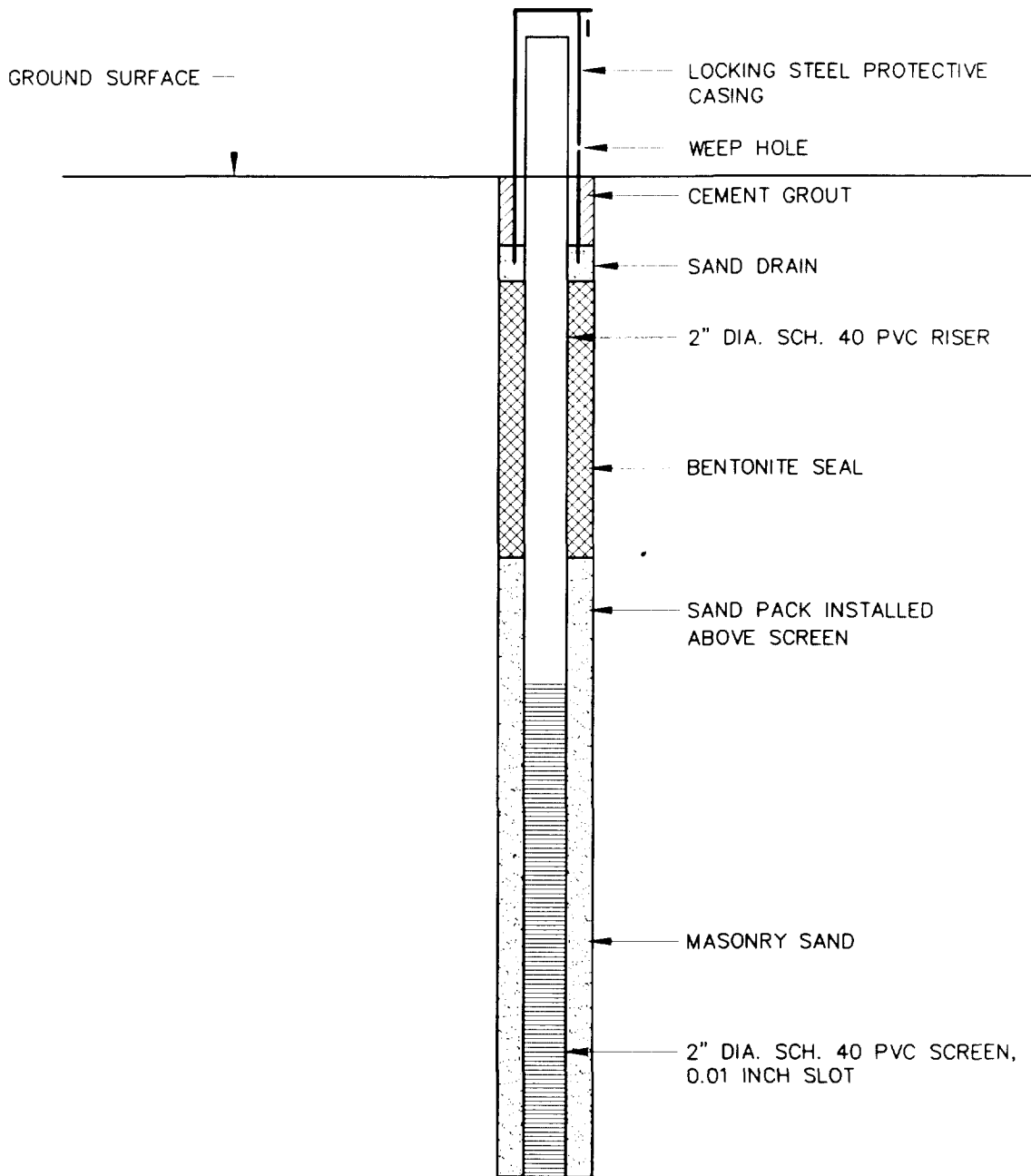
IX. Disposal Methods

Materials generated during monitoring well installation and development will be placed in appropriate containers. Containerized waste will be disposed of by GE consistent with its on-going disposal practices.

Attachment T-1

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Stick-Up Monitoring Well Detail



(DRAWING NOT TO SCALE)

**STICK-UP
MONITORING WELL DETAIL**

BBL

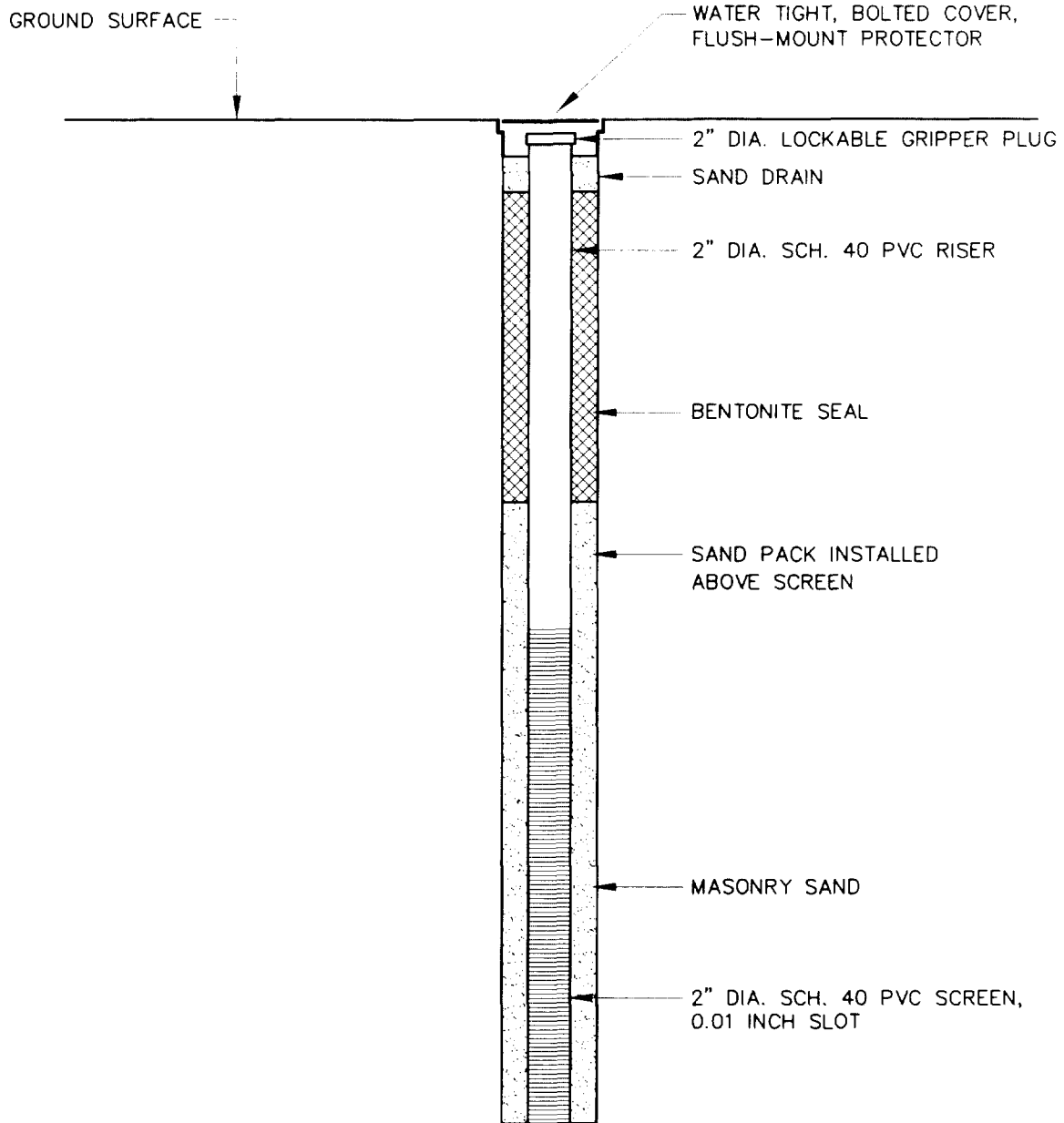
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FIGURE
T-1

Attachment T-2

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Flush-Mount Monitoring Well Detail



(DRAWING NOT TO SCALE)

FLUSH MOUNT MONITORING WELL DETAIL

BBL

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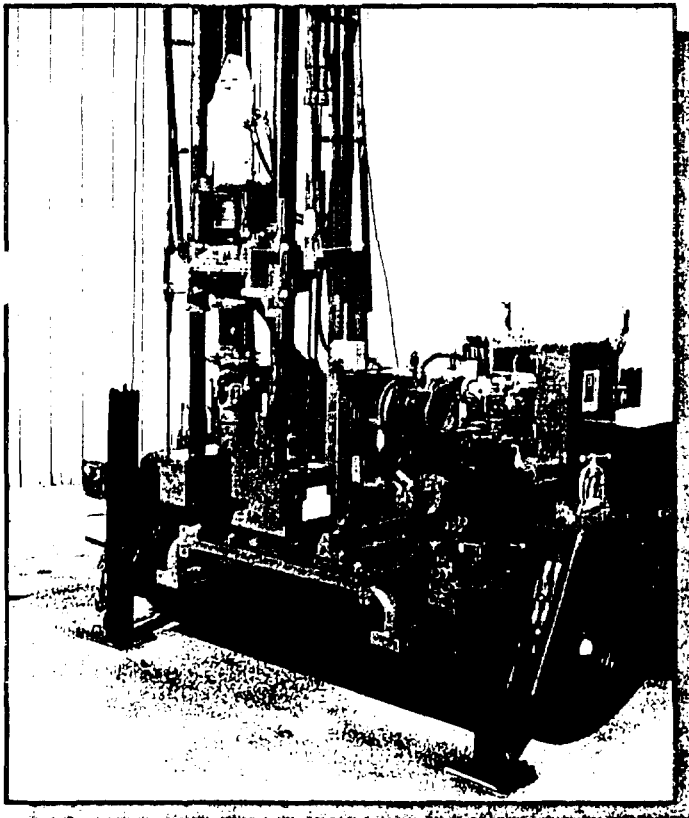
FIGURE
T-2

Attachment T-3

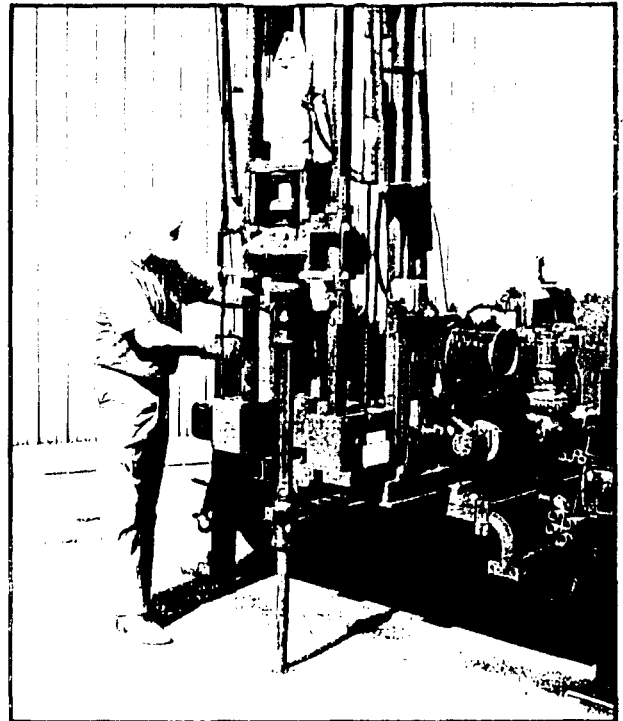
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Direct-Push Drilling Method Vendor Specifications

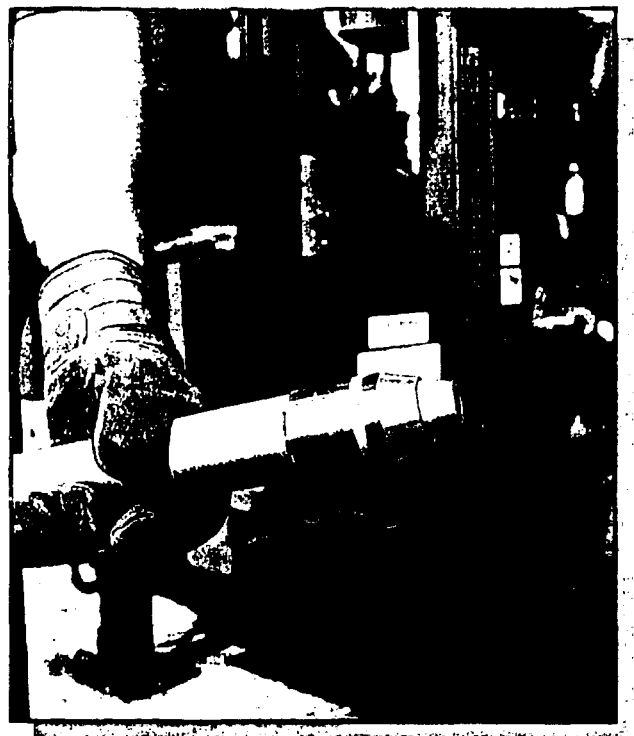
*Cuttings free
exploration with
the ESP System
eliminates
disposal problems*



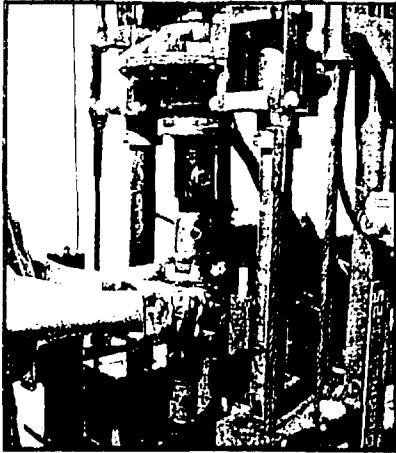
▲ The ESP System allows you to utilize your drill for maximum equipment efficiency and adapts to most open spindle drill rigs



▲ Hydraulically operated hammer fits to your drilling 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.



▲ Easy change over to drill past obstructions and continue probe through hollow stem augers eliminates multiple setups and numerous surface repairs.

Four different methods for sampling soil are available: split spoon, segmented sampler, 1.0" I.D. Shelby tube, and fixed piston sampler for soft soils or flowing sands.

Soil Gas sampling tools complete with 12 volt vacuum pump operated from rig battery capable of 22" Hg for rapid borehole evacuation are available. Assembly includes pressure gauge and separate sampling port.

An air compressor that runs off the rig battery provides 50 PSI for use in clearing concrete cuttings and air drying sampling tools.

Two discreet water sampling tools are available. Samples are gathered utilizing a peristaltic pump. The pump can be operated hydraulically by connection to drill rig system. The sample is collected through 1/4" I.D. tubing allowing faster collection using a vacuum pump or a peristaltic pump.

General water sampling is achievable through the outer tubing using a 3/4" bailer. Leading outer tube can be proved with screen section to facilitate water inflow and to stabilize water level inside tubing to control flowing sands.

Hydraulically operated grout mixer is available for rapid mixing of bentonite or cement slurries.

Hand operated diaphragm type grout pump attaches to mixing container for transferring grout to borehole. Pump has no parts to corrode and is simple and easy to clean.

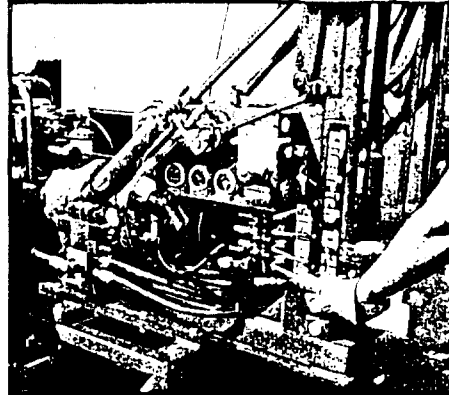
Inside diameter of outer tubing provides clearance for the installation of 3/4" I.D. piezometers.

Easy installation of sparge points by setting through outer casing or by direct drive to depth.

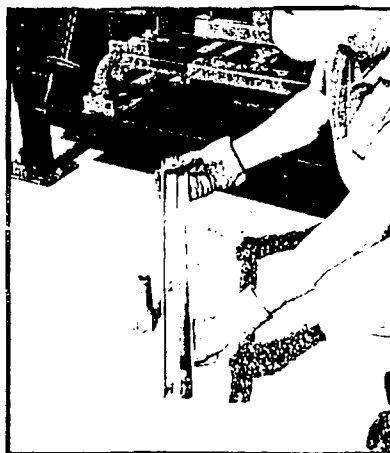
Set temporary dewatering points quickly and efficiently using hydraulically operated hammer.

The ESP System can be used to conduct subgrade investigations quickly and efficiently.

► The double rod system provides a groutable bore hole that can be properly backfilled using slurry or dry materials. Backfilling is completed the right way from the bottom up. The tremie method is available for slurries. Dry materials such as bentonite chips or granules can be placed through the outer casing. This provides protection from surface contamination through the boreholes. You leave the site the same or better than you found it.



▲ This driller friendly system uses standard controls and is simple to operate. The double rod system provides quick retrieval of samples using high speed winch lines to handle tools. The tubing is available in 2.0' or 4.0' lengths to fit existing probe power units and drilling rigs with longer feed strokes. With the Diedrich double rod system you control the bore hole to depth. There is no chance for sloughing of soils or cross contamination of samples. The sampler only contacts the soil at the shoe. Sample liners can be used to hold samples for laboratory shipments. Liners are available in butyrate plastic, brass, and stainless steel.



▲ Conventional split spoon sampling technology offers quick and easy sample handling. The 24" length provides optimum sampling span for good sample recovery and uniform spacing for accurate depth control. A 7/8" inside diameter gives good sample volume for classification and laboratory processing.



▲ ESP offers easy pavement penetration through asphalt or concrete.



▲ The change over from auger drilling to soil probing using the standard hollow stem auger connection is accomplished with ease. Rotation of tools is provided by drill rig drive train.

Enviro-Core® Sampling System

The Enviro-Core system consists of small-diameter drive casing and an inner sample barrel that are simultaneously pushed, pounded, or vibrated into the ground. Continuous soil cores are collected in stainless steel liners inside the inner barrel. After being advanced three feet, the inner barrel is retrieved, while the drive casing is left in place to prevent the borehole from collapsing. The drive casing ensures that subsequent soil samples are collected from the targeted interval, rather than potentially-contaminated slough from higher up in the borehole.

The custom-made drive casing has an outer diameter (OD) of 2.4 inches and is machined into 3-foot-long, threaded sections. A heat-treated steel drive shoe is threaded onto the bottom piece of drive casing, and a steel drive head is threaded onto the top section of casing.

The inner barrel is made of a 3-foot-long section of 1.8 inch OD, thin-walled steel tubing. The inner barrel has an OD slightly smaller than the inside diameter (ID) of the drive casing, to allow it to be raised and lowered freely inside the drive casing. The bottom of the inner barrel rests on a shoulder cut into the drive shoe.

The inner barrel contains six

1.7-inch-diameter by 6-inch-long stainless steel liners. A custom-made synthetic sample catcher is affixed to the bottom of the inner barrel to prevent loose sediments from falling out of the sample liners when the inner barrel is retrieved.

The inner barrel and drive casing are advanced into the ground simultaneously, requiring an effective means of coupling the inner barrel with the outer drive casing. This is achieved in one of two ways. The first method, referred to as the internal rod method, uses internal steel rods to keep the inner sample barrel seated against the drive shoe (Figure 1). The internal rods (three-foot sections of 1.38-inch-diameter steel EW sampling rods) serve several purposes. First, they are used to lower the empty inner barrel to the bottom of the steel-cased borehole. Then, the rods are placed in compression inside of the drive casing by attaching the drive head. This keeps

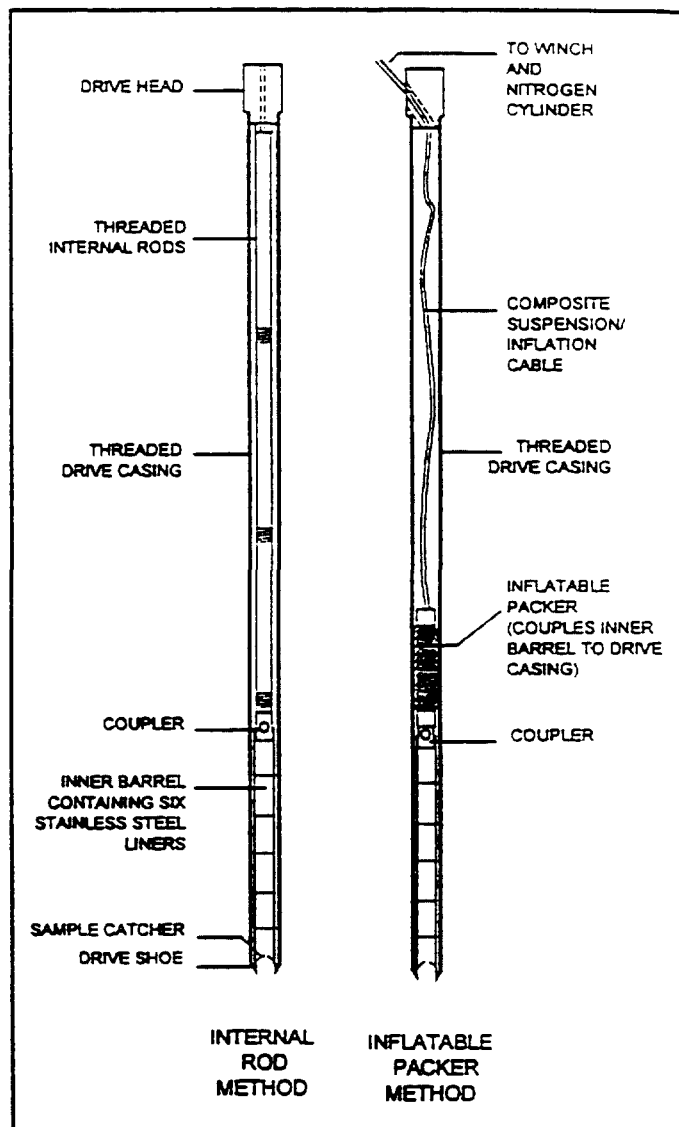


Figure 1. The Enviro-Core® System

the bottom of the inner barrel snug against the shoulder of the drive shoe, so that the soil moves easily into the sample liners as the drive casing is advanced. Finally, after the three foot run is complete and the sample liners are full of soil, the inner barrel (containing the stainless steel sample liners and soil) is retrieved by removing the internal rods.

While the internal rod method works well at shallow depths, recent improvements in the methods used to advance the Enviro-Core system have resulted in greater depths being attained. This, in turn, required a faster way to withdraw the inner barrel from the drive casing. An inflatable packer, commonly used to seal boreholes during hydraulic testing, was modified to provide a means of connecting the inner barrel to the drive casing (Figure 1). With this method, the inner barrel is coupled to the inflatable packer. The packer/inner barrel assembly is quickly lowered to the bottom of the drive casing with a wireline. The shoulder in the drive shoe prevents the inner barrel from protruding beyond the bottom of the drive casing. The packer is then inflated with compressed nitrogen, rigidly coupling the inner barrel to the drive casing. The inner barrel and drive casing are then simultaneously advanced three feet, filling the sample liners inside of the inner barrel with soil. The packer is then deflated, and the inner barrel/packer assembly is quickly removed from the boring with a winch. The sampling sequence is shown in Figure 2.

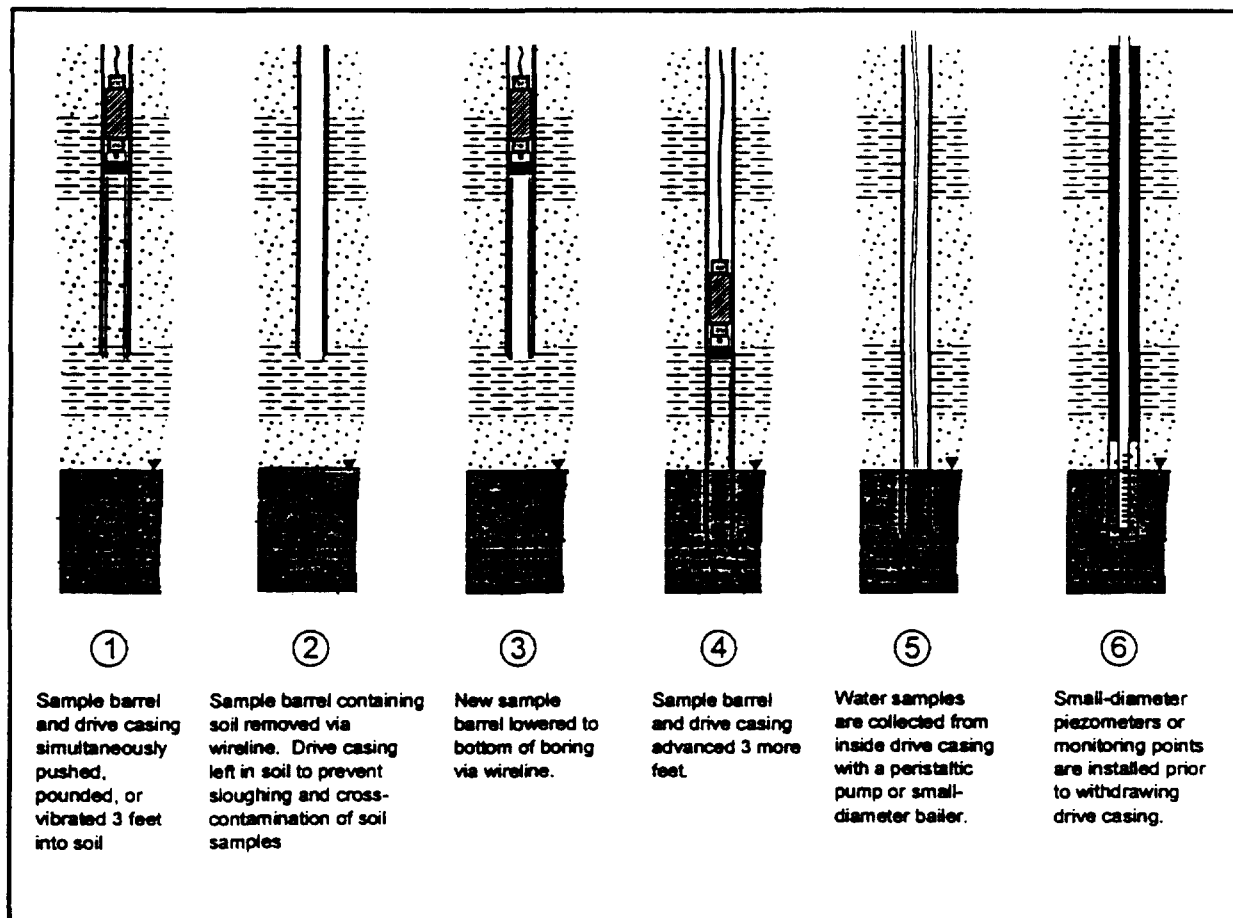


Figure 2. Sampling with the Enviro-Core® System

With both methods, after the inner barrel has been retrieved, the sample liners are removed for chemical analysis or lithologic identification. A new three-foot inner barrel, fitted with empty stainless steel liners, is quickly lowered to the bottom of the borehole. The inner barrel is then secured to the bottom of the drive casing (using either the internal rods or the inflatable packer). An additional three-foot section of drive casing is attached at the surface, followed by re-attachment of the drive head. The inner barrel and drive casing are simultaneously advanced three more feet, and then the inner barrel is again de-coupled and removed from the drive casing. This process is repeated until the desired depth is reached.

Methods for Advancing the Enviro-Core® System

Small, portable rigs push, pound, or vibrate the Enviro-Core system into the ground. The rigs are either stand-alone units, or can be mounted on Bobcat®-like construction vehicles (Figure 3) or in the back of utility trucks. To facilitate sampling beneath underground tanks and buildings, the rigs have been designed to advance the Enviro-Core system at angles up to 30 degrees from vertical.

As with most DP methods, the primary driving mechanism is the weight of the vehicle used to push the down-hole sampling equipment into the ground. The weight applied to the sampling equipment is commonly referred to as "reactive weight" or "crowd". The Enviro-Core system has been used with sampling rigs that transmit from 500 pounds to 3,500 pounds of reactive weight. As expected, the rates of penetration and maximum sampling depths are proportional to the reactive weight. Reactive weights greater than 3,500 pounds can be expected to further increase the rate of penetration and maximum sampling depth.

For maximum penetration, the Enviro-Core drive casing is usually pounded with a hydraulic hammer or vibrated with high-frequency industrial vibrators while the reactive weight is applied.

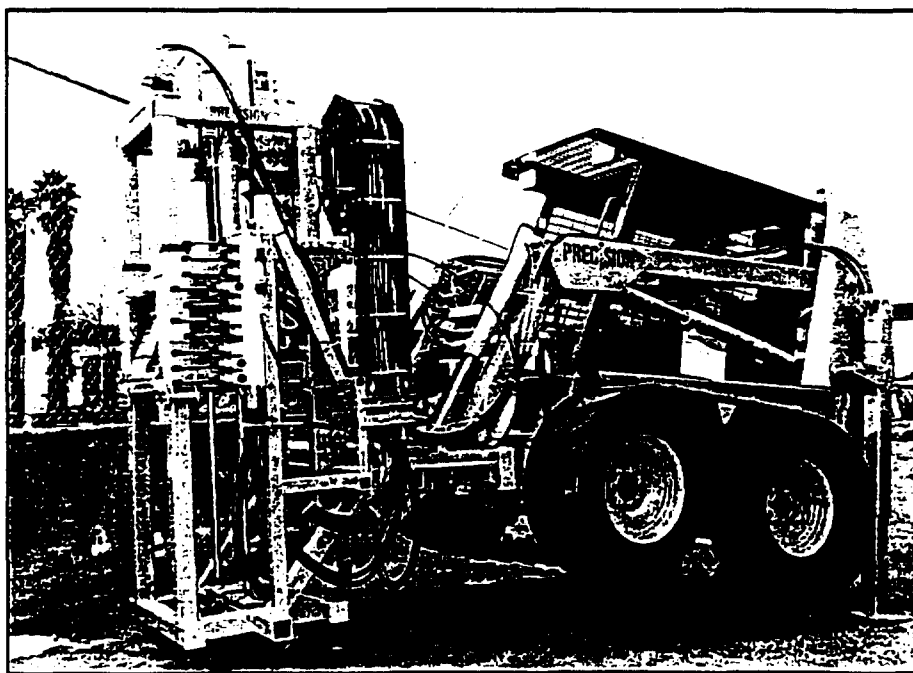


Figure 3. Precision's XD-1 Sampling Rig

The hydraulic hammer delivers 350 foot-pounds of impact force, compared to 135 foot-pounds delivered by most Geoprobe rigs. An anvil on the bottom of the hydraulic hammer transmits the striking force to the Enviro-Core drive head. The hydraulic hammer slides out of the way when not in use, to provide access to the drive casing (Figure 4).

The vibratory head is an important component of the driving mechanism and can greatly increase the rate of penetration and maximum sampling depth in certain types of soil. Vibratory techniques were initially used by the USGS for sampling unconsolidated off-shore sediments in the 1960's, and have been increasingly applied to environmental investigations. The vibrators used to advance the Enviro-Core system are twin hydraulically-operated, eccentric cam-type vibrators that clamp onto the outside of the Enviro-Core drive casing (Figure 4). Each vibrator spins at revolutions up to 4,500 revolutions per minute (rpm), creating a multi-directional vibration. When mounted opposed to one another, the opposing vibrations are canceled out, resulting in a uni-directional, up-and-down vibration (Barrow, 1994). In certain types of sediments (e.g., fine sands and silts), vibrating the Enviro-Core drive casing as the reactive weight is applied results in better sample recovery and a dramatic increase in the rate of penetration.

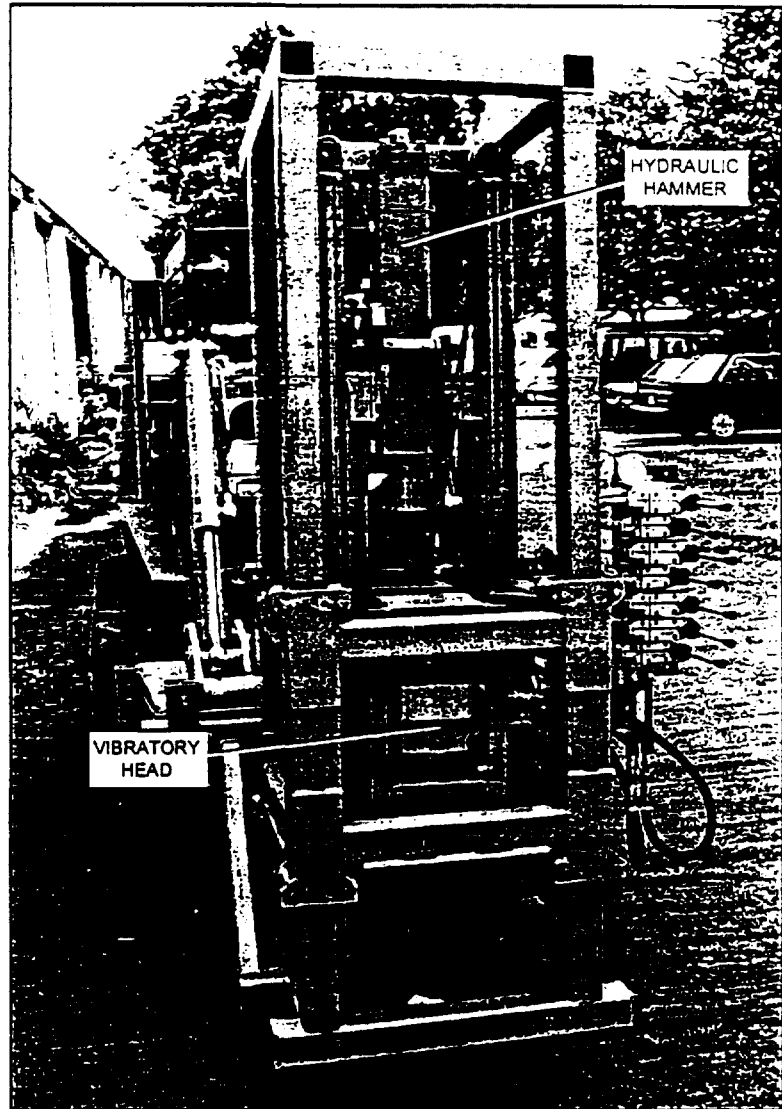


Figure 4. Components used to advance the Enviro-Core® System

The reactive weight (i.e., the weight of the sampling rig) is applied using hydraulic cylinders that press the hydraulic hammer and vibratory head down on the Enviro-Core drive casing. The rig operator can use either the hydraulic hammer or the vibrators in conjunction with the reactive weight, depending on the type of soil being penetrated. In stratified sediments, the rig operator continually alternates between pounding and vibrating the Enviro-Core system to gain the maximum sample recovery and rate of penetration.

Groundwater and Soil Gas Sampling with the Enviro-Core® System

The Enviro-Core system has several advantages over other DP tools for collecting soil gas and grab groundwater samples. First, unlike most other DP tools, the Enviro-Core is not a "blind"

Appendix U

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Magnetometer Survey Procedures

Appendix U

Magnetometer Survey Procedures

I. Introduction

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 proton magnetometer, Model G-816/G826 or the equivalent.

III. Procedures

- Step 1 - Identify the traverse location on the appropriate form (Attachment U-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 - Utilizing a Geometrics proton magnetometer, Model G-816/G826 or the equivalent, conduct the survey. Operate the magnetometer in accordance with the operating manual.
- Step 5 - Establish a base station in an area with no known buried or surface ferrous-metallic objects. Record readings at the base station every hour and at the beginning and end of each day.
- Step 6 - At each point of the grid system, record the time, station location, and magnetometer readings on a standard form (Attachment U-1), or in the magnetometer's digital memory.
- Step 7 - The data from the magnetometer surveys will be corrected for drift using the base station measurements before plotting or reporting.

Attachment U-1

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Magnetometer Survey Form

[illegible]

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Appendix V

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Seismic Refraction Survey Procedures

Appendix V

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 W

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Ground Penetrating Radar (GPR) Procedures

Appendix W

Ground Penetrating Radar (GPR) Procedures

I. Introduction

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;
- GSSI model SIR-3 radar instrument or equivalent;
- One 500 megahertz antenna (or equivalent);
- Non-conductive measuring tape; and
- Connecting cables 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 - Layout the measuring tape along the desired traverse, or mark a reference grid on the ground using the measuring tape.
- Step 4 - Connect the GPR instrumentation with appropriate cables, and adjust the instrument gains to obtain a satisfactory record throughout the desired survey depth range.
- Step 5 - 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 6 - If available, calibrate the GPR signal depth penetration over a buried pipe (or other object) of known depth. Re-calibrate the equipment if the antenna or record-length setting is changed.
- Step 7 - Conduct data analysis in accordance with the manufacturer's recommendations and industry practice.

Appendix X

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Standard Operating Procedures for Equipment Cleaning

Appendix X

Standard Operating Procedure for 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 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. Field Cleaning Procedures

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);
- 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

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 - 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. Heavy Equipment Decontamination Area Procedures

A. Decontamination of Heavy Equipment

Drilling rigs and other larger equipment utilized during sampling episodes may be cleaned at GE's Building 12Y Equipment Decontamination Area (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. Equipment Cleaning Procedure

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 health and safety plan 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 health and safety plan.

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 (1500 psi) washing chamber. Water is treated and reused.
- 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.



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