



Designation: D8543 – 24

Standard Test Method for Determination of Pesticides and Polychlorinated Biphenyls (PCBs) in Aqueous Solution by a Tandem Gas Chromatography/Mass Spectrometry/Mass Spectrometry (GC/MS/MS)¹

This standard is issued under the fixed designation D8543; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the identification and simultaneous measurement of extractable chlorinated pesticides and polychlorinated biphenyls (PCBs) by gas chromatography/mass spectrometry/mass spectrometry (GC/MS/MS).

1.2 This test method has been validated for wastewater influents, effluents, industrial discharges, surface water, and ground water.

1.3 This test method is not limited to these particular aqueous matrices; however, the applicability of this test method to other aqueous matrices shall be demonstrated.

1.4 This test method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph with tandem mass spectrometry. Each laboratory that uses this test method shall demonstrate the ability to generate results that meet or exceed the performance criteria of this test method.

1.5 If sensitivity permits, compound tentative identification of unknowns may be made by analyzing the extract in full-scan mode or, if the system allows simultaneous timed single-reaction monitoring (SRM)/full-scan acquisition. Identify unknown peaks according to Guide [D4128](#).

1.6 This test method is performance-based. Minor modifications, as allowed by CFR 40 Part 136.6, may be made to improve the method performance, but changes may not be made to the extraction, the extraction solvent, sample-to-solvent ratio, or the MS/MS detection technique.

1.7 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

- [D1129 Terminology Relating to Water](#)
- [D1193 Specification for Reagent Water](#)
- [D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water](#)
- [D3370 Practices for Sampling Water from Flowing Process Streams](#)
- [D4128 Guide for Identification and Quantitation of Organic Compounds in Water by Combined Gas Chromatography and Electron Impact Mass Spectrometry](#)
- [D4448 Guide for Sampling Ground-Water Monitoring Wells](#)
- [D6089 Guide for Documenting a Groundwater Sampling Event](#)
- [D6538 Guide for Sampling Wastewater With Automatic Samplers](#)
- [D6759 Practice for Sampling Liquids Using Grab and Discrete Depth Samplers](#)
- [E355 Practice for Gas Chromatography Terms and Relationships](#)
- [E2857 Guide for Validating Analytical Methods](#)

¹ This test method is under the jurisdiction of ASTM Committee [D19](#) on Water and is the direct responsibility of Subcommittee [D19.06](#) on Methods for Analysis for Organic Substances in Water.

Current edition approved May 1, 2024. Published June 2024. Originally approved in 2023. Last previous edition approved in 2023 as D8543 – 23. DOI: 10.1520/D8543-24.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

2.2 EPA Standards:³

EPA Method 625.1 Base Neutrals and Acids by GC/MS

EPA Method 608.3 Organochlorine pesticides and PCBs by GC/HSD

EPA Method 3534A (SW-846) Solid-Phase Extraction (SPE)

EPA Method 8082A Polychlorinated Biphenyls and PCBs by Gas Chromatography

2.3 Other Standards:

ISO Guide 34 General requirements for the competence of reference material producers⁴

40 CFR Part 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants⁵

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129 and Practice E355.

4. Summary of Test Method

4.1 A measured volume of sample is extracted with methylene chloride using a separatory funnel, continuous liquid-liquid extraction, or solid-phase extraction equipment. The extract is dried, concentrated, and injected into a capillary gas chromatography (GC) column interfaced to a tandem mass spectrometer (MS/MS). The GC column is temperature programmed to separate the test method analytes, which are detected with the MS/MS. Compounds eluting from the GC column are identified by comparing their MRM transition and retention times to reference standards. MRM transitions and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for the samples. The concentration of each identified component is measured by relating the SRM response of the SRM produced by that compound to the SRM transition response produced by a compound that is used as an internal standard. Surrogate analytes, whose concentrations are known in every sample, are measured with the same internal standard calibration procedure. Qualitative identity is made by measuring the relative abundance ratios of MRMs from the same compound and comparing it to the relevant standard. Quantitative analysis is performed by using the authentic standard(s) to produce a response factor and calibration curve. The calibration data, along with the sample volume extracted and the final sample extract volume, are used to determine the concentration of a target compound in the sample extract.

4.2 This test method is based on EPA Methods 608 and 8082A that determine organochlorine pesticides and polychlorinated biphenyls (PCBs), respectively, in water samples after liquid/liquid or solid-phase extraction using GC with a halogen-specific detector, such as an electron capture detector (ECD).

³ Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

⁵ Available from www.govinfo.gov.

5. Significance and Use

5.1 Pesticides and PCBs are Environmental Protection Agency (EPA)-regulated contaminants in treated drinking water, wastewater, and ground water. Liquid-liquid and solid-phase extraction (SPE) are generally applicable procedures for extracting these target analytes before GC/MS/MS analysis.

5.2 This test method is applicable to pesticides that are extracted from aqueous solution using methylene chloride and can be chromatographed and detected using tandem mass spectrometry procedures. In Table 1, pesticides are listed and in Table 2, PCBs validated by this test method are listed. This test method is not limited to the compounds listed in Table 1 or Table 2; however, the applicability of the test method to other compounds shall be demonstrated. Refer to Guide E2857 for guidance in validating the method for additional parameters.

5.3 Analyte concentrations up to approximately 250 ng/L may be determined. Higher concentrations may be determined by dilution of the extract. Analytes that are inefficiently extracted from water may not be detected when present at low concentrations.

5.4 Analytes that are not separated chromatographically but that have different transitions can be identified and quantitatively measured.

5.5 This test method may be used to determine the concentrations of Aroclor⁶ mixtures or PCB congeners or both present in the sample.

⁶ From 1929 to 1976, PCBs were produced and commercially marketed in the United States under the Aroclor trademark by Monsanto Chemical Corporation.

TABLE 1 Pesticides

Parameter	CAS No.
Aldrin ^A	309-00-2
α-BHC ^A	319-84-6
β-BHC ^A	319-85-7
δ-BHC ^A	319-86-8
γ-BHC (Lindane) ^A	58-89-9
Cis-chlordane (alpha) ^A	5103-71-9
Trans-chlordane (gamma) ^A	5103-74-2
2,4'-DDD	53-19-0
2,4'-DDE	3424-82-6
2,4'-DDT	789-02-6
4,4'-DDD ^A	72-54-8
4,4'-DDE ^A	72-55-9
4,4'-DDT ^A	50-29-3
Dieldrin ^A	60-57-1
Endosulfan I ^A	959-98-8
Endosulfan II ^A	33212-65-9
Endosulfan sulfate ^A	1031-07-8
Endrin ^A	72-20-8
Endrin Aldehyde ^A	7421-93-4
Endrin Ketone	53494-70-5
Heptachlor ^A	76-44-8
Cis-Heptachlor epoxide ^B	1024-57-3
Trans-Heptachlor epoxide ^B	28044-83-9
Mirex	2385-85-5
Pentachlorobenzene ^C	608.93-5
Hexachlorobenzene ^B	118-74-1

^A Priority pollutant pesticide listed in Table 1 of Method 608.3.

^B Method 608.3 lists this analyte as heptachlor epoxide, CAS # 1024-57-3.

^C Priority pollutant pesticide listed in Table 1 of Method 608.3.

TABLE 2 PCBs (as Aroclors)

Parameter	CAS No.
PCB-1016 ^A	12674-11-2
PCB-1221 ^A	11104-28-2
PCB-1232 ^A	11141-16-5
PCB-1242 ^A	53469-21-9
PCB-1248 ^A	12672-29-6
PCB-1254 ^A	11097-69-1
PCB-1260 ^A	11096-82-5
Chlorobiphenyl	
Dichlorobiphenyl	
Trichlorobiphenyl	
Tetrachlorobiphenyl	
Pentachlorobiphenyl	
Hexachlorobiphenyl	
Heptachlorobiphenyl	
Octachlorobiphenyl	
Nonachlorobiphenyl	

^A Priority pollutant Aroclor listed in Table 2 of Method 608.3.

See [Appendix X1](#) and [Appendix X2](#) for suggested transitions and collisional energies for all 209 congeners. Separation of all 209 congeners may not be possible and may require additional GC columns and operating conditions. Analysis of all congeners is not expected to be achieved by this test method.

5.6 Method detection limits (MDL) and minimum reporting level (MRL) for analytes in [Tables 1 and 2](#) are given in [Table 3](#). The required MDLs and MRLs are identical to those published in Method 608.3.

6. Interferences

6.1 Glassware shall be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and reagent water. The glassware should then be drained dry and heated in a muffle furnace at 400 °C for 15 min to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

6.2 Matrix interferences may be caused by contaminants with similar transitions that are co-extracted from the sample. Use of additional transitions may be useful in overcoming these interferences.

6.3 Variable amounts of pesticides and PCBs adhere to glass surfaces. Sample transfers should be minimized.

7. Apparatus

7.1 *Separatory Funnel*, size appropriate to hold the sample volume and extraction solvent (typically 2000 mL) with fluoropolymer stopcock.

7.2 *Drying Column*—Chromatography column with a coarse frit or equivalent, sufficient to hold 30 g of anhydrous sodium sulfate. Smaller drying columns with, or membrane-

TABLE 3 Method Detection Limits

Compound	Single Lab MDL, ng/L	Method Required MDL, ng/L	Method Required MRL, ng/L
Pentachlorobenzene	0.2		
Alpha-BHC ^A	0.2	3	9
Hexachlorobenzene ^B	0.4		
Gamma-BHC ^A	0.2	4	12
Beta-BHC ^A	0.3	6	18
Delta-BHC ^A	0.2	9	27
Heptachlor ^A	0.3	3	9
Aldrin ^A	0.5	4	12
cis-Heptachlor Epoxide ^A	3	83	249
Oxychlordan	3		
trans-Heptachlor Epoxide	2	83	129
cis-Chlordane (alpha) ^A	0.3	14	42
2,4'-DDE	0.3		
Endosulfan I ^A	6	14	42
trans-Chlordane (gamma) ^A	0.5	14	42
trans-Nonachlor	2		
4,4'-DDE ^A	0.3	4	12
Dieldrin ^A	3	2	6
2,4'-DDD	0.6		
Endrin ^A	5	6	18
Endosulfan II ^A	3	4	12
4,4'-DDD	0.4	5	15
cis-Nonachlor	2		
2,4'-DDT	0.8		
Endrin Aldehyde ^A	0.5	23	70
Endosulfan Sulfate ^A	0.3	7	21
4,4'-DDT ^A	0.3	11	33
Endrin Ketone	Not determined		
Mirex	0.5		
PCB-1016 ^B			
PCB-1221 ^B			
PCB-1232 ^B			
PCB-1242 ^B		65	95
PCB-1248 ^B			
PCB-1254 ^B			
PCB-1260 ^B			
Toxaphene ^B		240	720
Hexachlorocyclopentadiene ^B			

^A Priority pollutant listed in Table 1 of EPA Method 608.3.

^B Priority pollutant listed in Table 2 of EPA Method 608.3.

based drying tubes without, sodium sulfate may be used provided the quality control criteria of this test method is met.

7.3 *Concentrator Tube, Kuderna Danish*, graduated 10 mL. Verify calibration volumes by weighing known mass of reagent water (1 g/mL). Discard tubes that do not agree within 2 % of the stated volume. A ground glass or fluoropolymer stopper is used to prevent evaporation.

NOTE 1—Other sample concentration techniques, such as rotary or automated concentrators, may be used. The use of concentrators that recover solvent and automatically concentrate sample to 1 mL is highly encouraged. Follow the manufacturer's instructions.

7.4 *Evaporative Flask, Kuderna Danish*—Attaches to concentrator tube with springs, 500 mL.

7.5 *Snyder Column, Kuderna Danish*, three ball.

7.6 *Continuous Liquid/Liquid Extractor*, equipped with fluoropolymer or glass connection joints requiring no lubrication.

7.6.1 Continuous liquid/liquid extraction is useful for samples containing up to 1 % solids that cause emulsions. This technique is more efficient than separatory funnel extraction.

but the glassware is more expensive, and the overall extraction process is more time-consuming.

7.7 Solid-Phase Extraction (SPE) Apparatus, suitable for extraction of up to 1000 mL of sample.

7.7.1 Filter, apparatus or multiple-position manifold.

7.7.2 Vacuum System, capable of achieving 0.1 bar Hg equipped with a shutoff valve and a vacuum gauge.

7.7.3 Vacuum Trap, made from a 500 mL to 1000 mL sidearm flask with a single-hole rubber stopper and glass tubing, or equivalent.

7.8 Nitrogen Evaporation Device, equipped with a heated bath that can be maintained at an appropriate temperature for the solvent and analytes.

7.9 GC/MS/MS with Data Acquisition System:

7.9.1 The GC shall be capable of temperature programming and should be equipped with variable-constant differential flow controllers so that the column flow rate or linear velocity will remain constant throughout temperature program operation.

7.9.1.1 Data for this test method was collected using helium as a carrier gas. If another gas is used, analytical conditions may need to be adjusted. Follow the manufacturer's instructions for safety precautions regarding the use of hydrogen as a carrier gas.

7.9.2 Capillary GC Columns—Any GC column that meets the performance specifications of this test method may be used. Separations of the calibration mixture shall be equivalent or better than those described in this test method. As examples, the following columns have been found to be suitable:

7.9.2.1 Column, 30 m by 0.25 mm, 5 % phenyl methylpolysiloxane-fused silica capillary with a 0.25 µm film thickness, or equivalent.

7.9.2.2 Other columns, such as those manufactured specifically for the analysis of pesticides, may be used.

7.9.3 Injection Port—Split, splitless, temperature programmable, solvent purge, back flushed, or other, suitable for injecting highly reproducible sample aliquots. An autosampler is highly recommended.

7.9.4 The mass spectrometer shall be capable of electron ionization at a nominal electron energy of 70 eV. The spectrometer shall be capable of acquiring and storing data for up to four MRMs per targeted analyte with dwell times sufficient to measure a minimum of ten or more data points during the elution of each GC peak.

NOTE 2—Other electron ionization energies or modes of ionization may be used, provided the quality control (QC) acceptance criteria of this test method is met.

7.9.4.1 GC/MS/MS Interface—Any interface that meets the requirements in this test method may be used.

7.9.5 A data system is required to acquire, store, reduce, and output mass spectral data. The computer software should have the capability of processing stored GC/MS/MS data by recognizing a GC peak within any given retention time window, comparing the MRM transition from the GC peak with MRM transition in a user-created database, and generating a list of identified compounds with their retention times. The software shall allow integration of the ion abundance of any specific transition between specified time limits. The software should

also allow calculation of response factors as defined in 11.2.3, linear regression; weighted least squares (WLS), also known as weighted linear regression; % relative standard error (RSE) (see 40 CFR 136.6); construction of a second order ($k=2$) polynomial forms a quadratic expression (parabolic curve); calculation of response factor statistics; and calculation of concentrations of analytes using either the calibration curve or Eq 5.

8. Reagents and Materials

8.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁷ Other grades may be used, provided it is pure enough to be used without lessening the accuracy of the determination.

8.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Types I or II.

8.3 SPE Sorbent—Non-polar C18 octadecyl, 10 g/60 mL, used for solid-phase extraction. Other media may be used, provided the QC performance criteria of this test method are met.

8.3.1 Each sorbent lot should be certified by the laboratory or commercial supplier to ensure recovery of the analytes of interest and removal of 2,4,6-trichlorophenol. Add the trichlorophenol mixture to the same standard used to prepare the laboratory control sample (LCS).

8.3.1.1 The recovery of all LCS analytes shall be within acceptable range except 2,4,6-trichlorophenol, which shall not be detected; otherwise, the SPE sorbent is not performing properly, the lot shall be rejected.

8.4 Ethyl Alcohol, ACS Grade.

8.5 Ethyl Ether, Nano Grade, and free from analytes.

8.5.1 Ethyl ether shall be shown to be free of peroxides before use. Test strips are available for this. Procedures that recommend how to remove peroxides are available with the strips. After removal of peroxides, add 20 mL of ethyl alcohol to preserve each litre of ether.

8.6 Methylene Chloride, ACS Grade, demonstrated to be free of analytes.

8.7 Sodium Thiosulfate, granular.

8.8 Sodium Sulfate, Granular Anhydrous—Bake in a shallow tray at 450 °C for a minimum of 1 h. Cool in a desiccator and store in a precleaned glass bottle with a screw cap that prevents moisture from entering.

NOTE 3—If membrane-based solvent drying tubes are used, sodium sulfate is not needed.

⁷ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

8.9 Standard Solutions, Stock—Standards that are to be used for calibration shall be Independent Reference Materials (IRMs), where available. If an IRM is not available, then the standards shall be Reference Materials (RMs). If an IRM or RM, or both, are not available, the standard shall be manufactured under the requirements of ISO Guide 34. Laboratories may also prepare their own calibration standards from neat materials according to [8.9.1](#).

8.9.1 Prepare stock solutions by accurately weighing 0.0100 g of pure material. Dissolve the material in methanol or another suitable solvent and dilute to 10 mL in a volumetric flask. When the assay is 96 % or greater, the weight may be used without a correction to calculate the concentration of the stock standard.

8.9.1.1 Transfer the stock solutions to fluoropolymer-lined lid screw cap bottles. Store at $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ and protect from light. Check periodically for degradation or evaporation.

8.9.1.2 Replace stock standards after six months or according to the manufacturer's instructions. Replace sooner if comparison with check standards indicates a problem.

8.10 Standard Solutions, Secondary Dilution—Use stock standard solutions to prepare secondary dilution standard solutions that contain the analytes in methylene chloride. The secondary solutions should be prepared at concentrations that can be easily diluted to calibration standards that will bracket the calibration range ([Table 4](#)).

8.10.1 Store secondary dilution standards at $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, protected from light. Check periodically for evaporation and degradation.

8.10.2 Replace secondary dilution standards after six months or according to the manufacturer's instructions. Replace sooner if comparison with check standards indicates a problem.

8.11 Standard Solutions, Calibration—For [Table 1](#) analytes, prepare a minimum of five calibration solutions in methylene chloride.

8.11.1 One of the calibration standards should be at a concentration of the analyte near the Minimum Reporting Limit (MRL). The lower value may be rounded to a whole number that is more convenient for preparing the standard, but shall not exceed the MRL.

8.11.2 The other concentrations should correspond to the expected range of concentrations expected in samples or should define the working range.

8.11.3 Use a minimum of three concentrations of each Aroclor in [Table 2](#) to demonstrate the linearity of the detector response for individual congeners identified in [Table 8](#). Calibrate in separate mixtures at concentrations ranging from 0.5 ppb to 1000 ppb. Choose three to five of the individual characteristic congeners to represent each Aroclor.

8.11.4 Alternatively to [8.11.3](#), a standard containing a mixture of Aroclor 1016 and Aroclor 1260 that includes many of the peaks represented in the other Aroclor mixtures may be used. As a result, a multi-point initial calibration using a mixture of Aroclors 1016 and 1260 at three concentrations is enough to demonstrate the linearity of the detector response without the necessity of performing multi-point initial calibrations for each of the seven Aroclors. In addition, such a mixture

can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample.

8.11.5 If results are to be determined for individual PCB congeners, then standards for the pure congeners shall be prepared. [Table 4](#) lists characteristic congeners that may be used in the identification of the individual Aroclors. [Tables X1.1 and X2.1](#) include information on the additional congeners, but the analyst shall either document the resolution of the congeners in question or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

8.12 Internal Standard Spiking Solution:

8.12.1 Select a minimum of two internal standard compounds from [Table 5](#) that closely represent the pesticide compounds of interest. The compounds in [Table 5](#) were chosen because they do not interfere in the analysis.

8.12.2 Prepare a mixed internal standard solution at a concentration of 2 µg/L in acetone.

8.13 Surrogate Standard Spiking Solution:

8.13.1 Select a minimum of two surrogate compounds from [Table 5](#) that closely represent the pesticide compounds of interest. The compounds in [Table 5](#) do not interfere in the analysis.

8.13.2 Prepare a mixed surrogate solution at a concentration of 2 µg/L in acetone.

9. Hazards

9.1 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

10. Sample Collection, Preservation, and Storage

10.1 Sample Collection, Dechlorination, and Preservation:

10.1.1 Collect all chlorinated samples in 1000 mL amber glass bottles containing 75 mg of sodium thiosulfate crystals. If collecting less sample, use less sodium thiosulfate as appropriate. Un-chlorinated samples do not require sodium thiosulfate.

10.1.2 Collect non-chlorinated, non-tap water samples in accordance with Practices [D3370](#), Guide [D4448](#), Guide [D6538](#), or Practice [D6759](#). Document field activities according to Guide [D6089](#).

10.1.3 The samples shall be chilled to above freezing and $\leq 6\text{ }^{\circ}\text{C}$ on the day of collection and shall be maintained at that temperature until extraction. Field samples that will not be received at the laboratory on the day of collection shall be packaged for shipment with enough ice to ensure that they will be at $\leq 6\text{ }^{\circ}\text{C}$ on arrival at the laboratory. Samples received on ice on the same day they were collected do not have to be $\leq 6\text{ }^{\circ}\text{C}$ upon arrival.

10.2 Sample Storage:

10.2.1 Store samples at above freezing and $\leq 6\text{ }^{\circ}\text{C}$ until analysis.

10.2.2 Extract all samples within seven days of collection.

TABLE 4 Example Calibration Range and Typical Response Factors % RSD

ID	Compound	Dynamic Range (ng/L)	RF %RSD
1	PCB01_Monochlorobiphenyl_01	5 - 250	10.43
2	Pentachlorobenzene	0.5 - 250	6.67
3	PCB01_Monochlorobiphenyl_02	5 - 250	5.89
4	PCB01_Monochlorobiphenyl_03	50 - 250	8.83
5	PCB02_Dichlorobiphenyl_02	2 - 250	14.47
6	2,4,5,6-Tetrachloro-m-xylene	ISTD	-
7	PCB9-13C12	ISTD	-
8	PCB02_Dichlorobiphenyl_05	20 - 250	5.7
9	PCB02_Dichlorobiphenyl_04	2 - 250	5.88
10	PCB02_Dichlorobiphenyl_01	2 - 250	15.8
11	alpha-BHC	0.5 - 250	9.71
12	Hexachlorobenzene	0.5 - 250	7.77
13	gamma-BHC	0.5 - 250	14.03
14	beta-BHC	0.5 - 250	13.99
15	PCB03_Trichlorobiphenyl_01	0.5 - 250	13.99
16	PCB02_Dichlorobiphenyl_03	0.5 - 250	15.07
17	delta-BHC	0.5 - 250	7.36
18	PCB03_Trichlorobiphenyl_05	0.5 - 250	19.24
19	PCB03_Trichlorobiphenyl_02	0.5 - 250	11.73
20	PCB03_Trichlorobiphenyl_03	1 - 250	15.79
21	PCB03_Trichlorobiphenyl_04	1 - 250	8.39
22	Heptachlor	0.5 - 250	10.34
23	PCB04_Tetrachlorobiphenyl_01	2 - 250	13.06
24	PCB04_Tetrachlorobiphenyl_03	2 - 250	14.69
25	PCB04_Tetrachlorobiphenyl_05	10 - 250	5.9
26	Aldrin	1 - 250	10
27	PCB04_Tetrachlorobiphenyl_02	5 - 250	10.47
28	4,4'-Dibromobiphenyl	ISTD	-
29	PCB04_Tetrachlorobiphenyl_04	5 - 250	5.29
30	cis-Heptachlor Epoxide	2 - 250	7.98
31	Oxychlordane	2 - 250	12.09
32	trans-Heptachlor Epoxide	5 - 250	18.23
33	PCB05_Pentachlorobiphenyl_02	20 - 250	11.57
34	cis-Chlordane	1 - 250	14.33
35	PCB05_Pentachlorobiphenyl_05	50 - 250	14.47
36	2,4'-DDE	0.5 - 250	15.95
37	PCB05_Pentachlorobiphenyl_01	20 - 250	7.25
38	Endosulfan I	5 - 250	17.39
39	trans-Chlordane	2 - 250	5.99
40	trans-Nonachlor	5 - 250	6.07
41	PCB111-13C12	ISTD	-
42	4,4'-DDE	0.5 - 250	12.74
43	Dieldrin	5 - 250	19.19
44	PCB05_Pentachlorobiphenyl_03	50 - 100	6.96
45	2,4'-DDD	0.5 - 100	16.79
46	PCB06_Hexachlorobiphenyl_04	20 - 250	14.83
47	Endrin	5 - 250	11.3
48	PCB06_Hexachlorobiphenyl_02	10 - 250	1.53
49	Endosulfan II	5 - 250	10.35
50	PCB05_Pentachlorobiphenyl_04	50 - 250	8.8
51	4,4'-DDD	0.5 - 250	14.28
52	cis-Nonachlor	5 - 250	7.78
53	2,4'-DDT	0.5 - 250	12.84
54	Endrin Aldehyde	5 - 250	7.53
55	PCB06_Hexachlorobiphenyl_01	5 - 250	19.71
56	PCB06_Hexachlorobiphenyl_05	50 - 250	6.81
57	Endosulfan Sulfate	1 - 250	6.82
58	4,4'-DDT	0.5 - 250	10.15
59	PCB06_Hexachlorobiphenyl_03	5 - 250	2.71
60	PCB07_Heptachlorobiphenyl_02	5 - 250	11.16
61	PCB07_Heptachlorobiphenyl_05	5 - 250	18.67
62	Endrin Ketone	5 - 250	13.54
63	PCB07_Heptachlorobiphenyl_03	20 - 250	5.01
64	PCB08_Octachlorobiphenyl_05	100 - 250	15.69
65	PCB07_Heptachlorobiphenyl_01	1 - 250	3.67
66	Mirex	0.5 - 250	11.24
67	PCB07_Heptachlorobiphenyl_04	20 - 250	9.63
68	PCB08_Octachlorobiphenyl_01	50 - 250	12.41
69	PCB08_Octachlorobiphenyl_02	50 - 250	20.52
70	PCB09_Nonachlorobiphenyl_02	50 - 250	10.85
71	PCB08_Octachlorobiphenyl_04	50 - 250	23.39
72	PCB09_Nonachlorobiphenyl_03	50 - 250	24.57
73	PCB08_Octachlorobiphenyl_03	50 - 250	9.61
74	PCB09_Nonachlorobiphenyl_01	50 - 250	22.29