



Standard Test Method for Organohalide Pesticides and Polychlorinated Biphenyls in Water by Microextraction and Gas Chromatography¹

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1. Scope

1.1 This test method (**1, 2, 3**)² is applicable to the determination of the following analytes in finished drinking water, drinking water during intermediate stages of treatment, and the raw source water:

Analyte	Chemical Abstract Service Registry Number ^A
Alachlor	5972-60-8
Aldrin	309-00-2
Chlordane	57-74-9
Dieldrin	60-57-1
Endrin	72-20-8
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
Hexachlorobenzene	118-74-1
Lindane	58-89-9
Methoxychlor	72-43-5
Toxaphene	8001-35-2
Aroclor ^B 1016	12674-11-2
Aroclor ^B 1221	11104-28-2
Aroclor ^B 1232	11141-16-5
Aroclor ^B 1242	53469-21-9
Aroclor ^B 1248	12672-29-6
Aroclor ^B 1254	11097-69-1
Aroclor ^B 1260	11096-82-5

^A Numbering system of CAS Registry Services, P.O. Box 3343, Columbus, OH 43210-0334.

^B Aroclor is a registered trademark of Monsanto Co.

1.2 Detection limits for most test method analytes are less than 1 $\mu\text{g/L}$. Actual detection limits are highly dependent on the characteristics of the sample matrix and the gas chromatography system. **Table 1** contains the applicable concentration range for the precision and bias statements. Only Aroclor 1016 and 1254 were included in the interlaboratory test used to derive the precision and bias statements. Data for other PCB products are likely to be similar.

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.

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² The boldface numbers in parentheses refer to a list of references at the end of this test method.

1.3 Chlordane, toxaphene, and Aroclor products (polychlorinated biphenyls) are multicomponent materials. Precision and bias statements reflect recovery of these materials dosed into water samples. The precision and bias statements may not apply to environmentally altered materials or to samples containing complex mixtures of polychlorinated biphenyls (PCBs) and organochlorine pesticides.

1.4 For compounds other than those listed in 1.1 or for other sample sources, the analyst must demonstrate the applicability of this test method by collecting precision and bias data on spiked samples (groundwater, tap water) (**4**) and provide qualitative confirmation of results by gas chromatography/mass spectrometry (GC/MS) (**5**) or by GC analysis using dissimilar columns.

1.5 This test 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. Each analyst must demonstrate the ability to generate acceptable results using the procedure described in Section 13.

1.6 Analytes that are not separated chromatographically, (analytes that have very similar retention times) cannot be individually identified and measured in the same calibration mixture or water sample unless an alternative technique for identification and quantitation exists (see section 13.4).

1.7 When this test method is used to analyze unfamiliar samples for any or all of the analytes listed in 1.1, analyte identifications and concentrations should be confirmed by at least one additional technique.

1.8 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.

1.9 *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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 9.

TABLE 1 Test Method Precision and Bias^A as Functions of Concentration

Compound	Applicable Concentration Range, μ g/L	Water Type ^{B C D E}	
		Reagent water	Ground water
Alachlor	0.50 to 37.50	$S_o = 0.077X + 0.09$ $S_t = 0.107X + 0.15$ $X = 1.004C - 0.08$	$S_o = 0.075X + 0.05$ $S_t = 0.086X + 0.29$ $X = 1.059C + 0.03$
Aldrin	0.04 to 1.42	$S_o = 0.030X + 0.02$ $S_t = 0.251X + 0.00$ $X = 1.066C + 0.00$	$S_o = 0.115X + 0.00$ $S_t = 0.189X + 0.01$ $X = 0.945C - 0.00$
Chlordane	0.51 to 50.90	$S_o = 0.083X + 0.06$ $S_t = 0.125X + 0.19$ $X = 1.037C + 0.06$	$S_o = 0.062X + 0.09$ $S_t = 0.147X + 0.24$ $X = 0.941C + 0.09$
Dieldrin	0.10 to 7.53	$S_o = 0.091X + 0.01$ $S_t = 0.199X + 0.02$ $X = 1.027C + 0.00$	$S_o = 0.089X + 0.04$ $S_t = 0.221X + 0.04$ $X = 0.961C + 0.01$
Endrin	0.10 to 7.50	$S_o = 0.116X + 0.01$ $S_t = 0.134X + 0.02$ $X = 0.958C + 0.01$	$S_o = 0.045X + 0.15$ $S_t = 0.196X + 0.07$ $X = 0.958C + 0.05$
Heptachlor	0.04 to 1.41	$S_o = 0.104X + 0.01$ $S_t = 0.206X + 0.02$ $X = 1.002C + 0.02$	$S_o = 0.058X + 0.02$ $S_t = 0.153X + 0.02$ $X = 0.964C + 0.02$
Heptachlor Epoxide	0.04 to 1.42	$S_o = 0.031X + 0.02$ $S_t = 0.127X + 0.02$ $X = 0.952C + 0.00$	$S_o = 0.032X + 0.00$ $S_t = 0.103X + 0.02$ $X = 0.932C + 0.01$
Hexachlorobenzene	0.01 to 0.37	$S_o = 0.104X + 0.00$ $S_t = 0.231X + 0.00$ $X = 1.028C - 0.00$	$S_o = 0.148X + 0.00$ $S_t = 0.301X + 0.00$ $X = 0.901C - 0.00$
Lindane	0.04 to 1.39	$S_o = 0.056X + 0.01$ $S_t = 0.141X + 0.00$ $X = 1.009C - 0.00$	$S_o = 0.095X + 0.00$ $S_t = 0.134X - 0.00$ $X = 0.909C + 0.00$
Methoxychlor	0.20 to 15.00	$S_o = 0.115X + 0.12$ $S_t = 0.122X + 0.21$ $X = 0.950C + 0.15$	$S_o = 0.179X + 0.02$ $S_t = 0.210X + 0.08$ $X = 1.014C + 0.07$
Toxaphene	5.63 to 70.40	$S_o = 0.132X - 0.32$ $S_t = 0.273X - 0.72$ $X = 1.087C + 0.24$	$S_o = 0.067X + 0.28$ $S_t = 0.181X + 1.52$ $X = 0.903C + 0.50$
PCB-1016	0.50 to 49.80	$S_o = 0.106X + 0.31$ $S_t = 0.144X + 0.46$ $X = 0.856C + 0.31$	$S_o = 0.141X + 0.13$ $S_t = 0.218X + 0.06$ $X = 0.958C + 0.07$
PCB-1254	0.50 to 50.40	$S_o = 0.122X + 0.12$ $S_t = 0.282X + 0.05$ $X = 0.872C - 0.01$	$S_o = 0.126X + 0.17$ $S_t = 0.396X + 0.02$ $X = 0.938C - 0.02$

^A Bias = $C - X$.

^B X = Mean recovery.

^C C = True concentration value.

^D S_t = Overall standard deviation.

^E S_o = Single analyst standard deviation.

2. Referenced Documents

2.1 ASTM Standards:³

[D1129 Terminology Relating to Water](#)

[D1193 Specification for Reagent Water](#)

[D3534 Test Method for Polychlorinated Biphenyls \(PCBs\) in Water \(Withdrawn 2003\)⁴](#)

[D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water](#)

[D4128 Guide for Identification and Quantitation of Organic Compounds in Water by Combined Gas Chromatography](#)

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

⁴ The last approved version of this historical standard is referenced on www.astm.org.

and Electron Impact Mass Spectrometry
D4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data (Withdrawn 2002)⁴

E355 Practice for Gas Chromatography Terms and Relationships

2.2 *EPA Standards:*

Method 505, Analysis of Organohalide Pesticides and Aroclors in Water by Microextraction and Gas Chromatography⁵

Method 680, Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry⁵

3. Terminology

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *field duplicates (FD 1 and FD 2), n*—two separate samples collected at the same time and placed under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of FD 1 and FD 2 give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

3.2.2 *field reagent blank (FRB), n*—reagent water placed in a sample container in the laboratory and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The reagent water must be transferred to an empty, clean sample container in the field. The purpose of the FRB is to determine if analytes or other interferences are present in the field environment.

3.2.3 *instrument performance check solution (IPC), n*—a solution of analytes used to evaluate the performance of the instrument system with respect to test method criteria.

3.2.4 *laboratory duplicates (LD 1 and LD 2), n*—two sample aliquots taken in the analytical laboratory and analyzed separately with identical procedures. Analyses of LD 1 and LD 2 give a measure of the precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures.

3.2.5 *laboratory fortified blank (LFB), n*—an aliquot of reagent water to which known quantities of the 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.

3.2.6 *laboratory fortified sample matrix (LFM), n*—an aliquot of an environmental sample to which known quantities of the analytes are added in the laboratory. The LFM is analyzed as 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.

3.2.7 *laboratory reagent blank (LRB), n*—an aliquot of reagent water that is treated as a sample including exposure to all glassware, equipment, solvents, and reagents 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.

3.2.8 *standard solution, secondary dilution, n*—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.

3.2.9 *standard solution, stock, n*—a concentrated solution containing a single certified standard that is an analyte or a concentrated solution of a single analyte prepared in the laboratory with an assayed reference compound. Stock standard solutions are used to prepare secondary dilution standards.

3.2.10 *quality control sample (QCS), n*—a sample containing analytes or a solution of analytes in a water-miscible solvent used to fortify reagent water or environmental samples. The QCS must be independent of solutions used to prepare standards and should be obtained from a source external to the laboratory. The QCS is used to check laboratory performance with externally prepared test materials.

4. Summary of Test Method

4.1 This is a microextraction method in which 35 mL of sample are extracted with 2 mL of hexane. Two μL of the extract are injected into a gas chromatograph equipped with a linearized electron capture detector for separation and analysis. Aqueous calibration standards are extracted and analyzed in an identical manner to compensate for possible extraction losses.

4.2 The extraction and analysis time is 30 to 50 min per sample depending upon the analytes and the analytical conditions chosen.

4.3 This test method is based largely on EPA Method 505.

5. Significance and Use

5.1 The extensive and widespread use of organochlorine pesticides and PCBs has resulted in their presence in all parts of the environment. These compounds are persistent and may have adverse effects on the environment. Thus, there is a need to identify and quantitate these compounds in water samples.

6. Interferences

6.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in gas chromatograms. All reagents and apparatus must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in **12.2**.

6.1.1 Glassware must be scrupulously cleaned (**2**). Clean all glassware as soon as possible after use by thoroughly rinsing with the last solvent used in it. Follow by washing with hot tap water and detergent and thoroughly rinsing with tap and

⁵ Available from US EPA, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268.