

STANDARD OPERATING PROCEDURE

For

USEPA METHOD 8082 + 3541 (soils), + 3510 (waters)

Determination of PCBs in Soils and Waters

SOP #: EPA8082

REVISION #: 1.2

DATE: December 2010

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LIST OF REVISIONS

Rev. #	Date	Description of Revision	Page #
0	November 2000	None	
1.0	December 2001	Reference <i>Table 1 Retention times of PCB Aroclors and surrogates</i> was made to the List of Tables, old Table 1 and Table 2 was moved to Table 2 and Table 3 respectively. Section 6.2.6 and section 6.2.15 were eliminated, Section 6.2.7 to section 6.2.14 were moved up. Section 6.6 Gas Chromatograph was rewritten. GC conditions have been updated. Section 6.7 and Section 6.8 were added. Section 7.4 to Section 7.12 were eliminated and rewritten as Section 7.4 to Section 7.10. Section 9.2, "PCNB" was deleted. Section 10.1, "WARNING: ..." and the %DDT Breakdown and %Endrin Breakdown equations were deleted. Section 10.2.1 to section 10.2.3 were rewritten. Old section 10.2.3 was moved to Section 10.2.4. Section 11.1, 11.2 and 11.3 were rewritten. Section 12.2, "The percent dry solids and calculated dry weight of extracted sample can be calculated from Equation 3 and 4." Was added. Section 12.2.1, the equation was rewritten. The caption "Equation 1" was added. "Where:" was added. " V(I) " was changed to " V(i) ". " V(t) = Volume of...(μL)" was changed to " V(t) = Volume of...(mL)". " V(s) = Volume of ...(mL)" was changed to " V(s) = Volume of ...(L)". Section 12.2.2, the equation was rewritten. The caption "Equation 2" was added. " Vt = Volume of...(μL)" was changed to " V(t) = Volume of...(mL)". " Vi = Volume of ...(μL)" was changed to " V(i) = Volume of ...(μL)". " Wt = Weight of ...(mg)" was changed to " W(t) = Weight of ...(mg)". Section 12.2.3, Equation 3 and Equation 4 were added Section 13.1, "...see Table 1" was changed to "...see Table 2 and Table 3." Section 16.0, Reference 16.2 and 16.3 were added. Section 17.0, New Table 1 <i>Retention times of PCB Aroclors and surrogates</i> was created and updated. Old Table 1 and Table 2 were moved to Table 2 and Table 3, respectively. Table 2 and Table 3 were updated for MDLs (08/14/01)	5 9 9 10 11 12 13-15 15 16 16 17
1.1	February 2003	Table 4 added	19
1.1	February 2007	Replaced Old DEP Logo with State Seal	
1.2	December 2010	Section 6.4 - Clarified type and use of water bath. Section 6.5 - Clarified type of analytical balance.	9 9



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Rev. #	Date	Description of Revision	Page #
		Section - 6.6 - Updated use of new computer and software.	9
		Section 9.5.1 - Added acceptance criteria of 70-130% for QC samples.	12
		Section 9.5.2 – eliminate	12
		Section 9.5.3 and 9.5.4 renumbered to 9.5.2 and 9.5.3	12
		Section 9.6.1 -Added acceptance criteria for matrix spikes at 65-135%.	12
		Section 10.1 -Typo fix.	12
		Section 11.1.6 - Clarified extraction step.	14
		Section 11.2.5 - Reduced Soxtec time from 2-3 hours to 1-2 hours.	14
		Section 11.2.9 -Clarified dry weight determination procedure.	14
		Section 11.3.1 - Changed water bath temperature.	14
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1.0 SCOPE AND APPLICATION

- 1.1 This is a gas chromatographic/ECD (GC/ECD) method applicable to the determination of PCB Aroclors in soils, sediments, and waters. The following Aroclors can be determined using this method.

ANALYTE:	CAS #
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

- 1.2 This method has been validated in a single laboratory and estimated detection limits (EDLs) have been determined for the analytes above. Observed detection limits may vary between waters, depending upon the nature of interferences in the sample matrix and the specific instrumentation used.
- 1.3 This method is restricted to use by or under the supervision of analysts experienced in the use of GC and in the interpretation of gas chromatograms.
- 1.4 Degradation of DDT and Endrin caused by active sites in the injection port and GC columns may occur. This is not as much a problem with new capillary columns as with packed columns. However, high boiling sample residue in capillary columns will create the same problem after injection of sample extracts.
- 1.5 When this method is used to analyze unfamiliar samples for any or all of the analytes above, analyte identifications must be confirmed by at least one additional qualitative technique.

2.0 SUMMARY OF METHOD

- 2.1 Using EPA Method 3510, a measured volume of sample of approximately 1 L is solvent extracted with methylene chloride by shaking in a separatory funnel or mechanical tumbling in a bottle. The methylene chloride extract is isolated, dried and concentrated to a volume of 10-mL after solvent substitution with Hexane. Using Method 3541 for soils, a 5-10 gram calculated dry weight sample is extracted with 50:50 hexane:acetone. The hexane:acetone extract is isolated, dried and concentrated to a volume of 10-mL with hexane. Chromatographic conditions are described which permit the separation and measurement of the analytes in the extract by capillary column/GC with an electron capture detector (ECD).

3.0 DEFINITIONS

- 3.1 Laboratory Duplicates (LD1 AND LD2) -- Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicate the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.



- 3.2 Field Duplicates (FD1 AND FD2) -- Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
- 3.3 Laboratory Reagent Blank (LRB) -- An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
- 3.4 Field Reagent Blank (FRB) -- An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.
- 3.5 Laboratory Fortified Blank (LFB) -- An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 3.6 Laboratory Fortified Sample Matrix (LFM) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.
- 3.7 Stock Standard Solution (SSS) -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial supplier.
- 3.8 Primary Dilution Standard Solution (PDS) -- A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.
- 3.9 Calibration Standard (CAL) -- A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 3.10 Quality Control Sample (QCS) -- A solution of method analytes of known concentrations which is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.
- 3.11 Method Detection Limit (MDL) -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.
- 3.12 External Standard (ES) -- A pure analyte(s) the concentration of which has been determined in an analysis separate from that used to measure the analyte(s) in the sample. The signal observed for a known quantity of the external standard(s) is used to calibrate the instrument response for the corresponding analytes(s). The instrument response is used to calculate the concentrations of the analyte(s) in the sample.



4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in liquid chromatograms. All reagents and apparatus must be routinely demonstrated to be free from interferences under the conditions of the analysis by analyzing laboratory reagent blanks as described in Sect. 9.0.

4.1.1 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

4.1.2 **WARNING:** When a solvent is purified, stabilizers added by the manufacturer are removed, thus potentially making the solvent hazardous. Removal of preservatives by distillation may also reduce the shelf-life of the solvent.

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 must be treated as a potential health hazard. Accordingly, exposure to these chemicals must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should also be made available to all personnel involved in the chemical analysis.

5.2 **Warning:** When a solvent is purified, stabilizers added by the manufacturer are removed, thus potentially making the solvent hazardous. Therefore, storage of large volumes of purified solvents may be hazardous. Therefore, only small volumes of solvents should be purified just before use.

6.0 EQUIPMENT, APPARATUS, AND SUPPLIES

6.1 Sample Containers

6.1.1 Waters - Bottles-Borosilicate, 1-L volume fitted with screw caps lined with TFE-fluorocarbon. Protect samples from light.

6.1.2 Soils - sample collection jars - 4-12 oz. jars with PTFE lined screw caps.

6.2 Glassware and Apparatus

6.2.1 Separatory funnel – 2000-mL, with TFE-fluorocarbon stopcock, ground glass or TFE-fluorocarbon stopper.

6.2.2 Flask, Erlenmeyer – 500-mL.

6.2.3 Concentrator tube, Kuderna-Danish (K-D) 10- or 25-mL, graduated (Kontes K-570050-1025 or K-570050-2525 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stoppers are used to prevent evaporation of extracts.

6.2.4 Evaporative flask, K-D 500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

6.2.5 Snyder column, K-D three-ball macro (Kontes K-503000-0121 or equivalent).



- 6.2.6 Vials - Glass, 5 to 10-mL capacity with TFE-fluorocarbon-lined screw cap.
- 6.2.7 Volumetric Flasks - glass - 10 and 100-mL.
- 6.2.8 Volumetric pipettes - various, glass.
- 6.2.9 Soxtec Extraction cups - glass, 100-mL capacity.
- 6.2.10 Whatman extraction Thimbles - (26 mm x 60 mm) for Tecator Soxtec Unit.
- 6.2.11 Soxtec Extraction Unit - Tecator 1044 unit for automated soxhlet extraction.
- 6.2.12 Glass funnels.
- 6.2.13 Chromatography Columns - 19 x 300 mm glass.
- 6.3 Boiling stones carborundum, #12 granules (Arthur H. Thomas Company #1590-033 or equivalent). Heat at 400° C for 30 minutes prior to use. Cool and store in a desiccator.
- 6.4 Water bath - Organomation Inc. 8-Station K-D capacity with solvent reclamation. Water bath set at 100° C and capable of temperature control ($\pm 2^{\circ}\text{C}$). The bath is contained in a fume hood.
- 6.5 Balance - Mettler-Toledo AB204 analytical balance. Balance is capable of accurately weighing to the nearest 0.0001g.
- 6.6 Gas Chromatograph - Varian CP-3800 GC with dual ECD detectors equipped with Varian 8200, 48-sample tray autosampler system. The Varian 3800 system is capable of temperature programming and is equipped with electronic pressure control. The data system is a Varian Star Workstation (Version 6.3) that is operated with Windows XP Professional and a Dell OptiPlex 990 computer workstation.
 - 6.6.1 Dual column analysis is conducted by using a borosilicate y-splitter.
 - 6.6.1.1 Column 1 (Primary column): DB-5, 60 m long x 0.25 mm ID bonded fused silica column, 0.25 μm film thickness.
 - 6.6.1.2 Column 2 (Confirmation column): DB-1701, 60 m long x 0.25 mm ID bonded fused silica column, 0.25 μm film thickness.
 - 6.6.2 Helium carrier gas flow is established at 38 cm/sec linear velocity and 95%Argon/5%methane is set at 25 mL/min as make-up gas.
 - 6.6.3 The oven temperature is programmed by holding the oven at 100°C for 1 min then ramping to 200°C at 15°C/min and held for 30 min. The oven is then programmed to 280°C at 5°C/min and holding for 10 min. Total run time is calculated to be 63.67 min. The injector temperature is set at 290 °C and both detector temperatures are held constant at 320°C. The injection volume is 5 μL split/splitless mode with a 1 min vent delay to achieve a 50:1 split ratio. After 10 min, a 5:1 split ratio is held constant to conserve carrier gas.
- 6.7 Detectors -- Electron capture detectors (2-Ni63 high activity).



- 6.8 Table 1 lists retention times observed for method analytes using the columns and analytical conditions described above.

7.0 REAGENTS AND STANDARDS

- 7.1 Acetone, methylene chloride, hexane - Pesticide grade or equivalent.
- 7.2 Sodium sulfate, granular, anhydrous, ACS grade. Heat treat in a shallow tray at 450° C for a minimum of 4 hours to remove interfering organic substances.
- 7.3 Sodium thiosulfate, granular, anhydrous, ACS grade.
- 7.4 Glass Wool—Pesticide grade or solvent washed with hexane.
- 7.5 Disposable Pasteur pipettes - Borosilicate glass.
- 7.6 Reagent Water – ASTM Type I reagent water.
- 7.7 Standard solutions may be purchased from EM Science or Ultra Scientific as certified solutions. Purchased stock standard solutions should be replaced after the indicated expiration date has surpassed or, sooner if comparison with laboratory fortified blanks or QC samples indicate a problem. Intermediate solutions should be replaced after six months or, sooner if comparison with instrument response factors indicate a problem.
- 7.7.1 PCB Aroclor (A1016, A1221, A1232, A1242, A1248, A1254, A1260), 100 µg/mL in methanol
- 7.7.2 Pentachloronitrobenzene (PCNB) solution, 100 µg/mL in MTBE for use as surrogate standard.
- 7.7.3 Pesticides surrogate standard spiking solution, 200 µg/mL in acetone (Decachlorobiphenyl (DCBP) and Tetrachloro-m-Xylene (TCMX))
- 7.8 Intermediate PCB Aroclor is individually prepared by pipetting each 1000 µL of each 100 µg/mL PCB Aroclor into a 10.00 mL volumetric flask partially filled with hexane. The intermediate is brought to the volume with hexane. The final concentration of the intermediate is 10.0 ng/µL. The solutions are transferred to PTFE lined screw cap vials. Store in refrigerator at 4° C and protect from light.
- 7.9 Surrogate standard spiking solution is prepared by accurately pipetting 500 µL of 200 µg/mL pesticides surrogate standard spiking solution, and 1000 µL of 100 µg/mL of PCNB solution into a 50.00 mL volumetric flask partially filled with MTBE. The solution is brought to the volume with MTBE. The final concentration is 2.0 ng/µL. Store the flask in the refrigerator at 4°C and protect from light.
- 7.10 Florisil - 60-200 mesh activated and stored at 105°C.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 Grab samples must be collected in glass containers.
- 8.2 Sample Preservation



8.2.1 Waters

- 8.2.1.1 If residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample to the sample bottle prior to collecting the sample.
- 8.2.1.2 After adding the sample to the bottle containing preservative(s), seal the sample bottle and shake vigorously for 1 minute.
- 8.2.1.3 Samples must be refrigerated at 4°C from the time of collection until extraction. Preservation study results indicate that most of the target analytes present in the samples are stable for 7 days when stored under these conditions.

8.2.2 Soils and Sediments

- 8.2.2.1 Collect 100 grams of representative sample in a glass jar following conventional sampling practices.
- 8.2.2.2 Collection of additional aliquots is recommended for laboratory QC.
- 8.2.2.3 Samples must be refrigerated at 4°C from time of collection to extraction. Preservation studies indicate that most of the target analytes present in the samples are stable for 14 days when stored under these conditions.

8.3 Extract Storage

- 8.3.1 Sample extracts should be stored at 4°C away from light. A 14-day maximum extract storage time is recommended. A 14-day holding time is established for waters, soils, sediments and solid matrices.

9.0 QUALITY CONTROL

- 9.1 Minimum quality control (QC) requirements are initial demonstration of laboratory capability, determination of surrogate compound recoveries in each sample and blank, monitoring internal standard peak area, analysis of laboratory reagent blanks, laboratory fortified samples, laboratory fortified blanks, and QC samples.
- 9.2 Laboratory Reagent Blank - All extraction sets include a laboratory reagent blank consisting of Type I water or contaminant-free sand and surrogate.
- 9.3 The analyst is permitted to modify GC columns, GC conditions, GC detectors, continuous extraction techniques, concentration techniques (i.e. evaporation techniques), internal standards or surrogate compounds. Each time such method modifications are made, the analyst must repeat the procedures for MDL determinations.
- 9.4 Assessing Surrogate Recovery
 - 9.4.1 When surrogate recovery from a sample or method blank is <70% or >130%, check: (1) calculations to locate possible errors, (2) fortifying solutions for degradation, (3) contamination or other obvious abnormalities, and (4) instrument performance. If those steps do not reveal the cause of the problem, reanalyze the extract.
 - 9.4.2 If a blank extract reanalysis fails the 70-130% recovery criterion, the problem must be identified and corrected before continuing.



- 9.4.3 If sample extract reanalysis meets the surrogate recovery criterion, report only data for the reanalyzed extract. If sample extract reanalysis continues to fail the surrogate recovery criterion, report all data for that sample as suspect.

9.5 Assessing Laboratory Performance – Laboratory Fortified Blanks

- 9.5.1 The laboratory must analyze at least one laboratory fortified blank (LFB) sample with every twenty samples or one per sample set (all samples extracted within a 24-hour period) whichever is greater. The fortified concentration of each analyte in the LFB should be 10 times EDL or the MCL whichever is less. Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the control limits, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses. The laboratory has established acceptance criteria of 70-130% for the laboratory fortified blanks.

- 9.5.2 The laboratory periodically determines its detection limit capabilities for the analytes of interest.

- 9.5.3 The laboratory analyzes a QC sample purchased from Ultra Scientific or EM Science at least 10% of samples.

9.6 Assessing Method Performance – Laboratory Fortified Sample Matrix

- 9.6.1 The laboratory adds a known concentration to a minimum of 10% of the routine samples or one sample concentration per set whichever is greater. The laboratory has established acceptance criteria of 65-135% for the laboratory fortified matrix sample.

- 9.7 The laboratory may adopt additional quality control practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. For example, field or laboratory duplicates may be analyzed to assess the precision of the environmental measurements or field reagent blanks may be used to assess contamination of samples under site conditions, transportation, and storage.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Establish GC operating parameters equivalent to those indicated in Sec 6.6. The GC system must be calibrated using the external standard technique.

10.2 External Standard Calibration Procedure

- 10.2.1 Prepare calibration standards at a minimum of three concentration levels for each PCB Aroclor from intermediate solution and dilute with hexane to the volume in 10 mL volumetric flasks. The concentration levels of PCB Aroclor are 0.005, 0.02, 0.05, 0.20, 0.50 ng/ μ L. The lowest standard should represent analyte concentrations near, but above their respective MDLs. The other concentrations should correspond to the range of concentrations expected in the sample concentrates, or should define the working range of the detector. The calibration standards must bracket the analyte concentrations found in the sample extracts.

- 10.2.2 Calibration standards of pesticides mixture may be used for calibration standards of surrogate compounds (TCMX, PCNB and DCBP). (See EPA Method 508 (ref. 16.2) or EPA Method 608 (ref. 16.3).



- 10.2.3 Starting with the standard of lowest concentration, analyze each calibration standard and tabulate response (peak area) versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (20% RSD or less), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.
- 10.2.4 The working calibration curve or calibration factor is verified on each working day by the measurement of a minimum of two calibration check standards, one at the beginning and one at the end of the analysis day. These check standards should be at two different concentration levels to verify the calibration curve. For extended periods of analysis (greater than 8 hrs.), it is strongly recommended that check standards be interspersed with samples at regular intervals during the course of the analyses. If the response for any analyte varies from the predicted response by more than $\pm 20\%$, the test must be repeated using a fresh calibration standard. If the results still do not agree, generate a new calibration curve.

11.0 PROCEDURE

11.1 Extraction (Method 3510 - Waters)

- 11.1.1 Measure volume of sample to 1 L with graduated cylinder. Record the sample volume to the nearest 5 mL. Pour the entire sample into a 2-L separatory funnel. Fortify the sample with 50 μL of the 2.0 ng/ μL surrogate standard solution.
- 11.1.2 Add 60-mL methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner walls. Transfer the solvent to the separatory funnel and extract the sample by vigorously shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.
- 11.1.3 Assemble a K-D concentrator by attaching a 10-mL K-D concentrator tube to a 500-mL K-D evaporative flask with spring or clip. Add 1 to 2 clean boiling stones to the K-D unit.
- 11.1.4 Prepare a glass funnel plugged with glass wool at the bottom and filled with about 20-g anhydrous sodium sulfate.
- 11.1.5 Pre-wet the sodium sulfate with methylene chloride and carefully pass the extract (bottom layer) through the sodium sulfate funnel. Collect the extract in the K-D concentrator unit.
- 11.1.6 Repeat step 11.1.2 two more times. Combine all the extracts in the K-D unit.
- 11.1.7 Rinse the sodium sulfate with about 20-30 mL methylene chloride and combine in the K-D concentrator unit.

11.2 Extraction (Method 3541 - Soils)

- 11.2.1 Decant and discard any water layer on a sediment sample. Mix sample thoroughly. Weigh 5-10 grams of sample.



- 11.2.2 Place the sample in a Whatman extraction thimble and onto Soxtec Unit.
- 11.2.3 Place boiling chips into Soxtec extraction cups and add 70-mL of 50:50 acetone:hexane.
- 11.2.4 Fortify the sample with 50 μ L of the 2.0 ng/ μ L surrogate spiking solution.
- 11.2.5 Place Soxtec cups onto respective Soxtec slot and heat gently for 1-2 hours. Add additional solvent to the top of the unit if evaporation is encountered.
- 11.2.6 Assemble a K-D concentrator by attaching a 10-mL K-D concentrator tube to a 500-mL K-D evaporative flask with spring or clip. Add 1 to 2 clean boiling stones to the K-D unit.
- 11.2.7 Prepare a glass funnel plugged with glass wool at the bottom and filled with about 20-g anhydrous sodium sulfate. Place the funnel on top of the K-D concentrator unit.
- 11.2.8 Pre-wet the sodium sulfate with hexane and carefully pass the extract through the sodium sulfate funnel. Rinse the extraction cup with hexane for a couple of times and pass through the sodium sulfate. Collect all the extract in the K-D concentrator unit.
- 11.2.9 Separately, place an aliquot of sample in a tared aluminum pan to determine the percent moisture of the sample and calculate results as percent dry solids. The sample is placed in a drying oven overnight at 105°C. After drying, the sample is allowed to cool at room temperature. Reweigh aluminum pan containing the dried sample to determine the moisture content.
- 11.3 Extract concentration and solvent exchange
- 11.3.1 Attach a 3-ball Snyder column. Pre-wet the Snyder column by adding about 1-mL methylene chloride (for waters) or hexane (for soils) to the top. Place the K-D apparatus on a hot water bath, 100°C, so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. When the apparent volume of liquid reaches 2-mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.
- For soils, Prepare a chromatography column plugged with glass wool and filled with 20 \pm 1 grams of florisil. Pre-elute with hexane. Carefully add the extract to the top of the Florisil column. Keep the K-D concentrator unit in place under the corresponding Florisil column. Let the extract travel down the column. Elute the Florisil column with two portions of 50 mL 6% diethyl ether/94% hexane. Collect the eluate in the K-D apparatus. Concentrate on steam bath. When the volume of liquid reaches 2 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min. Remove the Snyder column. Thoroughly wipe the water around the joint between K-D concentrator tube and evaporative flask, and carefully remove the joint. Adjust the final volume to 10.00 ml with hexane. If further concentration is needed, use an air-blow down apparatus or TurboVap
- For waters, remove the Snyder column and add 50 mL of hexane. Re-attach the Snyder column to the flask and pre-wet the column by adding about 0.5-mL of hexane to the top. Place K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. When the apparent volume of liquid reaches 2-mL, remove the micro K-D from the bath and allow it to drain and cool. Adjust the final volume to 10.00-mL with hexane. If further concentration is needed, use an air-blow down apparatus.



11.3.2 Transfer extract to an appropriate-sized TFE-fluorocarbon sealed screw-cap vial and store, refrigerate at 4°C, until analysis by GC-ECD.

11.4 Gas Chromatography

11.4.1 Section 6.6 summarizes the recommended operating conditions for the gas chromatograph. Other GC columns, chromatographic conditions, or detectors may be used if the requirements of this method are met.

11.4.2 Calibrate the system daily by running calibration standards

11.4.3 Inject 5.0 µL for PCB Aroclor analysis. Record the resulting peak size for 3 peaks of the Aroclor in area units.

11.4.4 If the response for the peaks exceeds the working range of the system, dilute the extract and reanalyze.

11.5 Identification of Analytes

11.5.1 If applicable, the analyst can use the Webb and McCall^{16.4} procedure to identify Aroclor mixtures. Identify a sample component by comparison of its retention time to the retention time of a reference chromatogram. If the retention time of an unknown compound corresponds, within limits, to the retention time of a standard compound, then identification is considered positive. For PCB Aroclor analysis, it is customary to choose 3-5 peaks of the Aroclor for quantitation.

11.5.2 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for a compound. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Calculate analyte concentrations in the sample from the response for the analyte using the calibration procedure described in the external calibration procedure.

12.2 In the external standard calibration procedure, determination of the amount of material injected is made by the peak response of the known versus the unknown. The concentration (C) in the sample can be calculated from Equation 1 and 2. The percent dry solids and calculated dry weight of extracted sample can be calculated from Equation 3 and 4.

12.2.1 Waters

$$C (\mu\text{g/L}) = \frac{A \bullet V(t)}{V(i) \bullet V(s)} \quad \text{Equation 1}$$

Where:

A = Amount of material injected (ng)

V(i) = Volume of extract injected (µL)



$V(t)$ = Volume of total extract (mL)
 $V(s)$ = Volume of water extracted (L)

12.2.2 Soils

$$C (\mu\text{g/g}) = \frac{A \bullet V(t)}{V(i) \bullet W(t)} \quad \text{Equation 2}$$

Where:

A = Amount of material (ng)
 $V(t)$ = Volume of total extract (mL)
 $V(i)$ = Volume of injected extract (μL)
 $W(t)$ = Weight of calculated dry sample (g)

12.2.3 Percent dry solids and calculated dry weight of extracted sample:

$$\% \text{Dry Solids} = \frac{\text{Dry weight of Separate Sample Aliquot (g)}}{\text{Wet Weight of Separate Sample Aliquot (g)}} \times 100 \quad \text{Equation 3}$$

$$\text{Calculated Dry Weight of Extracted Sample (g)} = \text{Weight of Extracted Sample} \times \% \text{Dry Solids} \quad \text{Equation 4}$$

13.0 METHOD PERFORMANCE

13.1 For precision and accuracy data, see Table 2 and Table 3.

14.0 POLLUTION PREVENTION

14.1 This method uses hexane, acetone, and methylene chloride as the extraction solvents. Waste solvents are discarded in waste containers. Once the waste containers reach capacity, they are transferred to a hazardous waste drum. The hazardous waste drum is transported off the premises by an environmental service company to an incinerator.

15.0 WASTE MANAGEMENT

15.1 The laboratory has implemented a chemical inventory database to track purchases of solvents and waste generated by use of the chemicals. The laboratory maintains a hazardous waste storage room for all waste solvents and samples. For further information on waste management, consult "The Waste Management Manual for Laboratory Personnel". The manual is available from the American Chemical Society.

16.0 REFERENCES

16.1 RCRA Methods 8082, 3541 and 3510 are from SW-846, 3rd edition, 1995, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods."



16.2 US EPA Method 508

16.3 US EPA Method 608

16.4 Webb, R.G. and McCall, A.C. "Quantitative PCB Standards for Election Capture Gas Chromatography," *Journal of Chromatographic Science*, 11.366 (1973).



17.0 TABLES, AND VALIDATION DATA

TABLE 1. Retention Times of PCB Aroclors and Surrogates (08/14/2001)

Aroclor	Retention Time (min) ^(a)	
	Primary	Confirmation
PCB A1232	11.55, 11.88, 12.09	NR
PCB A1242	13.69, 13.80, 14.57	NR
PCB A1248	18.11, 18.44	NR
PCB A1254	29.97, 31.49	NR
PCB A1260	51.10, 51.40	NR
TCMX	10.94	11.26
PCNB	13.57	15.68
DCBP	57.61	58.71
(a) Columns and analytical conditions are described in Sect. 6.6 NR = Data not available		

TABLE 2. Accuracy, Precision and Method Detection Limits (MDL) for RCRA Method 8082 and 3541 in Soils and Sediments

Analyte	Date of Study	No. of Samples Spiked (n)	Spiked Conc. (µg/g)	Accuracy (Mean % Recovery) ^a	Precision (SD ^b in µg/g)	MDL (µg/g)
PCB A1232	08/14/01	7	0.040	77	0.0084	0.026
PCB A1242	08/14/01	7	0.040	67	0.0017	0.0052
PCB A1248	08/14/01	7	0.040	93	0.0037	0.012
PCB A1254	08/14/01	7	0.040	85	0.0034	0.011
PCB A1260	08/14/01	7	0.040	103	0.0013	0.0042
^a Recovery of spiked concentration ^b SD = standard deviation of mean concentration measured						



TABLE 3. Accuracy, Precision and Method Detection Limits (MDL) for RCRA Method 8082 and 3510 in Waters and Wastewaters

Analyte	Date of Study	No. of Samples Spiked (n)	Spiked Conc. (µg/L)	Accuracy (Mean % Recovery) ^a	Precision (SD ^b in µg/L)	MDL (µg/L)
PCB A1232	08/14/01	7	0.50	96	0.025	0.078
PCB A1242	08/14/01	7	0.50	76	0.019	0.058
PCB A1248	08/14/01	7	0.20	72	0.010	0.030
PCB A1254	08/14/01	7	0.50	102	0.030	0.10
PCB A1260	08/14/01	7	0.50	95	0.037	0.12
^a Recovery of spiked concentration						
^b SD = standard deviation of mean concentration measured						



Table 4 Quality Control Elements and Acceptance Limits for the Determination of PCBs in Soils and Waters: EPA Method 8082 + 3541 (soils), + 3510 (waters).

QC Elements	Frequency	Acceptance Criteria	Corrective Action
Sample storage	Upon sample arrival and before extraction	Refrigerated at 4°C, 7 days for waters and 14 days for soils	Flag sample and run it or contact sample collectors to obtain new samples
Extract storage	After extraction and before analysis	Refrigerated at 4°C, 14 days	Flag sample and run it or contact sample collectors to obtain new samples
Laboratory Extraction Blank	Every sample batch	No presence of target analytes or concentration of contaminated analytes < MDL	Re-extract and reanalyze the sample(s). If not enough sample(s), recollect, re-extract and reanalyze the sample(s).
Laboratory Fortified Blank	Every sample batch	70 - 130% recovery	Reanalyze sample set
Laboratory Fortified Matrix	At 10% of the routine samples	65-135 % recovery	Result is suspect due to matrix effect.
Field blanks	If available.	No presence of target analytes or concentration of contaminated analytes < MDL	Contact collector.
Solvent check	Run at the beginning of sample sequence	No presence of contaminated peak(s).	Replace solvent and rerun.
Initial Calibration	Run at the beginning of sample sequence or when response of any analyte in calibration check solution varies from predicted response by more than 20%	Correlation coefficient (R) > 0.99 for all target analytes	Recalibrate before continue.
Continuing Calibration	Run at the beginning of sample sequence, include within sample set at every 15-20 samples during the course of the analyses and run at the end of the sample sequence.	Response of any analyte in calibration check solution varies from predicted response < 20%	Recalibrate and reanalyze.
MDL	Annually	< MCL	Redo MDL
Quality Control Sample	Quarterly		
Surrogate (PCNB)	Every sample including QC samples	70 – 130% Recovery	Reanalyze sample set.