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APPENDICES

APPENDIX 7

STANDARD OPERATING PROCEDURE (SOP) GEHR3540C

- 1.0 TITLE** General Electric (GE) Hudson River Design Support Sediment Sampling and Analysis Program Standard Operating Procedure for the extraction and cleanup of sediment/solid samples for Polychlorinated Biphenyl (PCB) analysis using the Soxhlet extraction technique by SW-846 Method 3540C for subsequent analysis by SW-846 Method 8082.
- (Acknowledgement: This SOP is based substantially on internal method SOPs provided by Northeast Analytical, Inc. of Schenectady, N.Y.)
- 2.0 PURPOSE** The purpose of this SOP is to provide to the chemist the procedures required to perform extractions of PCBs, in sediment/solid sample, using the soxhlet extraction technique and to perform the subsequent extract volume reduction and cleanup for the GE Hudson River Design Support Sediment Sampling and Analysis Program.
- 3.0 SCOPE** The following procedure is utilized by the project laboratories for the extraction and cleanup of PCBs from sediment/solid samples using the soxhlet extraction method for analysis by SW-846 Method 8082.
- 4.0 COMMENTS** The automated solvent extraction may be used in place of the soxhlet extraction at the discretion of the supervising chemist. Time restraints (*i.e.* requested turn around time) may render this method inapplicable, as it requires 18 +/-2 hours of extraction reflux time.

5.0 SAFETY The chemist should have received in-house safety training and should know the location of first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment. Safety glasses and gloves must be worn when handling glassware and samples.

Polychlorinated biphenyls have been tentatively classified as known or suspected carcinogens. The chemist must review the Material Safety Data Sheets (MSDS) for PCBs and all reagents used in the procedure before handling them. All equipment and solvents should be handled within a lab fume hood.

6.0 REQUIREMENTS The chemist must have an understanding of the methods and requirements of USEPA-SW- 846A "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition. Methods 3540C, 3500B, 3620B, 3665A, 3660B. An approved instructor must also certify the chemist to perform the procedure.

7.0 EQUIPMENT

7.1 Water Cooled Condenser: Pyrex 45/50 #3840-MCO or equivalent.

7.2 250mL Round Bottom Flask: Pyrex #4100 or equivalent.

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

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- 7.4 Heating Mantle: Type "VF" laboratory heating mantle #HM0250VF1. (or equivalent)
- 7.5 Heating Mantle Controller: Glass-Col #PL3122 Minitwin (or equivalent) regulates temperature control of the mantle.
- 7.6 Analytical Balance: Mettler AG-204 (or equivalent) used to determine sample mass.
- 7.7 Cellulose Extraction Thimble: Contains sample during soxhlet extraction.
- 7.8 Sodium Sulfate: Anhydrous (12-60 Mesh), washed with hexane and baked overnight. Used for the laboratory method blank.
- 7.9 Boiling Chips: Chemware PTFE Boiling Stones P#0919120 (or equivalent)
- 7.10 Chiller: Pump driven water circulating cooling system cool flow #75 NESLABS Instruments, Inc. (or equivalent)
- 7.11 Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208 (or equivalent).
- 7.12 Acetone: High Purity Solvent Baxter (Burdick/Jackson) #UN1090 (or equivalent).
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- 7.13 TurboVap Evaporator: Zymark #ZW640-3 (or equivalent).
- 7.14 TurboVap Evaporator concentrator tubes: Zymark 250 mL (or equivalent), 0.5 mL endpoint.
- 7.15 Beakers: Assorted Pyrex: 250 mL, 600 mL, and 1000 mL, used for liquid containment and pipette storage.
- 7.16 1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.
- 7.17 Vials: glass, 8 dram & 4 dram (with Polyseal sealed cap) (20 mL & 10 mL) capacity, for sample extracts.
- 7.18 Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.
- 7.19 Centrifuge: International Equipment Co., Model CL (or equivalent).
- 7.20 Wrist Shaker: Burrell wrist action shaker, Model 75 and 88 (or equivalent).
- 7.21 Florisil[®]: 10% deactivated.
- 7.22 TBA Reagent: Tetrabutylammonium Hydrogen-Sulfite Reagent (prepared in the laboratory).
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- 7.23 Mercury: Triple distilled Mercury Waste Solutions, Inc, (or equivalent).
- 7.24 Sulfuric Acid: H₂SO₄ (concentrated) Mallinkrodt #2468 #UN1830 (or equivalent).
- 7.25 Pipettes: S/P Disposable Serological Borosilicate Pipettes.
1. 1 mL × 1/10
 2. 5 mL × 1/10
 3. 10 mL × 1/10
- Fisher Pasteur Borosilicate glass pipette 9" #72050 (or equivalent)
- 7.26 4 oz. Jars: Industrial Glassware

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, these problems should be brought to the attention of the supervisor and/or quality assurance manager for guidance to proceed and then documented in the extraction logbook.

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- 8.1.2 If the sample is sediment and contains a water layer, decant and discard the layer as aqueous PCB waste. Mix the sample thoroughly and discard any foreign objects such as sticks, rocks or leaves. **Note:** however that the sample may be composed entirely of rock, concrete or some other solid material in which case the entire sample is treated as the solid.

8.2 Sample Extraction

- 8.2.1 Fill a Pyrex pan with ice cubes and cold water about 1/2 inch deep. As the samples are weighed out, place the beakers or 4-oz. jars in the Pyrex pan to chill for at least 15 minutes prior to the drying step.
- 8.2.2 Rinse all extraction thimbles with hexane to remove extraneous material. Place thimble into a 100-mL beaker and allow to dry.
- 8.2.3 Set up one 250-mL glass beaker or 4-oz. jar for each sample. Using the first sample, label a beaker with the sample number, and tare the beaker. Using a metal spatula, add 10 g to 11 g of the wet sample to the beaker. Samples that are observed to be very wet will require additional mass of sample such that the project sensitivity requirements are met. The moisture content of the sample as determined in Section 8.2.4 should be evaluated so that a larger wet-weight sample can be obtained to provide a dry amount of solids to meet the project sensitivity requirements. The amount taken must consider the size limitations of the Soxhlet thimble. The laboratory should target a wet-weight amount of 15 g for very wet samples. Record the weight in the PCB

solid extraction logbook to the nearest tenth of a gram. At this point, a sample for percent total solids may also be taken (see 8.2.4). Place the beaker in the ice bath to chill.

- 8.2.4 The PCB concentration is to be determined on a dry weight basis and therefore, the percent total solid must be determined. Weigh approximately 5 grams of the previously homogenized sample in a previously weighed, aluminum weighing pan. Record the weight of the pan and the weight of the (pan and sample) in the percent total solids log. Place the sample in a drying oven at 100 to 110°C for at least 8 hours. Record the time placed in the oven and the oven temperature in the percent total solids log. Remove the samples from the drying oven and allow to cool in a desiccator. Weigh the pan and sample.

Calculate the percent solids by:

$$\frac{\{(wt. of pan + dried sample) - (wt. of pan)\} \times 100\%}{(wt. of wet sample)}$$

NOTE: ALL SAMPLE CONTAINERS ARE TO BE RETURNED TO THE APPROPRIATE REFRIDGERATOR. FOR ALL EMPTY SAMPLE CONTAINERS, SEE THE LABORATORY'S INTERNAL CHEMICAL HYGIENE PLAN FOR PROPER DISPOSAL.

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- 8.2.5 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 metal spatula. If the sample has not dried after a few minutes, another 10 g may be added. Once the sample is well-dried and free flowing, transfer the sample to a pre-rinsed extraction thimble. 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.
- 8.2.6 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 the sample number. Place the corresponding thimble into the soxhlet extractor. Rinse corresponding beaker & metal spatula with a few pipettes 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. The final extract volume concentrations of the surrogate compounds tetrachloro-*meta*-xylene (TCMX) and decachlorobiphenyl (DCB) should be 10 ng/mL and 100 ng/mL, respectively. At this time, the GE Hudson River Design Support Sediment Sampling and Analysis Program does not require the preparation and analysis of matrix spike and/or matrix spike duplicate samples. If requested in the future, the final extract volume
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concentration for the spiked Aroclor (Aroclor-1242) in the matrix spike and matrix spike duplicate sample should be 20,000 ng/mL (**Note:** this spike concentration will require a sample dilution to be performed). The final extract volume concentration of the laboratory control sample (LCS) should be 500 ng/mL.

- 8.2.7 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.
- 8.2.8 Place the round bottom flask with attached soxhlet extractor onto a heating mantle and attach condenser unit. Turn corresponding thermostats on to a temperature that will achieve 4-6 solvent cycles per hour. At this time double check soxhlets 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.
- 8.2.9 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. Turn off the chiller and 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.
- 8.2.10 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
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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.

8.2.11 Rinse the soxhlet with several pipettefull of hexane and tip again to drain into the round bottom. Set the soxhlet aside at this time. Procure the same number of TurboTubes 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 drops out of the mouth of the round bottom. Rinse the round bottom with several pipettefull of hexane, swirl gently, and decant into same TurboTube. Repeat this step twice for same sample then repeat all preceding steps for all other samples.

8.2.12 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 and allow to heat up to the specified temperature for individual solvent use.
- 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 gas pressure regulators is off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty TurboTube into the water bath and close the lid. Make sure that the nitrogen gas pressure regulator is turned off. Bleed any residual gas until the regulator output pressure gauge reads "0" psi. Proceed to 8.3.4. Make sure to wipe down all surfaces with Hexane before concentrating samples.
- 8.3.4 Place the TurboTube 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 5.0 mL. Remove the samples from the TurboVap and place in the rack. **Note:** Not all samples will evaporate at the same rate; sample extracts containing large amounts of petroleum or other non-volatile liquids may stop reducing before the 5.0 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 Pasteur pipette into an appropriate volumetric flask (25 mL for soil extracts). Rinse the TurboTube with 3 Pasteur pipettes of hexane, and then transfer the hexane rinse to the volumetric. Repeat the hexane rinse two more times for a total of three Hexane rinses of the TurboTube. After the sample has been transferred, rinse the Pasteur pipette with 0.5 mL of hexane into the volumetric flask. Add hexane to the volumetric meniscus mark. Invert the volumetric flask at least three times to mix completely. Decant the contents into a pre-labeled 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.

Sulfuric acid, sulfur removal and Florisil[®] clean-ups should be performed on every sample. The sample preparation chemist in the extraction logbook records the sequence and number of repeats of cleanup steps performed.

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

- 8.4.1 Sulfuric Acid Wash
- 8.4.2 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds that are co-extracted with the PCB residues.
- 8.4.3 Chill the sample to approximately 0°C. Add 5.0 mL of concentrated H₂SO₄ and shake for 30 seconds by hand, centrifuge for approximately 1 minute, transfer approximately 20 mLs of the Hexane upper layer to an 8-dram vial.

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- 8.4.4 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 Elemental Sulfur Clean-up

- 8.5.1 Elemental sulfur is soluble in the extract solvents used for sediment and soil samples. It is commonly found 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 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 minutes. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental sulfur.

8.6.2 Remove the sample extracts from the wrist shaker and place in the centrifuge at a setting and duration appropriate to spin down the solids.

8.6.3 Transfer the sample extract to a new 8-dram vial.

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

8.7 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.

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- 8.7.2 Add 2.0 mL TBA Sulfite Reagent, 1.0 mL 2-propanol, and approximately 0.65 g of sodium sulfite crystals to the extract and shake for at least 5 minutes on the wrist shaker and observe. An excess of sodium sulfite must remain in the sample extract during the procedure. If the sodium sulfite crystals are entirely consumed add one or two more aliquots (approximately 0.65 g) to extract and observe.
- 8.7.3 Place the samples on the wrist shaker for 45 minutes observing at 15-minute intervals to make sure that the sodium sulfite is not consumed. Add 5 mL organic free water and shake for ten minutes.
- 8.7.4 Place the samples into the centrifuge at a setting and duration appropriate to spin down the solids.
- 8.7.5 Transfer the hexane layer to a new 8-dram vial and cap.
- 8.8 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 tested and approved 10% deactivated Florisil[®] to each vial containing the sample extract.

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- 8.8.3 Vigorously shake the vial for approximately 1 minute by hand or on the wrist shaker.
- 8.8.4 Place the vial(s) into the centrifuge at a setting and duration appropriate to spin down the solids.
- 8.8.5 Transfer the extract to a clean 8-dram vial.

8.9 Extract Screening and Dilution:

- 8.9.1 Screening PCB extracts by GC to determine the approximate concentration before final analysis is highly recommended. If possible, prior site history and estimates of sample concentration will be provided by the field personnel and may be used to determine what, if any, extract dilution is necessary.
- 8.9.2 The supervising chemist is responsible for determining initial screening dilutions. Extract dilutions are prepared by transferring an aliquot of the original sample extract into a vial containing the correct amount of "make up" volume of hexane. Dilutions must be recorded in the instrument logbook or in the data system.
- 8.9.3 Perform the dilution using appropriate disposable volumetric pipettes to transfer the extract and to add the make-up volume of hexane. Make sure that the vial is properly labeled. Cap and invert the vial at least three times to thoroughly mix the extract with the solvent.

8.9.4 Transfer 1 mL of the extract to a labeled 1.5-mL GC autosampler vial. Record the screening dilution along with the extract volume, the sample mass, and the percent total solids. Submit the information with the sample extracts to the GC analyst.

9.0 QUALITY CONTROL

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

9.2 9.2 With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation steps (including clean-up steps). For sediment/solid samples, a laboratory sodium sulfate blank is processed.

9.3 At this time, the GE Hudson River Design Support Sediment Sampling and Analysis Program does not require the preparation and analysis of matrix spike and/or matrix spike duplicate samples. If requested in the future, a matrix spike for Aroclor-1242 is to be analyzed at a rate of 1 matrix spike per every 20 samples at a concentration of 20,000 ng/mL in the extract (**Note:** this spike concentration will require a sample dilution to be performed). Also a matrix spike duplicate sample is to be analyzed at a rate of 1 per every 20 samples.

9.4 A QC reference check standard (Laboratory Control Sample [LCS]) is also prepared and analyzed for Aroclor-1242 at a concentration of 500 ng/mL in the extract. For sediment/solid samples, sodium sulfate is used.

9.5 Surrogate compounds are added to each sample, matrix spike, matrix spike duplicates, duplicate, method blank, and QC reference check standard LCS at time of extraction. The surrogate compounds TCMX and DCB are to be added for final extract concentrations of 10 ng/mL and 100 ng/mL, respectively.

10.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

10.1 Pollution Prevention: see laboratory's internal SOPs

10.2 Waste Management: see laboratory's internal SOPs

11.0 REFERENCES

1. U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.
2. "Guide to Environmental Analytical Methods", Third Edition, Genium Publishing Corporation, 1996.

12.0 ATTACHMENTS

1. Method Outline

**ATTACHMENT 1
PCBs IN SOIL/SEDIMENT
OUTLINE FOR SOXHLET EXTRACTION**

- 1. PREPARE SAMPLE FOR EXTRACTION**
- 2. RINSE EXTRACTION THIMBLES**
- 3. WEIGH SAMPLE AND RECORD WEIGHT**
- 4. DRY SAMPLES**
- 5. ADD SAMPLE TO THIMBLES**
- 6. SET UP SOXHLET EXTRACTOR APPARATUS**
- 7. ADD SURROGATES, MATRIX SPIKE, AND LABORATORY CONTROL SAMPLE SPIKE**
- 8. EXTRACT SAMPLE FOR APPROXIMATELY 16 HOURS**
- 9. BREAKDOWN SOXHLET EXTRACTOR APPARATUS**
- 10. TRANSFER SOLVENT TO TURBOTUBE**
- 11. SOLVENT REDUCTION, USING THE ZYMARK TURBOVAP EVAPORATION SYSTEM**
- 12. TRANSFER AND SET VOLUME**
- 13. EXTRACT CLEANUP (ACID, MERCURY OR TBA, AND FLORISIL)**
- 14. EXTRACT DILUTION**
- 15. GC SCREENING/ ANALYSIS**