ROUTINE ANALYSIS OF PCBs IN WATER AND SOIL/SEDIMENT SAMPLES BY GC/ECD (EPA/SW-846 Methods 3500B/3510C/3540C/3541/8000B/8082) (EPA/SW-846 Methods 3600C/3620B/3640A/3660B/3665A - Optional)

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CONTENTS

CONTENTS (cont)

CONTENTS (cont'd)

* These sections affected by Revision 2.0

SUPERSEDES: SOP #1801, Revision 1.0, 11/27/05: U.S. EPA Contract EP-W-09-031

ROUTINE ANALYSIS OF PCBs IN WATER AND SOIL/SEDIMENT SAMPLES BY GC/ECD (EPA/SW-846 Methods 3500B/3510C/3540C/3541/8000B/8082) (EPA/SW-846 Methods 3600C/3620B/3640A/3660B/3665A - Optional)

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is applicable to the determination of polychlorinated biphenyls (PCBs) in water and soil/sediment matrices, using a gas chromatograph (GC) with a narrow-bore fused silica column and an electron capture detector (ECD). This SOP is based on Environmental Protection Agency (EPA) Methods SW846/3510C/3540C/3541/8000B/8082 and those requirements set forth in the latest approved version of the National Environmental Laboratory Accreditation Committee (NELAC) Quality Systems section. Extracts may be subjected to optional cleanup procedures (Florisil, gel permeation chromatography [GPC], tetrabutylammonium [TBA] sulfite, activated copper powder or acid) based on EPA/SW-846 Methods 3600C/3620B/3640A/3660B/3665A. The compounds of interest and typical reporting limits (RLs) in water and soil/sediment matrices are found in Table 1, Appendix A.

This method may not be changed without the expressed approval of the Organic Group Leader, the Analytical Section Leader and the Quality Assurance Officer (QAO). Only those versions issued through the SERAS document control system may be used. Modifications made to the procedure due to interferences in the samples or for any other reason must be documented in the case narrative and on a nonconformance memo.

2.0 METHOD SUMMARY

2.1 Water Samples

Approximately 1 liter (L) of a water sample is serially extracted at a neutral pH with methylene chloride. The extract is concentrated to 10 milliliters (mL), then 60 mL of hexane is added as an exchange solvent, and the extract is concentrated to a final volume of 1 mL. The extracts are analyzed for PCBs using GC/ECD. A second column confirmation is optional for PCB analysis.

2.2 Soil Samples

Approximately 30 grams (g) of a soil/sediment sample mixed with 30 g of anhydrous sodium sulfate is extracted with 140 milliliters (mL) of 1:1 acetone/hexane using a Soxtherm extractor for 2 hours or 300 milliliters (mL) of 1:1 acetone/hexane using a Soxhlet extractor for 16 hours. The extract is concentrated to 10 mL, 60 mL of hexane is added as an exchange solvent, and the extract is concentrated to a final volume of 5 mL. The extracts are analyzed for PCBs using GC/ECD. A second column confirmation is optional for PCB analysis.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Storage

Water samples should be collected in 1-L amber glass containers fitted with Teflon-lined caps. Soil samples should be collected in wide-mouth glass containers with Teflon-lined caps.

From the time of collection until after analysis, extracts and unused samples must be protected from light and refrigerated at 4 ± 2 degrees Celsius (°C) for the periods specified by SERAS Task Leader (TL) and/or the Work Assignment Manager (WAM) for the project.

Samples and sample extracts must be stored separately from standards in an atmosphere free of all potential contaminants.

3.2 Holding Time

Extraction of water and soil/sediment samples must be completed within 7 days from the date of collection and analysis completed within 40 days of sample extraction.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences in the sample extracts for analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing laboratory reagent blanks on a routine basis. Interferences co-extracted from the samples may vary considerably from sample to sample. Cleanup procedures may be necessary if the extract contains analytes that interfere with quantitation or peak separation.

Phthalate esters are present in many types of products commonly found in the laboratory. Some plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

Soap residue on glassware may cause degradation of certain analytes. This problem is especially pronounced with glassware that may be difficult to rinse. These items should be hand-rinsed very carefully to avoid this problem.

Elemental sulfur is encountered in many sediment samples such as marine algae and some industrial wastes. Sulfur will be quite evident in gas chromatograms obtained from electron capture detectors. If the GC is operated under normal conditions for PCBs analysis, the sulfur interference can completely mask the region from the solvent peak through most of the Aroclor peaks. Three techniques, GPC cleanup, activated copper powder, or TBA sulfite for the elimination of sulfur may be used. Florisil cleanup may be used to reduce matrix interferences caused by polar compounds.

Weathering of PCBs in the environment may alter the pattern of the PCBs to the point where the pattern is not recognizable. "Weathering" is defined as a change in the typical PCB pattern. Selected Aroclor peaks may be used for quantitation when a sample exhibits a pattern similar to this effect.

In some instances, the presence of multiple PCBs may affect the identification and quantitation of all PCBs present in a sample. In this case, the analyst will note interferences/anomalies in the case narrative and document the reasoning for reporting the Aroclors present.

5.0 EQUIPMENT/APPARATUS

The following equipment/apparatus is required:

- Soxtherm extractor, including its accessories (e.g., extraction flask, sample holding vessel, chiller, etc.),

manufactured by Gerhardt or equivalent

- Waters GPC instrument or equivalent
- Separatory funnel, 2000 mL with stopcock (glass or Teflon).
- Erlenmeyer flasks, 500 mL
- Graduated cylinder, 1-L, Class A
- Buchner funnels.
- Bench top shaker (Glas-Col) or equivalent.
- Soxhlet extractor, 40 millimeter (mm) inner diameter (ID), with 500-mL round bottom flask, fits 45/50 condenser
- Teflon boiling chips, approximately 10/40 mesh, rinsed three times with methylene chloride
- Spoon and/or spatula, stainless steel or Teflon
- Glass container(s)
- Glass wool, Pyrex baked at 400^{o}C for 2 hours
- Balance, capable of accurately weighing 100 g to the nearest 0.01 g
- Kuderna-Danish (K-D) apparatus, consisting of a 10-mL graduated concentrator tube, 500-mL evaporation flask, and three-ball macro Snyder column
- Water bath heated with concentric ring cover, capable of maintaining temperature within ±2°C. The bath should be used in a hood.
- Disposable glass pasteur pipettes
- Nitrogen evaporation device, equipped with a water bath that can be maintained at 35-40°C (N-Evap by Organomation Associations Model Number 111 or equivalent)
- TurboVap concentrator, with concentrator cells and racks
- Clean Bath solution, for use in TurboVap II concentrator
- Drying oven
- Desiccators

- Vials and caps, 2 mL for GC autosampler
- Vials, 4-mL, for GPC cleanup
- Test tubes with screw caps, 25 mL
- pH paper, wide range.
- Ring stand
- Gas chromatograph - An analytical system complete with GC and all required accessories including syringes, autosampler, analytical columns, gases, an electron capture detector, and data system. A data system is required for measuring peak areas or peak heights and recording retention times.

  RTX-XLB fused silica capillary column, 30 meter (m) x 0.32 mm ID, 0.50 micron (µm) film Thickness or equivalent

  RTX - CLPesticides fused silica capillary column, 30 m x 0.32 mm ID, 0.50 µm film thickness or Equivalent
- Cellulose/Fiberglass thimbles , pre-washed with methylene chloride
- Teflon filters, 0.45µm, for filtering extracts for gel permeation chromatography (GPC) cleanup (Gelman Acrodisc CR or equivalent)
- Florisil cartridge, 12-mL tube (Supelco CAT # 57155 or equivalent)
- Visiprap SPE Vacuum manifold, 12 port or equivalent
- Valve liners, disposable or equivalent.
- Syringes, miscellaneous
- Class “S” weight for balance calibration

6.0 REAGENTS

- Sodium Sulfate, anhydrous granular reagent grade, heated at 400 C for four hours, cooled in a desiccator, and stored in a glass bottle
- Methylene Chloride, pesticide residue analysis grade or equivalent
- Hexane, pesticide residue analysis grade or equivalent

- Acetone, pesticide residue analysis grade or equivalent
- Methanol, pesticide residue analysis grade or equivalent
- 2-Propanol, pesticide residue analysis grade or equivalent
- Tetrabutylammonium sulfite solution - Prepare by dissolving 3.39 g of tetrabutylammonium hydrogen sulfate in 100 mL of reagent water. Extract this solution three times with 20 mL portions of hexanes to remove any impurities. Discard the hexane layer and add 25g of sodium sulfite to the aqueous layer. Store this solution at room temperature.
- Copper powder, activated, commercially available
- Pesticide/PCB Internal Standard Solution. Prepare a solution containing 4,4'-Dibromooctafluorobiphenyl, 4,4'- Dibromobiphenyl, and 3,3',4,4'-Tetrabromobiphenyl at concentration of 5 microgram/milliliter (µg/mL) in hexane.
- PCB Stock Calibration Standards, 1000 µg/mL commercially available.
- PCB Working Calibration Standards - Prepare a minimum of five concentration levels at 0.25, 0.5, 1.0, 2.0, and 5.0 milligrams/liter (mg/L). Each calibration standard must contain surrogates at a concentration range of 20, 50, 100, 200 and 500 micrograms/liter (µg/L) and contain the pesticide/PCB internal standard compounds at concentration of 100 µg/L.
- Surrogate Stock Standard, 200 mg/L, commercially available.
- Pesticide/PCB Surrogate Working Solution - Prepare a solution containing decachlorobiphenyl (DCBP) and 2,4,5,6-tetrachloro-meta-xylene (TCMX)at a concentration of 0.2 µg/mL (for water)and 2 µg/mL (for soil) in methanol or acetone.
- Stock PCB Matrix Spiking Solution, 1000 mg/L, commercially available, source must be independent of the calibration standards.
- PCB Matrix Spike Working Solution - Prepare a spiking solution in methanol or acetone that contains Arolclor 1016 and Aroclor 1260 at a concentration of 10 µg/mL. Depending on the project, another Aroclor may be used for the matrix spike solution.
- Sodium hydroxide (NaOH), 10 Normal (N)-Weigh out 40g of NaOH and dissolve in 100mL of deionized water.
- Sulfuric acid, concentrated
- Sulfuric acid (H_2SO_4), 1:1-Add and equal volume of concentrated H_2SO_4 to an equal volume of deionized water.
- Deionized (DI) water, Type II or equivalent

7.0 PROCEDURES

7.1 Sample Preparation and Extraction for Water

1. Transfer the sample container into a fume hood. Mark the meniscus of the sample level with an indelible marker, and pour the sample into a 2-liter separatory funnel. Check the pH of the sample with wide range pH paper and record it in the extraction log. If the pH is not neutral, adjust the pH between 5 and 9 with 10 N sodium hydroxide and/or 1:1 sulfuric acid solution. Pour the tap water into the sample bottle to the meniscus line. Measure the tap water volume using a 1 liter graduated cylinder and record the volume on the extraction log.

2. Prepare a method blank and laboratory control sample (LCS) by transferring 1-L of DI water into a 2-L separatory funnel. A method blank and LCS must be prepared for every 20 samples or per batch.

3. Measure two additional 1-L portions of sample for use as a matrix spike and matrix spike duplicate (MS/MSD) at a rate of one MS/MSD per every 10 samples or 10%.

   NOTE: This sample may be specified on the chain of custody record for this purpose by the SERAS Task Leader.

4. Add 1 mL of the 200 nanograms/milliliter (ng/mL) surrogate working solution to the method blank, LCS, MS/MSD and all the samples or add sufficient volume to result in a final concentration of 200 parts per billion (ppb) in the final extract.

5. Add 100 µL of the 10 µg/mL Aroclor spiking solution to the LCS and MS/MSD or add sufficient volume to achieve a final concentration of 1.0 parts per million (ppm) in the final extract if a higher extract volume will be obtained.

6. Rinse the sample bottle with 60 mL of methylene chloride, transfer the rinsate to the separatory funnel and extract the sample by shaking the funnel for two minutes, with periodic venting to release excess pressure. Allow the organic layer (generally the bottom layer) to separate from the water phase. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the extraction chemist must employ mechanical techniques to complete the phase separations. The optimal techniques employed depend upon the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical means. If using a bench top shaker, vent and release excess pressure, place it on shaker and shake for 5 minutes.

7. Filter the extract (bottom layer) through a funnel containing glass wool and anhydrous sodium sulfate into a 500-mL Erlenmeyer flask. Add a second 60-mL portion of methylene chloride to the separatory funnel and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner. After the third extraction, rinse the sodium sulfate in the funnel with

sufficient methylene chloride. If using the bench top shaker, shake the sample(s) for 3 minutes for the 2nd and 3rd extractions.

7.2 Sample Preparation and Extraction for Soil

1. In a fume hood, place 140 mL of 1:1 acetone/hexane into a Soxtherm extraction vessel containing 2 boiling chips. If using Soxhlet extraction, place 300 mL of 1:1 acetone/hexane in a 500-mL round bottom flask containing 3 or more clean boiling chips.

2. Transfer the sample container into the fume hood. Open the sample bottle and discard any foreign objects such as sticks, leaves, and rocks. Mix the sample thoroughly.

3. Calibrate the balance with Class "S" Weights prior to weighing samples or on a daily basis when the balance is in use. The balance should be calibrated with a weight that is similar to the weight used to extract the samples (i.e., 30 g).

4. Weigh approximately 30 g of each sample to the nearest 0.1g into a glass container and add a sufficient amount (approximately 30-100g) of anhydrous granular sodium sulfate. Mix well. The sample should have a sandy texture at this point. A method blank and LCS must be prepared by using 30 g of sand (or baked sodium sulfate) according to the same procedure as the samples, at the frequency of one per 20 samples or per batch.

5. Transfer sample to a pre-cleaned extraction thimble. Place the thimble containing samples to extraction beaker. For Soxhlet extraction, take a piece of baked glass wool and place it in the Soxhlet extractor so it covers the bottom of the inner diameter. Add some sodium sulfate to hold the glass wool in place; this will prevent any soil/sediment from being caught and clogging the Soxhlet. Add the blended sample and anhydrous sodium sulfate into the Soxhlet extractor on top of glass wool and sodium sulfate.

6. Weigh two additional 30 g portions of the sample chosen for spiking to the nearest 0.1 g for use as a MS/MSD at a rate of one per ten samples per project, or ten percent.

   NOTE: This sample may be specified on the Chain of Custody record for this purpose by the SERAS Task Leader.

7. Add 0.5 mL of the 2 µg/mL surrogate working solution to the method blank, LCS, MS/MSD and all the samples or add sufficient volume to result in a final concentration of 200 ppb in the final extract.

8. Add 0.5 mL of the 10 µg/mL matrix spiking solution to the LCS and MS/MSD or add sufficient volume to achieve a final concentration of 1.0 µg/mL in the final extract.

9. Attach the extraction vessel to the Soxtherm Extractor and extract for 2 hours. The Soxtherm extraction conditions are specified at Appendix C. For Soxhlet extraction, attach condenser to the extractor and flask, and extract the sample(s) for 16 hours.

NOTE: Care must be taken to supervise the beginning of the extraction to ensure that the condenser is cooling the evaporating solvent sufficiently to guarantee that the solvent will condense and continue to extract the sample in a continuous cycle for the entire extraction. Allow the extract to cool after the extraction is complete.

7.3 Sample Concentration for Water and Soil Extracts

1. If concentrating using a TurboVap apparatus, skip to step 5. Otherwise, assemble a Kuderna-Danish (K-D) apparatus by attaching a 10-mL concentrator tube to a 500-mL evaporation flask. Transfer the extract to the K-D concentrator

2. Add one or two clean boiling chips to the evaporation flask and attach a three-ball Snyder column. Place the K-D apparatus on a hot water bath (70 to 75 C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Add approximately 1 mL of hexane to the top of Snyder column. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid is below 10 mL, add another 60 mL of hexane and evaporate down to below 10 mL. Remove the K-D apparatus, and allow it to drain and cool.

   NOTE: DO NOT ALLOW THE EXTRACT TO GO TO DRYNESS. However, if the extract goes to dryness, document the situation in the extraction logbook.

3. Remove the Snyder column; use 1-2 mL of hexane to rinse the flask and its lower joint into the concentrator tube. Remove the concentrator tube and place it onto the N-Evap preheated to 35 C.

4. Evaporate the extract to a final volume of 1 mL for water and 5 mL for soil. During evaporation, rinse the wall of the concentrator tube with 1-2 mL of hexane. Continue with step 7.

   NOTE: DO NOT ALLOW THE EXTRACT TO GO TO DRYNESS. However, If the extract goes to dryness, document the situation in the extraction logbook.

5. If using the TurboVap concentrator, fill the TurboVap water bath with approximately one gallon of deionized water mixed with 10-15 drops of Clean Bath solution. Set the water bath temperature at 55 C.

6. Transfer the extract to the concentrator cells in the hood. Begin concentrating by blowing a gentle stream of nitrogen into the cells so that no solvent is splashed out. As the solvent level is reduced, add any remaining extract, rinse the flask with hexane, and add the rinsate to the concentration cell. Once all the extract has been transferred to the concentrator cell and the solvent level is well below the 200-mL mark, the flow of nitrogen can be increased to speed up the concentration. Periodically rinse the cell with hexane. Concentrate the extract below 10 mL, add 60 mL of hexane and concentrate it down to a final volume of

1mL of water and 5 mL for soil.

NOTE: DO NOT ALLOW THE EXTRACT TO GO TO DRYNESS. However, If the extract goes to dryness, document the situation in the extraction logbook.

1. Take a 4-mL aliquot of the sample extract from Steps 4 or 6 and proceed to the optional GPC cleanup for soil in Section 7.5, optional Florisil cleanup described in Section 7.6, optional TBA-sulfite cleanup described in Section 7.7 or optional copper cleanup described in Section 7.8, and sulfuric acid cleanup described in section 7.9. Store the remaining extract(s) at 4 C± 2 C.

   Note: Record the date and the applicable samples subjected to cleanup on the extraction log.

7.4 Total Solids

The sample aliquots for total solids are weighed in conjunction with the samples for the extraction. The total solids for the MS/MSD are based on the corresponding sample. The blank is assumed 100% total solids.

Weigh and record (in the % solid logbook) an empty aluminum sample dish to the nearest 0.01 g. Weigh at least 10 g of the soil/sediment into the aluminum dish and record the weight. Preheat the oven to 103 – 105^{O}C. Place the aluminum dishes in the oven and record the initial temperature in the logbook. Determine the percent total solids by drying the sample(s) in an oven that is placed inside a fume hood overnight. In the morning, record the final temperature of the oven in the logbook. Turn off the oven and allow the dishes to cool in the desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of the soil or sediment. Calculate the percent total solids using the following equation.

7.5 Gel Permeation Chromatography Cleanup

1. Calibrate the GPC instrument by injecting 10 µL of GPC standard (corn oil, bis (2-ethylhexyl) phthalate, methoxychlor, perylene, and sulfur) and eluting it with methylene chloride to establish collection time window to collect the fraction from the beginning of ethoxychlor peak to the end of perylene peak.

2. When the collection time window has been established, inject a methylene chloride blank to make sure all calibration components are washed from the column.

3. Before injecting the samples, dilute the 1 mL water extracts to 4 mL including method blanks, LCSs and MS/ MSDs with methylene chloride. Filter the 4 mL of each extract through acrodisc CR PTFE filter (Gelman, 0.45µm) into a clean 4-mL vial.

   Load the 4-mL vials, which contain pre-filtered extracts onto the autosampler and start the sequence.

Collect the cleaned extracts from the fraction collector, transfer to the concentrator tubes and concentrate the extracts to a final volume of 2 mL using TurboVap.

$$\%\,Total\ Solids = \frac{Weight\ of\ Dried\ Sample\ with\ Dish\ (g) - Dish\ Weight\ (g)}{Weight\ of\ Wet\ Sample\ with\ Dish\ (g) - Dish\ Weight\ (g)}$$

NOTE: GPC Pump flow rate is 5.0 mL/minute
GPC Run time is 25 minutes

7.6 Florisil Cleanup

Florisil cleanup significantly reduces matrix interferences caused by polar compounds.

1. Place one Florisil cartridge into the manifold for each sample extract to be subjected to cleanup.

2. Prior to the cleanup of samples, the cartridges must be washed with 90:10 hexane/acetone. This is accomplished by passing through at least 10 mL of the hexane/acetone solution through each cartridge. NOTE: DO NOT ALLOW THE CARTRIDGES TO DRY AFTER THEY HAVE BEEN WASHED.

3. After the cartridges in the manifold are washed, a rack containing labeled 25-mL concentrator tubes is placed inside the manifold. Care must be taken to ensure that the solvent line for each cartridge is placed inside of the appropriate concentrator tube as the manifold top is replaced.

4. After the concentrator tubes are in place, add approximately 1 mL of the 90:10 hexane/acetone solution to the Florisil bed in the cartridge. Allow the solvent to pass into the sorbent bed and immediately transfer 2 mL from each sample, blank and MS/MSD extract from Section 7.3, Step 7 to the top of the Florisil bed in the appropriate Florisil cartridge.

5. The extracts are then eluted through the cartridge with 18 mL of 90:10 hexane/acetone and collected in 25-mL concentrator tubes held in the rack inside the manifold. NOTE: Be sure to add the 18 mL of mobile solution immediately after the 2-mL extract crosses the Florisil bed.

6. Transfer the concentrator tubes from step 5 to the TurboVap LV and concentrate the extracts to a final volume of 2 mL using nitrogen blow down.

7.7 Tetrabutylammonium (TBA)-Sulfite Cleanup

Elemental sulfur is encountered in many soil/sediment samples. The solubility of sulfur in the extraction and exchange solvents is very similar to the multi component PCBs; therefore, the sulfur is extracted along with the PCBs. If the GC is operated under normal conditions for PCB analysis,

the sulfur interference can completely mask the region from the solvent peak through PCB patterns. This cleanup is used to remove the sulfur interference.

1. Transfer the extract from Section 7.3, Step 4 or Section 7.5, Step 5, to a 25-mL test tube.

2. Add 2 mL of TBA-sulfite reagent and 2 mL of 2-propanol; cap and shake vigorously with a mechanical shaker such as Vortex for at least two minutes. If the sample is colorless or if the initial color is unchanged, and if clear crystals (precipitated sodium sulfite) are observed, sufficient sodium sulfite is present. If the precipitated sodium sulfite disappears, add more TBA-sulfite reagent until a solid residue remains after repeated shaking.

3. Add 6 mL of deionized water and shake for at least two minutes. Allow the sample to stand for 5-10 minutes. Transfer the hexane layer (top) to two 1-mL injection vials.

7.8 Copper Cleanup

Copper cleanup requires that the copper powder be very reactive.

1. Transfer the sample extract from Section 7.3, step 7 or Section 7.5, step 5 or Section 7.6, step 6 to a 4-mL screw-top vial.

2. Add approximately 0.5 to 2 g of copper powder (depends on the color and viscosity of sample) to the vial. Vigorously mix the extract and copper powder for at least 1 minute on a mechanical shaker such as Vortex. Allow the copper to settle.

3. Separate the extract from the copper by drawing off the extract with a disposable glass pipette into two 1-mL injection vials.

7.9 Sulfuric Acid Cleanup

Rigorous sulfuric acid cleanup is suitable for the sample extracts of PCBs. Acid cleanup must be used whenever elevated baselines or overly complex chromatograms prevent accurate quantitation of PCBs.

1. Transfer 2 mL of sample extract from Section 7.3 step 7 or Section 7.5, step 5 or Section 7.6 step 6, or Section 7.7 Step 3 to a 4-mL screw-top vial .

2. Add 2 mL of concentrated sulfuric acid and cap the vial tightly and vortex for one minute. A vortex must be visible in the vial.

3. Allow the phases to separate for at least 1 minute. Transfer the top (hexane) layer into the injection vial for analysis on GC/ECD. If the extract is colored, repeat the step for 2nd and 3rd time by transferring the hexane layer to another 4-mL vial.

4. If there is separation problem, a centrifuge can be used to separate the layers.

7.10 GC/ECD Conditions

Sample analyses are performed using a Hewlett Packard (HP) 6890 GC/ECD, equipped with dual injector, column, and electron capture detector capabilities.

The HP 6890 conditions used for the PCB analysis are listed below:

Injector Temperature	250°C
Oven Temperature Program	120°C hold for 1 minute (min) 9°C/min to 285°C, 10 min at 285°C
Detector Temperature	300°C
Carrier Gas	Helium
Make-up Gas	Argon/Methane
Column Flow Rate	RTX-XLB 3.0 milliliters/minute (mL/min); RTX-CL Pesticides 1 mL/min
Amount Injected	1 microliter (µL)
Data System	HP Chem Station

The instrument conditions listed above are guidelines to be used for standards and sample analysis on a HP 6890 GC/ECD system. Any suitable conditions may be used as long as quality control criteria and peak separation is achieved.

2

7.11 Retention Time Windows

Due to advances in electronic pressure controls in modern GCs such as the HP6890, the RTs usually remain constant and may exhibit a negligible shift (nearly zero) over the traditional 72-hour period. A default standard value of ±0.030 minutes will be used for the two surrogates and a value of ±0.020 minutes will be used for the internal standards. These default values will be applied unless the instrument and EPC unit cannot maintain constant retention times. If the instrument cannot maintain reproducible RTs, the analyst must investigate the cause and implement corrective action

7.12 Standard and Sample Analysis

The analytical sequence listed in Figure 1, Appendix B must be followed.

1. Prepare calibration standards as in Section 6.0. For the initial determination of PCB presence in a sample extract, inject 1 uL each of the five PCB 1016/1260 working calibration standards. Choose five peaks (peaks must be >25% of full scale) to calculate response factors (RFs). Once the type of Aroclor is known or suspected, inject 1 uL each of the five working calibration standards for those Aroclors. The average RF and percent relative standard deviation (%RSD) must also be calculated for each Aroclor peak on the column used for quantitation, using the equations below.

$$RRF = \frac{(A_X)(C_{IS})}{(A_{IS})(C_X)}$$

where:

A_X = Peak Area or Peak Height of each target analyte
A_{IS} = Peak Area or Peak Height of each internal standard assigned to target analytes
C_{IS} = Concentration of each internal standard (ng/mL)
C_X = Concentration of each target analyte (ng/mL)

$$RRF_{avg} = \frac{RF_1 + \ldots + RF_5}{5}$$

$$SD = \sqrt{\frac{\sum_{i=} \quad - \quad ^2}{N - \ }}$$

$$\%RSD = \frac{SD}{RRF\ average} \times 100$$

2. External Standard Method

Prepare calibration standards as in Section 6.0. For the initial determination of PCB presence in a sample extract, inject 1 uL each of the five PCB 1016/1260 working calibration standards and tabulate the peak height or peak area for each standard concentration. Calculate the response factor (RF) for each compound at each standard concentration using the equation below. Once the type of Aroclor is known or suspected, inject 1 uL each of the five working calibration standards for those Aroclors. The average RF and percent relative standard deviation (%RSD) must also be calculated for both columns using the equations above in Section 7.9.1 Step 3.

$$RF = \frac{Peak\ Height\ or\ Peak\ Areao\ f\ the\ Analyte}{Mass\ Injected\ (\ pg\ )}$$

The %RSD (average of all 5 peaks) for each Aroclor must be ≤20.0% for the internal standard method and external standard method. If analytes are reported from both columns, the %RSD must be supplied for both columns.

NOTE: An initial calibration curve must be run every six months at a minimum or sooner if the daily calibration check doesn't meet the required percent difference (%D) as

specified in Section 7.12, Step 4.

NOTE: If a sample extract is initially run for pesticides/PCBs and it is determined that no PCBs are present, a PCB initial calibration will not be run.

3. Inject 1 ppm each of the remaining Aroclors (Ar), Ar 1016, Ar 1232, Ar 1242, Ar 1248, Ar 1254, Ar 1260, and Ar 1268 and 2 ppm of Ar 1221 for the fingerprints.

   NOTE: The fingerprints are used for qualitative pattern matching only. The fingerprints need not be run with each initial calibration curve as long as the date of the fingerprints do not exceed one year or a new source of standards is received.

4. For every 12 hours of sample analysis, inject the 1 ppm mid-point calibration standard. Calculate and tabulate the %D for each Aroclor peak using the following equation:

$$Percent\ Difference(\%D) = \frac{ARF_{INT} - RF_{CALC}}{A_{INT}} x\ 100$$

where:
ARF_{INT} = Initial Average Response Factor
RF_{CALC} = Calculated Response Factor

Note: the %D for the Daily Check standard must be ≤ 15%

5. Inject a group of sample extracts. It is recommended to inject the method blank and LCS first. All sample extracts must be analyzed within 12 hours of the injection of the 1 ppm continuing calibration Aroclor standard (step 4).

6. Repeat steps 4 and 5, if necessary, until the %D requirement of the continuing calibration check fails or the sample sequence is complete.

7.13 Evaluation of Chromatograms

All standard and sample chromatograms must be evaluated to determine if re-injection and/or dilution is necessary.

7.13.1 Standard/Sample Chromatograms

The following requirements apply to all data presented for multi component analytes.

1. The PCB chromatograms must display the multi-component analytes present in each standard or sample at greater than (>) 25% and less than (<) 100% of full scale. The chromatogram must be printed in landscape mode with the time scale of the chromatogram from approximately 5 (before the TCMX) to 25 minutes (after the DCBP peak). If the time scale is modified, all chromatograms will be scaled the same

for comparative purposes.

2. If an extract is diluted, chromatograms must display multi-component PCBs between 10% and 100% of full scale.

3. If a chromatogram is re-plotted electronically to meet requirements, the scaling factor used must be displayed on the chromatogram.

4. If a chromatogram indicates carryover from a previous injection, subsequent sample extract(s) must be re-analyzed, preferably immediately.

5. The retention time of each surrogate and internal standard must fall within the RT window criteria in Section 7.10. If the RT window has shifted in the thousandth place (>0.030 but <0.040), professional judgment may be used to determine the acceptability of the data. If the RT is shifted by more than 0.040 minutes, the analytical sequence (acquisition) must be interrupted for corrective action. After corrective action, acquisition of data can be resumed only after an acceptable 1 ppm PCB standard is obtained. No retention time windows are required for PCBs analysis since the identification of PCBs is done by pattern recognition.

6. If a sample chromatogram has interfering peaks, a high baseline, or off-scale peaks, the sample extract must be re-analyzed using a dilution, further cleanup, or re-extraction. Samples that do not meet acceptance criteria after one re-extraction and cleanup must be reported in the case narrative and do not require further analysis.

7. A sample which under going additional cleanup must also include the method blank

8. If it is determined that the matrix may be causing a RT shift, the internal standard and the MS/MSD in conjunction with the original sample chosen for spiking may be used to assess matrix effects.

9. If manual integrations have been performed, refer to SERAS SOP #1001, *Chromatographic Peak Integration Procedures* for appropriate documentation.

7.13.2 PCB Identification

The identification of PCBs is based on pattern recognition, which can only be verified from an on scale chromatogram. Second column confirmation is optional for PCB analysis.

7.14 Sample Dilution

Target compound concentrations must not exceed the upper limit of the initial calibration range. If analytes are detected in the extract at a level greater than the highest calibration standard, the extract must be diluted (to a maximum of 1:100,000) or until the analyte response is within the linear range established during calibration. Guidance in performing dilutions and exceptions to this

requirement are given below.

1. If the analyst has reason to believe that diluting the final extracts will be necessary based on historical data or visual observation of the extracts, an undiluted run may not be required. However, if no peaks are detected above 25% of full scale on the diluted sample, analysis of a more concentrated sample extract or the undiluted sample extract is required.

2. If the response is still above the highest calibration point after the dilution of 1:100,000, the analyst should contact the Organic Group Leader immediately for further instruction.

3. The results of the original analysis are used to determine the approximate dilution factor required to bring the largest analyte peak within the initial calibration range.

4. The dilution factor chosen should keep the response of the largest peak for a target compound in the initial calibration range of the instrument.

5. Submit data for any reportable analyses.

6. All chromatograms for the dilution analyses must meet the requirements described in Section 7.13.

8.0 CALCULATIONS

Reporting of target compounds and surrogates can be performed on any column that passed all the quality control (QC) criteria specified in this SOP. In order to be quantitated, the detector response (peak area or peak height) of all the analytes must lie within the calibration range.

8.1 Reporting Limit for Water

$$RL\ (\mu L) = \frac{(C_{STD})(V_T)(DF)}{(V_O)}$$

where:

C_{STD} = Concentration of the lowest standard in the calibration range (µg/mL)
V_T = Volume of the extract (mL)
DF = Dilution factor
V_O = Volume of water extracted

The typical RL for each analyte can be found in Appendix A.

8.2 Reporting Limit for Soil

$$RL\ (\mu g/kg)\ dry\ weight = \frac{(C_{STD})(V_T)(DF)}{(V_O)(S)}$$

where:

C_{STD}	=	Concentration of the lowest standard in the calibration range (µg/mL)
V_T	=	Volume of the extract (mL)
DF	=	Dilution factor
V_O	=	Volume of water extracted (L)
S	=	Decimal Percent Solids

The typical RL for each analyte can be found in Appendix A.

8.3 Sample Concentration for Water Using Internal Standard Method

Use the following equation to calculate the concentration of the identified analytes using the relative response factor (RRF) obtained from the initial calibration curve.

$$Concentration\ (\mu g/L) = \frac{(A_X)(I_S)(V_T)(DF)}{(A_{IS})(RRF_{avg})(V_O)(V_i)}$$

where:

A_X	=	Peak Area or Peak Height of each target analyte
I_S	=	Amount of each internal standard injected (ng)
V_T	=	Volume of the concentrated extract (mL)
DF	=	Dilution factor
A_{IS}	=	Peak Area or Peak Height of each internal standard
RRF_{avg}	=	Average Relative response factor
V_o	=	Volume of water extracted (L)
V_i	=	Injection volume (usually 1 µL)

8.4 Sample Concentration for Water Using External Standard Method

$$Concentration\ (\mu g/L) = \frac{(A_X)(V_T)(DF)}{(RF_{avg})(V_O)(V_I)}$$

where:

A_X	=	Peak area or peak height for the compound to be measured
V_T	=	Volume of the concentrated extract in microliters (µL)
DF	=	Dilution factor
RF_{AVG}	=	Average response factor
V_O	=	Volume of water extracted in milliliters (mL)

V_I = Volume of extract injected in microliters (µL)

8.5 Sample Concentration for Soil Using Internal Standard Method

Identified target analytes will be quantitated by the internal standard method. The internal standard used must be the one nearest the retention time to that of the given analyte listed in Table 2, Appendix A. Use the following equation to calculate the concentration of the identified analytes using the relative response factor (RRF) obtained from the initial calibration curve.

$$Concentration\ (\mu\ /kg) = \frac{(A_X)(I_S)(V_T)(DF)}{(A_{IS})(RRF_{avg})(W)(S)(V_i)}$$

where:

A_X = Peak Area or Peak Height of each target analyte
I_S = Amount of each internal standard injected (ng)
V_T = Volume of the concentrated extract (mL)
DF = Dilution factor
A_{IS} = Peak Area or Peak Height of each internal standard
RRFavg= Average Relative response factor
W = Weight of soil/sediment extracted (kg)
S = Decimal percent solid
V_i = Injection volume (usually 1 µL)

8.6 Sample Concentration for Soil Using External Standard Method

Unusual sample matrices which interfere with the internal standards, and/or unusual circumstances during analysis may warrant the use of the external standard method at the discretion of the Organic Group Leader.

$$Concentration\ (\mu\ /kg)\ dry\ weight = \frac{(A_X)(V_T)(DF)}{(RF_{avg})(W)(V_I)(S)}$$

where:

A_X = Peak Area or Peak Height for the compound to be measured
V_T = Volume of the concentrated extract (mL)
DF = Dilution factor (if any)
RF_{AVG} = Average response factor
W = Weight of soil/sediment extracted (kg)
V_I = Volume of extract injected (µL)
S = Decimal percent solids

NOTE: If any analytes are detected below the quantitation limit at >25% of the quantitation limit, report the concentration and flag as estimated (J).

8.7 Surrogate Spike Recoveries

$$Percent\ Recovery\ (\%R) = \frac{Q_D}{QA}\ x\ 100$$

where:

Q_D = quantity determined by analysis
Q_A = quantity added to sample

8.8 Matrix Spike Recoveries

The percent recoveries and the relative percent difference (RPD) between the recoveries of each MS and MSD are calculated and reported using the following equations:

$$Matrix\ Spike Recovery(\%R) = \frac{SSR - SR}{SA}\ x\ 100$$

where:

SSR = spike sample result
SR = sample result
SA = spike added

$$RPD = \frac{|MSR - MSDR|}{(MSR + MSDR)/2}\ x\ 100$$

where:

RPD = Relative Percent Difference
MSR = matrix spike recovery
$MSDR$ = matrix spike duplicate recovery

The vertical bars in the formula above indicate the absolute value of the difference; hence, RPD is always expressed as a positive value.

8.9 Laboratory Control Sample Recoveries

The recoveries of each of the compounds in the LCS solution will be calculated using the following equation:

$$Laboratory\ Control\ Sample\ Recovery\ (\%R) = \left(\frac{LCSR - 3}{SA}\right) x\ 100$$

where:

LCSR = Concentration of target analyte in LCS
B = Concentration of target analyte in blank
SA = Concentration of spike added

8.10 Dixon's Criterion

Dixon's criterion at the 95% confidence level is used to identify statistical outliers in a data. This test should be applied sparingly and never more than once to a single data set. For weathered samples, all peaks are included in the quantitation even if any of the peaks fail Dixon's criterion.

To determine if a peak may be omitted from the average of the peaks used for quantitation, arrange the results in ascending order (X_1, X_2, ...X_n). If X_n is to be tested as an outlier, use the following equation:

$$\frac{X_n - Y_{n-}}{X_n - Y_1}$$

Reject X_n as an outlier if the ratio is greater than Dixon's Criterion for n (see Table 2, Appendix A)

If X_1 is to be treated as an outlier, use the following equation:

$$\frac{X_2 - Y_1}{X_n - Y_1}$$

Reject X_1 as an outlier if the ratio is greater than Dixon's Criterion for n (see Table 2, Appendix A).

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

9.1 Holding Time

Extraction of soil/sediment samples must be completed within 7days of sampling, and analysis completed within 40 days of sample extraction.

9.2 Identification of Target Compounds

The identification of multi-component PCBs is based primarily on pattern recognition; secondary column confirmation is optional.

1. If <3 peaks are used in quantitation, the analyst will report the result as estimated and flag as "J". Document in the case narrative the reason why the original five peaks were not used for the quantitation.

2. For samples in which several Aroclors can be identified as present and quantitation would be difficult due to overlapping peaks with other Aroclors, the analyst will note the interference in the case narrative and report data based on the major Aroclor(s) present.

The Organic Group Leader will discuss whether the lower concentration Aroclor(s) is (are) significant for the project with the SERAS Task Leader.

9.3 Initial Calibration for Target Compounds and Surrogates

For the initial determination of PCBs, prior to the analysis of any sample, method blank, LCS or MS/MSD, the GC/ECD system must be initially calibrated with a minimum of five 1016/1260 working calibration standards to determine the linearity range. If reporting data from only one column, the %RSDs will be reported from only that column. If reporting data from both columns, all corresponding calibrations will be required for both columns. Once the type of Aroclor is known or suspected, the GC/ECD system must be calibrated with a minimum of five working calibration standards for the particular Aroclors known or suspected to be present to determine the linearity range.

1. The concentration of all calibration standards that are specified in Section 6.0 must be used.

2. The standards are to be analyzed according to the procedures given in Section 7.12 using the GC operating conditions in Section 7.10.

3. The response factors are determined according to the procedure in Section 7.12.

4. The initial calibration is also evaluated on the basis of the stability of the response factors of each target compound and surrogate. The average %RSD of each five peak must and the two surrogates must not exceed 20.0%.

9.4 Continuing Calibration for Target Compounds and Surrogates

Once the GC/ECD system has been calibrated, the calibration must be verified each 12-hour time period during which samples are analyzed. If reporting data from only one column, the %D will be reported only for that column. If reporting data from both columns, all corresponding %Ds will be required for both columns.

1. The %D for the average of five peaks and surrogates must not exceed 15.0%.

2. The continuing calibration is evaluated on the GC column used for analysis in the instrument.

3. If the %D exceeds 15.0% for a specific analyte and the analyte is present in the sample extract, re-injection is required to ensure an accurate concentration.

4. If the end of sequence (EOS) exceeds 15.0% for the average of the 5 peaks, the samples must be re-analyzed. If the EOS does not meet acceptance criteria after re-analysis, it must be documented and further analysis is not required if the failing compounds are not detected in the associated samples. The analytical sequence may be altered on the reanalysis by introducing additional calibration check standards at a greater frequency.

9.5 Retention Time Windows

A default standard value of ±0.030 minutes will be used for the two surrogates. Since the identification of multi-component PCBs by GC methods is based primarily on pattern recognition of a group of peaks, specific retention time windows are not used for the individual PCB peaks.

1. The retention time shifts of the surrogates and internal standard may be used to evaluate the stability of the GC system during the analysis of standards only.

2. Retention time windows must be established on both columns for the surrogates and submitted with the data package.

9.6 Analytical Sequence

The standards and samples analyzed by this SOP must be analyzed in the sequence outlined in Figure 1, Appendix B. This sequence includes requirements that apply to the initial and continuing calibrations, as well as the analysis of samples.

9.7 Method Blank and Laboratory Control Sample Analysis

A method blank and LCS are a known volume of deionized water or a known weight of a clean reference matrix (pure sand or baked sodium sulfate) that is carried through the entire analytical procedure. The volume or weight used for the method blank must be approximately equal to the volume or weight of the samples associated with the blank. The purpose of the method blank is to determine if there are any contaminants associated with the processing and analysis of samples.

1. For PCB analysis only, a method blank and LCS must be prepared for each group of up to 20 samples extracted at the same time or per batch and analyzed on each GC/ECD system used to analyze samples.

NOTE: If a sample has been extracted for pesticides/PCBs, the LCS will contain pesticide compounds only.

2. A method blank must not contain any of the compounds listed in Table 1, Appendix A at concentrations greater than or equal to ( ) the reporting limit.

3. The method blank must be analyzed on all the instrument on which samples were analyzed.

4. All samples associated with an unacceptable method blank must be re-extracted and re-analyzed if sufficient volume or mass is available and the holding time has not been exceeded. Otherwise, it will be documented in the case narrative of the data package.

5. When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the sample cleanup procedure.

6. Method blank results must not be subtracted from any associated samples.

Use 70 to 130% for LCS recoveries until ranges are established. LCS %recovery less than 70% must be reported to the Organic Group Leader.A solvent blank may be used to check for contamination or carryover from a previous sample. If an analyte present in the solvent blank is found in subsequent samples, the samples should be re-analyzed. A solvent blank may also be run prior to any standard

9.8 Surrogate Recoveries

1. Surrogates are added to each sample, blank, LCS, MS, and MSD prior to extraction at the concentrations described in Sections 6.0 and 7.1.

2. The surrogate spike recoveries are calculated according to the procedures in Section 8.7.

3. The quality control limit for both surrogate recoveries is 30 - 150%. These limits are only advisory, and no further action is required if the limits are exceeded. However, frequent failures to meet the limits for surrogate recovery warrant investigation by the laboratory.

9.9 Internal Standards

1. Add 20 µL of 5 ppm internal standard to all standards and samples including the lab blanks, LCSs and MS/MSDs or add sufficient amount to achieve a final concentration of 100 µg/L.

2. The peak height (or area) of the internal standards in each sample must be monitored by the analyst and to assure the height (or area) falls between 50% and 150% of the corresponding internal standard in the daily calibration check. If any the internal standards do not meet the criteria (50% to 150%), then it will be flagged with (*).

Note: Only the internal standards used to calculate the target compounds will be monitored

3. If one or more internal standard areas do not meet criteria, the GC system must be inspected for malfunctions and corrections made as appropriate. When corrections are made, re-analysis of all affected samples is required. If re-analysis is not feasible due to matrix interference (i.e., coeluting with IS peak) on both columns, the analyst may choose to dilute the sample to remove the interference instead of re-analyzing.

4. If after re-analysis, the areas for all internal standards meet criteria (between 50% and 150%), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, only data from the analysis with the ISs within limits are required to be submitted. If re-analysis confirms matrix effects, submit both sets of data but report the initial run

9.10 Matrix Spike and Matrix Spike Duplicate Analysis

The purpose of spiking target compounds into two aliquots of a sample is to evaluate the effects of the sample matrix on the methods used in this SOP.

1. The MS/MSD must be prepared with every 10 samples per matrix or per project, whichever is more frequent.

2. For PCB only analysis, the matrix spiking solution specified in Section 6.0 must be used and result in the concentration specified in Sections 7.1 and 7.2.

   NOTE: If a sample has been extracted for pesticides/PCBs, the MS/MSD will contain pesticide compounds only.

3. The MS/MSD recoveries and the % RPD are calculated according to the procedures in Section 8.8. There are no acceptance limits for the PCB MS/MSD recoveries.

4. Dilution and re-analysis of the MS/MSD is required to bring the target analytes within the linear range of the calibration curve. Dilution and re-analysis of the MS/MSD is not performed for non-target analytes outside the calibration range.

5. The MS/MSD must be extracted and analyzed within holding time.

9.11 Initial Demonstration of Capability

Initial proficiency in PCBs analysis must be demonstrated by each analyst initially and each time significant changes are made in the procedure or for instrumentation. Each analyst will generate precision and accuracy data using a reference standard other than the source used for calibration. Four replicate of a well-mixed reference standard is analyzed using the procedures outlined in this SOP. Calculate the average mean in µg/kg for soil and µg/L for water and the standard deviation (S) in µg/kg for soil and µg/L for water. The QAO will tabulate the results from all of the analysts per matrix per parameter, and calculate control limits.

9.12 Dilution Analysis

If the concentration of any sample extract exceeds the initial calibration range, that sample extract must be diluted and re-analyzed as described in Section 7.12. If there are no peaks detected above 25% of the full scale in the dilution analysis, a lower dilution of the sample extract must be analyzed.

9.13 Reporting Limit

The lowest concentration of the calibration standard that is analyzed during the initial calibration determines the method reporting limit based on the initial volume or weight of the sample, final volume of extract obtained from the extraction, and percent total solids, if appropriate.

9.14 Method Detection Limit Studies

Method detection limit (MDL) studies will be run on an annual basis for the water and soil matrix to verify the minimum concentration that can be measured and reported with 99% confidence. A minimum of seven replicates will be used for the study (EPA 1984).

9.15 Nonconformance Memo

A nonconformance memo will be generated any time an employee notices a deficiency suspected of being a nonconformance. This nonconformance memo will be forwarded to the QAO for verification of corrective action.

10.0 DATA VALIDATION

Data will be assessed in accordance with the guidelines set forth in the most recent version of the SERAS data validation SOPs. However, data is considered satisfactory for submission purposes when ALL the requirements mentioned below are met.

1. All samples must be analyzed as part of a valid analytical sequence, i.e., an acceptable initial calibration, and continuing calibration check at the required frequency.

2. All the QC requirements described in Section 9.0 must be met at all times. Any deviations or anomalous conditions should be discussed with the Organic Group Leader and must be documented in the case narrative.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, refer to U.S. EPA, Occupational Safety and Health Administration (OSHA) and corporate health and safety practices. More specifically, refer to SERAS SOP #3013, SERAS *Laboratory Safety Program* and SERAS SOP #1501, *Hazardous Waste Management*.

12.0 REFERENCES

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13.0 APPENDICES

A - Tables
B - Figures
C - Soxtherm Extractor Operating Conditions

APPENDIX A
Tables
SOP #1801
January 2006

TABLE 1. Target Compound List and Typical Reporting Limits for Water and Soil

Analyte	Water RL (µg/L)	Soil RL (µg/kg)[1]
Aroclor 1016	0.250	41.7
Aroclor 1221	0.500	83.3
Aroclor 1232	0.250	41.7
Aroclor 1242	0.250	41.7
Aroclor 1248	0.250	41.7
Aroclor 1254	0.250	41.7
Aroclor 1260	0.250	41.7
Aroclor 1268	0.250	41.7

RL denotes Reporting Limits
[1] Reported on a wet weight basis

TABLE 2. Target Compound List and Internal Standards

4,4'-Dibromooctafluorobiphenyl (IS)
TCMX
Aroclor 1016
Aroclor 1221
4,4'-Dibromobiphenyl (IS)
3,3',4,4'-Tetrabromobiphenyl (IS)
Aroclor 1232
Aroclor 1242
Aroclor 1248
Aroclor 1254
Aroclor 1260
Aroclor 1268
DCBP

TABLE 3. Dixon's Criterion for n

Number of results, n	Dixon's Criterion
3	0.941
4	0.765
5	0.642
6	0.560
7	0.507

APPENDIX B
Figures
SOP #1801
January 2006

FIGURE 1. Multi Component PCB Analytical Sequence

Time	Injection #	Material Injected
	1-5	Initial Calibration (5-point)
	6-13	PCB Fingerprints
0 hr.	14	1st sample
12 hr.	n	1 ppm Continuing Calibration Standard and/or End of Sequence
	n+1	1st Sample
	:	
12 hr	○	*1 ppm Continuing Calibration Standard and/or End of Sequence*
	:	Samples
	:	
another 12 hr.	○	1 ppm Continuing Calibration Standard and/or End of Sequence
	:	
	:	Samples
	:	
another 12 hrs	o	1 ppm Continuing Calibration Standard and/or End of Sequence:
	:	
	:	Samples
	:	
	etc.	
	last	1 ppm Continuing Calibration Standard and/or End of Sequence

NOTE: All subsequent 12-hour periods are timed from the injection of the mid-point calibration standard. The analytical sequence must end with a continuing calibration check standard.

APPENDIX C
Soxtherm Extractor Operating Conditions
SOP #1801
January 2006

ROUTINE ANALYSIS OF PCBs IN WATER AND SOIL/SEDIMENT SAMPLES BY GC/ECD
(EPA/SW-846 Methods 3500B/3510C/3540C/3541/8000B/8082)
(EPA/SW-846 Methods 3600C/3620B/3640A/3660B/3665A - Optional)

Soxtherm Extraction Operating Conditions for PCBs

Extraction Temp.	Solvent Volume	Solvent Type	Hot Extraction Time	Evaporation A	Rinsing	Evaporation B Interval	Evaporation C
160	140 ml	hex/ace (1:1)		5x Interval			
Time Elapsed			50 min	25 min	30 min	0	15 min
Volume Expected			140 mL	65 mL	50 mL	50 mL	50 mL
Total Elapsed Time			55 min	80 min	110 min	110 min	120 min