

Standard Operating Procedure

**TITLE: Extraction of PCBs from soils and sediments using Automated Soxhlet by Method 3541 for the Fox River**

**MATRICES**

This SOP pertains to the extraction of Solid Matrices.

**DETECTION LIMITS**

Reporting Limits (see Appendix A Table A) are used in the absence of Project or State Specific Required Detection Limits. The analytical method used to analyze samples extracted using this procedure contains the current method detection limits (MDL).

**1.0 SCOPE AND APPLICATION:**

1.1 Automated soxhlet is a procedure for isolating nonvolatile and semi-volatile organic compounds from solids such as sludges, soils, sediments and biological samples. The process ensures intimate contact of the sample with the extraction solvent.

**2.0 SUMMARY OF THE TEST METHOD:**

2.1 A measured mass of sample, typically 10 grams, is mixed with sodium sulfate until it is free flowing. The sample mixture is then transferred into a cellulose extraction thimble. The thimbles are then placed in extraction beakers and solvent is added. The beakers are then loaded into the automated soxhlet unit. The samples are then subjected to a pre-programmed heat and pressure extraction cycle which isolates the organic components from the sample mixture by contact with the solvent. The extract is concentrated to a final volume. Sample extracts may then be subject to necessary cleanups prior to analysis.

**3.0 DEFINITIONS:**

3.1 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the extraction and analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

3.2 Matrix Spike (MS) and Matrix Spike Duplicate (MSD): Aliquots of an environmental sample to which a known quantity of the analyte(s) of interest is added in the laboratory. The MS and MSD are analyzed exactly like a sample. Their purpose is to quantify the bias and precision caused by the sample matrix.

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- 3.3 Lab Control Spike (LCS) and Lab Control Spike Duplicate (LCSD): A method blank to which a known quantity of the analyte(s) of interest is added. The LCS, and LCSD when needed, is processed simultaneously with and under the same conditions as samples through all steps of the extraction and analytical procedures. Their purpose is to show complete extraction technique.
- 3.4 Surrogate: A substance with properties that mimic the analyte(s) of interest that is unlikely to be found in environment samples. Surrogate should be added to all samples, laboratory control spikes, matrix spikes, and method blanks and are used to monitor unusual matrix effects, sample processing problems, etc.

4.0 INTERFERENCES

- 4.1 En Chem purchases high quality solvents and reagents to minimize the potential of contamination from the solvents used in the extraction process. En Chem has SOPs addressing proper glassware cleaning techniques.
- 4.2 Solvents, reagents, glassware, and other sample processing equipment are demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.
- 4.3 Interferences co-extracted from samples will vary considerably depending on the source of the material. Contaminants which may interfere with the analysis of PCBs may be removed from the extracts using any combination of cleanups including, but not limited to column chromatography with Florisil (En Chem SOP SVO-57), the removal of sulfur using mercury (En Chem SOP 3-SVO-27) or copper (En Chem SOP K-SVO-78), or acid washing (En Chem SOP 3-SVO-28).

5.0 SAFETY:

- 5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means possible.
- 5.2 All department personnel shall be familiar with company safety policies as covered in the employee manual (section 5).
- 5.3 Due to the high volatility of hexane extreme pressure can be generated quickly; use caution when using hexane in closed containers.
- 5.4 Care should be taken not to use cracked or broken glassware.
- 5.5 MSDS sheets are kept in each department. They should be consulted whenever there is a question related to material handling and safety. Also, read all chemical labels for special handling requirements, precautions, and warnings.

6.0 EQUIPMENT AND SUPPLIES:

- 6.1 See Appendix A Tables B and C for a summary.

7.0 REAGENTS AND STANDARDS:

7.1 Where the purity of reagents and standards are not specified analytical reagent grade will be used. A grade no less than those specified by the references will not be used.

7.2 Laboratory Control and Matrix Spiking Solution:

**Example:** Dilute 50  $\mu$ L of the stock standard ( Restek Aroclor 1242 mix catalog number 32009 at 1000  $\mu$ g/ml ) to 5.0 mL with hexane. The concentration or Aroclor type may be adjusted if necessary for a particular project.

7.3 Surrogate Spiking Solution:

**Example:** Dilute 250  $\mu$ L of the stock standard surrogate (Restek pesticide surrogate mix catalog number 3200 at 200  $\mu$ g/ml), to 10 mL in a volumetric flask with acetone to prepare surrogate intermediate #1.

7.4 See Appendix A Table D for a summary of the reagents and standards.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE:

8.1 Not applicable.

9.0 QUALITY CONTROL:

9.1 All quality control measures shall be subjected to the same preparation procedures as those used on actual samples.

9.2 Method blanks shall be performed at a frequency of one per extraction batch, not to exceed 20 environmental samples. The method blank includes the sodium sulfate and any solvents or materials used for extracting the samples.

9.3 Laboratory control spikes (LCS) shall be performed at a frequency of one per extraction batch, not to exceed 20 environmental samples. A laboratory control spike consists of 10 grams of sodium sulfate or a background matrix fortified with about five times the reporting limit of an Aroclor of interest. The control spike is extracted and analyzed along with the samples. In the absence of mass available to analyze matrix spikes, a laboratory control spike duplicate (LCSD) may be analyzed to assess precision.

9.4 Matrix spikes (MS) and matrix spike duplicates (MSD) shall be performed at a frequency of one pair per extraction batch, not to exceed 20 environmental samples. These are field samples fortified with a known amount of one of the Aroclors of interest. If low matrix spike recoveries are found, and the LCS data is in control, the sample exhibits a matrix effect of low accuracy. If the matrix spike duplicate recoveries are similar to the matrix spike the matrix effect has been confirmed.

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- 9.5 Surrogate compounds are added to all samples and quality control samples. The compounds chosen mimic the properties of the targeted compounds but are not themselves found in environmental samples. Low recoveries can indicate a matrix effect that results in low bias. This is confirmed either by re-analysis of the sample or by observing similar recoveries of samples from the same project site. High recovery can indicate the presence of co-eluting compounds with similar properties as the surrogate. Surrogate recoveries in control spikes and method blanks confirm the efficacy of the method.
- 9.6 See Appendix A Table E for a summary.

## 10.0 PROCEDURE:

- 10.1 Verify sample field IDs and LIMS numbers on containers.
- 10.2 Lay out and record sample LIMS numbers as a batch with quality control measures at a frequency as described in Section 7 and summarized in Table E. Headings at the top of the page identify the sampling date, date of extraction, sample mass extracted, final volume of the sample extract, and a comments column concerning special circumstances regarding the sample.
- 10.3 Sediment samples are air-dried and ground with a mortar & pestle prior to extraction. Discard any foreign objects, such as sticks, rocks or leaves in soil samples.
- 10.4 Mix sample thoroughly, especially when compositing samples.
- 10.5 Weigh out a mass of sample as close to 10 grams as possible into a clean 250 mL beaker. Record the sample mass in the extraction logbook to the nearest tenth of a gram. Repeat process for all samples and quality control measures.
- 10.6 Add enough sodium sulfate to the sample in the beaker to create a dry free flowing mixture.
- 10.7 Transfer the mixture from the 250 ml beaker to a cellulose extraction thimble. Place the thimble in an automated soxhlet extraction beaker equipped with a wire thimble holder. Transfer all samples and quality control samples using the same process. Label each extraction beaker with the LIMs number or quality control measure identifier. **Retain the 250 ml beaker for solvent addition.**
- 10.8 Spike each thimble with surrogate solution. Apply directly to dried sample in thimble. **Example:** 500  $\mu$ L of a 2.0  $\mu$ g/mL surrogate solution intermediate #1.
- 10.9 Spike each laboratory control spike (LCS) and matrix spike (MS/MSD) with the appropriate amount of Aroclor spiking solution. **Example:** 100  $\mu$ L of the Aroclor 1242 spike solution.
- 10.10 Pour 140 mls of 4:1 hexane acetone into the retained 250 ml weighing beaker. Pour this solvent mixture into the cellulose thimble slowly. **Retain the beaker.**

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- 10.11 Push the wire thimble basket down into the extraction beaker, which will immerse the samples in solvent. Add teflon boiling chips, or copper chips (which will aid in sulfur removal), to each extraction beaker. Load the extraction beakers onto the automated soxhlet unit.
- 10.12 Verify the automated soxhlet extraction settings (program 02) as summarized in Table 3 below.

Table 3

Extraction temp	180 C
Boil time	45 min
Solvent Reduction	2 x 15 ml
Extraction Time	45 min
Cycle time	1 hour 38 minutes
Solvent	4:1 Hexane/ Acetone

- 10.13 Start the extraction process. Rotate the extraction beakers slightly to insure seal of top o-ring. The process will produce approximately 90 mL of extract.
- 10.14 Quantitatively transfer the extracts from the extraction beakers to the retained 250 ml weighing beakers.
- 10.15 Extract Concentration: This document mentions one method of concentrating the sample extracts. Equivalent extract concentration methods such as a Turbovap, Rotovap, or using a Kuderna-Danish apparatus, may be used. Transfer a portion of each extract from the 250 ml beakers to labeled Kontes tube ebullators. Place the tubes into a Kontes tube heater.
- 10.16 Set the heater dial 8. Place one stainless steel tube connected to the nitrogen regulator into each of the tubes. A high purity nitrogen stream sufficient to create continuous agitation is used to assist in the concentration process. As sample extracts concentrate add remaining solvent in portions. Rinse empty beaker with hexane and add to the concentrator tube.
- 10.17 Stop the concentration process when the extract is concentrated to approximately 6-7 mL. Remove the steel nitrogen tube and rinse it with a small portion of hexane as it is drawn out. Immediately remove the tube from the heater to avoid bumping. Swirl the tube to dissolve any oils stuck to the glass.
- 10.18 The tube and ground glass joints are rinsed with hexane.
- 10.19 After cooling, the extract is brought to a final volume of 10.0 mL.
- 10.20 Transfer each extract to a labeled amber screw top vial and seal with a teflon lined cap.

**NOTE: A variety of cleanups may be performed as determined necessary on the extracts prior to analysis. A column chromatography cleanup using Florisil (En Chem Method SVO-57) separates the Aroclors from most other typical environmental interference's. Soil and sediment samples typically need to have sulfur removed using elemental mercury (En Chem Method SVO-27), copper filings (En Chem**

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Method K-SVO-78) or by gel permeation chromatography (En Chem Method SVO-26). A sulfuric acid cleanup may be used, as required, to further remove contaminants (En Chem Method SVO-28). Then the extract is ready for analysis in accordance with K-SVO-77.

11.0 **CALCULATIONS:**

11.1 Not applicable.

12.0 **METHOD PERFORMANCE:**

12.1 Method performance is validated through the study of performance check and calibration standards and by the analysis of laboratory reagent blanks put through the copper cleanup method to meet quality control criteria for this procedure.

13.0 **POLLUTION PREVENTION:**

13.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff order, whenever possible, acceptable non-toxic alternative supplies. Staff also prepares only those quantities of reagents or standards that will be used prior to the expiration date. Any appropriate measures to minimize waste generation are brought to the attention of laboratory management.

14.0 **DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:**

14.1 Not applicable.

15.0 **CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA:**

15.1 Assessment of quality control measures provides a level of confidence in the data generated. The measures provide documentation that the instrument conditions were reliable during the analysis. Corrective actions are found in the determinative SOP.

16.0 **CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA:**

16.1 During analysis, events occur specific to the physical and chemical characteristics of the environmental sample. When possible, based on received sample volumes, data generated that do not meet statistical goals are re-analyzed to see if the statistical goal can be achieved. When environmental samples do not meet statistical goals, unacceptable data is generated. These events are different from those pertaining to instrument operating conditions and occur when the instruments are operating under ideal conditions.

17.0 **WASTE MANAGEMENT:**

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- 17.1 To minimize waste during sample preparation has two benefits. The first benefit is a cost savings to the lab in materials and supplies. The second is a benefit to the environment, as fewer materials need to be disposed.

**REFERENCES:**

40CFR Part 136

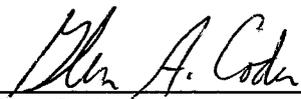
National Environmental Laboratory Accreditation Conference (NELAC), July 1998

Test Methods for Evaluating Solid Waste, Third Edition. SW846 Method 3541, Revision 0, September 1994.

MANAGEMENT APPROVAL AND REVIEW OF SOPS - POLICY AND DOCUMENTATION

APPROVED BY:   
Julie Trivedi  
Quality Assurance Officer

6/25/03  
Date

  
Glen Coder  
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6/25/03  
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Periodic Review Record

Review Date					
Initials					

**Appendix A**

**TABLES**

<b>Table A</b>	<b>Reporting Limits</b>
<b>Table B</b>	<b>Equipment</b>
<b>Table C</b>	<b>Supplies</b>
<b>Table D</b>	<b>Reagents</b>
<b>Table E</b>	<b>Quality Control</b>
<b>Table F</b>	<b>Initial Demonstration of Capability</b>

**Table A**  
**REPORTING LIMITS**

**PCB Solids**

Parameter	Reporting Limit
AROCLOR 1016	100 ug/kg
AROCLOR 1221	100 ug/kg
AROCLOR 1232	100 ug/kg
AROCLOR 1242	100 ug/kg
AROCLOR 1248	100 ug/kg
AROCLOR 1254	100 ug/kg
AROCLOR 1260	100 ug/kg

**Table B**  
**EQUIPMENT**

Equipment	Manufacturer	Model(s)	Serial #	Date In Service
Soxtherm	Gerhardt	SE 3A	491867	5/00
Soxtherm Controller	Gerhardt	SE VA	491862	5/00
Kontes heater tube blocks # 1	Kontes	NA	1776	
Kontes heater tube blocks # 2	Kontes	NA	1860	6/96
Kontes heater tube blocks # 3	Kontes	NA	2023	6/97

**Note: Equivalent equipment may be used.**

**Table C**  
**Supplies**

Supplies	Manufacturer	Vendor	Catalog #
evaporative column	Kontes	Fisher Scientific	569261
Kontes concentrator tube	Kontes	Fisher Scientific	570051-2526
heater block	Kontes	Fisher Scientific	720000-0000
ebullator	Kontes	Fisher Scientific	569401
Cellulose Extraction Thimbles	Whatman	Fisher Scientific	2800338
Extraction Beakers 54x130	Gerhardt	Visco Alpha	8400
Wire Thimble holders	Gerhardt	Visco Alpha	8323
Chemware PTFE boiling stones	Norton		A1069103
2 mL autosampler vials	VWR	VWR	66020-953
12 mL screw cap vials with caps		Fisher Scientific	03-391-7D
Aluminum crimp seals	VWR	VWR	66010-847

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Rubber pipette bulb	NJ Rubber	Fisher Scientific	R5002-2
Insert Rack for extraction beakers	Gerhardt	Visco Alpha	8466
N-Dex Nitrile Glove Powder free	Best	Fisher Scientific	7005L
Labtone glassware detergent	VWR	VWR	21851-006
Tube and bottle brush	VWR	VWR	PK-5-17041-002
5 3/4" disposable pipets	Fisher Scientific	Fisher Scientific	13-678-20-A
Amber jars 9 oz.	QEC	Fisher Scientific	2124-0009
25 mL adjustable repipetor	Brinkman	Fisher Scientific	
Lab notebooks	VWR	VWR	51280-108
250 mL beakers	Kimble	Fisher Scientific	14005-250
150 mL beakers	Kimble	Fisher Scientific	02539J
2.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	20-814-B
5.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	29623-300
25.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-A
50.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-B
25 uL gastight syringe	Hamilton	Fisher Scientific	24559
50 uL gastight syringe	Hamilton	Fisher Scientific	111111
100 uL gastight syringe	Hamilton	Fisher Scientific	13-684-100
250 uL gastight syringe	Hamilton	Fisher Scientific	81100
500 uL gastight syringe	Hamilton	Fisher Scientific	81217
1000 uL gastight syringe	Hamilton	Fisher Scientific	1482425
PCB 1016	Restek	Restek	32006
PCB 1221	Restek	Restek	32007
PCB 1232	Restek	Restek	32008
PCB 1242	Restek	Restek	32009
PCB 1248	Restek	Restek	32010
PCB 1254	Restek	Restek	32011
PCB 1260	Restek	Restek	32012
PCB surrogate (TCMX; DCB)	Restek	Restek	32000

Note: Equivalent equipment may be used.

Table D  
Reagents

Reagent	Purity	Manufacturer	Vendor	Catalog #
Nitrogen	99.99%	Whatman	----	----
Di-ionized Water	Type I ASTM	----	----	----
Sodium sulfate (anhydrous; 10-60 mesh)	ASC grade	Fisher Scientific	Fisher Scientific	MK802406
Acetone	Pesticide Quality	Burdick & Jackson	Fisher Scientific	010-4
Hexane	Pesticide Quality	Burdick & Jackson	Fisher Scientific	217-4

Note: Equivalent solvents may be used.

Table E

## QUALITY CONTROL

<b>Quality Control Measure</b>	<b>SW846 8082C</b>
<b>Initial Calibration</b>	Minimum of five levels; lowest level near but above MDL.
<b>Method Blank</b>	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
<b>Calibration Verification Standard (CVS)</b>	One at the beginning of a 12 hour time clock or more frequent.
<b>Independent Calibration Verification</b>	Independent source run immediately after the calibration curve.
<b>Laboratory Control Spike and Duplicate</b>	One LCS per batch of samples, up to 20 environmental samples, whichever is more frequent.
<b>Matrix Spike and Duplicate</b>	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
<b>Method Validation</b>	Annually
<b>MDL</b>	Annually
<b>Surrogate Standards</b>	Added to every sample.

Table F

En Chem Initial Demonstration of Capability

- PCB1 INSTRUMENT

Compound/Element	Acceptable Recovery from Method	En Chem Initial Demonstration Recovery
AROCLOR 1016 SOIL	70%-130%	112%
AROCLOR 1242 SOIL	70%-130%	
AROCLOR 1254 SOIL	70%-130%	
AROCLOR 1260 SOIL	70%-130%	114%