



Standard Test Method for Determination of Organochlorine Pesticides in Water by Capillary Column Gas Chromatography¹

This standard is issued under the fixed designation D 5812; 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.

^{e1} NOTE—Editorial changes were made in July 2002.

1. Scope

1.1 This test method covers the capillary gas chromatographic determination of various organochlorine pesticides, including some of their degradation products and related compounds in finished drinking water. This test method is not limited to this particular aqueous matrix; however, its applicability to other aqueous matrices must be determined. The tested compounds include the following:

Pesticide	Chemical Abstract Service Registry Number ^A
Aldrin	309-00-2
α -BHC	319-84-6
β -BHC	319-85-7
γ -BHC	319-86-8
δ -BHC	58-89-9
α -Chlordane	5103-71-9
γ -Chlordane	5103-74-2
Chlorobenzilate	501-15-6
Chloroneb	2675-77-6
Chlorothalonil	2921-88-2
DCPA	1897-45-6
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-0708
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Etridiazole	2593-15-9
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Hexachlorobenzene	118-74-1
Methoxychlor	72-43-5
<i>cis</i> -Permethrin	52645-53-1
<i>trans</i> -Permethrin	52645-53-1
Propachlor	1918-16-7
Trifluralin	1582-09-8

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

¹ This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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1.2 Table 1 and Table 2 list the applicable concentration ranges and precision and bias statements for this test method. The applicability of this test method to other compounds must be demonstrated.

1.3 The extract derived from this procedure may be analyzed for these constituents by using the gas chromatography (GC) conditions prescribed in Test Method D 5175 (capillary column). Although the columns used in this test method may be adequate for analyzing PCBs, no data were collected for any multi-congener constituents during methods development.

1.4 This test method is restricted to use by or under the supervision of analysts experienced in the use of GC and interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results using the procedures described in Section 12.

1.5 Analytes that are not separated chromatographically by either the primary or secondary chromatographic columns (for example, analytes having very similar retention times) cannot be identified and measured individually in the same calibration mixture or water sample unless an alternative technique for identification and quantitation exists (see 7.9 and 13.4).

1.6 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.

1.7 The values stated in SI units are to be regarded as the 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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 9.

2. Referenced Documents

2.1 ASTM Standards:

D 1129 Terminology Relating to Water²

D 1192 Specification for Equipment for Sampling Water

² *Annual Book of ASTM Standards*, Vol 11.01.

TABLE 1 Regression Equations for Method Precision and Mean Recovery for Reagent Water

Compound	Concentration Range, μ g/L	Single-analyst Precision, s_r	Overall Precision, s_R	Mean Recovery, X
Aldrin	0.03–1.38	$0.061X + 0.004$	$0.130X + 0.009$	$0.909C + 0.007$
α -BHC	0.02–1.00	$0.059X + 0.001$	$0.127X + 0.005$	$1.015C + 0.004$
β -BHC	0.02–1.00	$0.034X + 0.004$	$0.148X + 0.005$	$0.975C + 0.006$
γ -BHC	0.03–1.51	$0.047X + 0.005$	$0.147X + 0.007$	$0.998C + 0.006$
δ -BHC	0.02–1.01	$0.050X + 0.001$	$0.119X + 0.002$	$0.958C + 0.005$
α -Chlordane	0.03–1.50	$0.062X + 0.000$	$0.138X + 0.000$	$1.008C + 0.003$
γ -Chlordane	0.03–1.51	$0.048X + 0.002$	$0.129X + 0.001$	$0.936C + 0.005$
Chlorobenzilate	1.00–50.00	$0.067X + 0.022$	$0.178X + 0.117$	$0.993C + 0.263$
Chloroneb	1.00–50.08	$0.111X - 0.016$	$0.159X + 0.275$	$0.942C + 0.280$
Chlorothalonil	0.05–2.51	$0.096X + 0.001$	$0.233X + 0.001$	$0.955C + 0.001$
DCPA	0.05–2.51	$0.047X + 0.002$	$0.161X + 0.002$	$0.998C + 0.013$
4,4'-DDD	0.05–2.50	$0.087X - 0.001$	$0.150X + 0.000$	$0.970C + 0.006$
4,4'-DDE	0.02–1.00	$0.093X + 0.001$	$0.166X + 0.000$	$0.982C + 0.000$
4,4'-DDT	0.12–6.01	$0.044X + 0.017$	$0.140X + 0.002$	$0.976C + 0.006$
Dieldrin	0.04–2.01	$0.089X + 0.000$	$0.150X + 0.009$	$0.962C + 0.009$
Endosulfan I	0.03–1.51	$0.070X + 0.000$	$0.127X + 0.009$	$0.957C + 0.006$
Endosulfan II	0.03–1.49	$0.059X + 0.001$	$0.120X + 0.002$	$0.974C + 0.003$
Endosulfan sulfate	0.03–1.51	$0.115X + 0.003$	$0.158X + 0.007$	$0.988C + 0.004$
Endrin	0.03–1.50	$0.108X - 0.002$	$0.134X + 0.002$	$0.991C + 0.002$
Endrin aldehyde	0.05–2.49	$0.105X - 0.004$	$0.121X + 0.003$	$0.940C + 0.007$
Etridiazole	0.05–2.48	$0.049X + 0.002$	$0.149X + 0.010$	$0.960C + 0.007$
Heptachlor	0.02–1.00	$0.068X + 0.001$	$0.100X + 0.011$	$0.961C + 0.009$
Heptachlor epoxide	0.03–1.50	$0.049X + 0.002$	$0.122X + 0.005$	$0.950C + 0.006$
Hexachlorobenzene	0.01–0.50	$0.049X + 0.000$	$0.124X + 0.003$	$0.841C + 0.003$
Methoxychlor	0.10–5.01	$0.108X - 0.004$	$0.190X - 0.003$	$1.044C + 0.016$
<i>cis</i> -Permethrin	1.00–50.08	$0.077X + 0.034$	$0.138X + 0.204$	$0.938C + 0.314$
<i>trans</i> -Permethrin	1.00–50.12	$0.096X - 0.001$	$0.233X + 0.001$	$0.955C + 0.001$
Propachlor	1.00–50.08	$0.052X + 0.098$	$0.119X + 0.370$	$0.978C + 0.317$
Trifluralin	0.05–2.51	$0.064X + 0.003$	$0.144X + 0.004$	$0.888C + 0.004$

TABLE 2 Regression Equations for Method Precision and Mean Recovery for Finished Drinking Water^A

Compound	Concentration Range, μ g/L	Single-analyst Precision, s_r	Overall Precision, s_R	Mean Recovery, X
Aldrin	0.03–1.49	$0.048X + 0.008$	$0.175X + 0.005$	$0.826C + 0.008$
α -BHC	0.02–1.00	$0.094X - 0.000$	$0.198X + 0.000$	$0.940C + 0.003$
β -BHC	0.02–1.00	$0.142X - 0.001$	$0.227X + 0.003$	$0.923C + 0.005$
γ -BHC	0.03–1.51	$0.070X - 0.001$	$0.138X + 0.006$	$0.938C + 0.002$
δ -BHC	0.02–1.01	$0.066X + 0.005$	$0.133X + 0.004$	$0.905C + 0.007$
α -Chlordane	0.03–1.50	$0.070X + 0.000$	$0.164X + 0.000$	$0.870C + 0.005$
γ -Chlordane	0.03–1.51	$0.072X + 0.000$	$0.138X + 0.001$	$0.865C + 0.005$
Chlorobenzilate	1.00–50.00	$0.146X - 0.042$	$0.243X + 0.292$	$0.874C + 0.207$
Chloroneb	1.00–50.08	$0.100X - 0.024$	$0.185X + 0.110$	$0.883C + 0.218$
Chlorothalonil	0.05–2.51	$0.100X + 0.001$	$0.180X + 0.004$	$0.920C + 0.000$
DCPA	0.05–2.51	$0.136X - 0.003$	$0.224X - 0.003$	$0.920C + 0.015$
4,4'-DDD	0.05–2.50	$0.102X + 0.001$	$0.146X + 0.002$	$0.908C + 0.008$
4,4'-DDE	0.02–1.00	$0.081X - 0.001$	$0.203X - 0.002$	$0.842C + 0.002$
4,4'-DDT	0.12–6.01	$0.110X - 0.005$	$0.162X + 0.012$	$0.858C + 0.009$
Dieldrin	0.04–2.01	$0.065X - 0.000$	$0.140X - 0.000$	$0.882C + 0.006$
Endosulfan I	0.03–1.51	$0.072X + 0.001$	$0.117X + 0.003$	$0.898C + 0.004$
Endosulfan II	0.03–1.49	$0.064X - 0.000$	$0.119X + 0.002$	$0.901C + 0.002$
Endosulfan sulfate	0.03–1.51	$0.132X - 0.000$	$0.233X + 0.007$	$0.948C + 0.009$
Endrin	0.03–1.50	$0.062X + 0.001$	$0.120X + 0.002$	$0.893C + 0.001$
Endrin aldehyde	0.05–2.49	$0.076X - 0.001$	$0.097X + 0.005$	$0.874C + 0.003$
Etridiazole	0.05–2.48	$0.074X + 0.001$	$0.240X - 0.000$	$0.916C + 0.009$
Heptachlor	0.02–1.00	$0.072X + 0.001$	$0.075X + 0.009$	$0.980C + 0.005$
Heptachlor epoxide	0.03–1.50	$0.066X + 0.001$	$0.084X + 0.004$	$0.944C + 0.006$
Hexachlorobenzene	0.01–0.50	$0.013X + 0.002$	$0.097X + 0.005$	$0.833C + 0.004$
Methoxychlor	0.10–5.01	$0.142X - 0.004$	$0.285X - 0.007$	$0.936C + 0.017$
<i>cis</i> -Permethrin	1.00–50.08	$0.112X + 0.012$	$0.161X + 0.292$	$0.833C + 0.200$
<i>trans</i> -Permethrin	1.00–50.12	$0.184X - 0.087$	$0.410X - 0.063$	$0.814C + 0.287$
Propachlor	1.00–50.08	$0.087X + 0.061$	$0.158X + 0.185$	$0.925C + 0.353$
Trifluralin	0.05–2.51	$0.066X + 0.002$	$0.147X + 0.004$	$0.847C + 0.006$

^A X = mean recovery; C = analyte true concentration.

and Steam in Closed Conduits²
D 1193 Specification for Reagent Water²
D 2777 Practice for Determination of Precision and Bias of
Applicable Methods of Committee D19 on Water²
D 3370 Practices for Sampling Water²

D 3694 Practices for Preparation of Sample Containers and
for Preservation of Organic Constituents³

³ Annual Book of ASTM Standards, Vol 11.02.

D 3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water²

D 4128 Practice for Identification of Organic Compounds in Water by Combined Gas Chromatography and Electron Impact Mass Spectrometry³

D 4210 Practice for Interlaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data²

D 5175 Test Method for Organohalide Pesticides and Polychlorinated Biphenyls in Water by Microextraction and Gas Chromatography³

D 5810 Guide for Spiking into Aqueous Samples³

E 260 Practice for Packed Column Gas Chromatography⁴

E 355 Practice for Gas Chromatography Terms and Relationships⁴

E 697 Practice for Use of Electron-Capture Detectors in Gas Chromatography⁴

E 1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs⁴

2.2 U.S. EPA Standards:

Method 508, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector (Revision 3.0, 1988)⁵

Analytical Methods for Pesticides/Aroclors (February 1991)⁶

Method 680, Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry (Revision 3.0, 1988)⁵

2.3 AOAC Standard:

Method 990.06, Organochlorine Pesticides in Water⁷

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129 and Practice E 355.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *field duplicates (FD 1 and FD 2)*—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 provide 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)*—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 whether analytes or other interferences are present in the field environment.

3.2.3 *instrument performance check (IPC) solution*—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)*—two sample aliquots taken in the analytical laboratory and analyzed separately with identical procedures. Analyses of LD 1 and LD 2 provide a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.2.4.1 *Discussion*—Analysis of laboratory duplicates or spiked samples requires the collection of duplicate 1-L sample bottles or the use of 2-L sample containers.

3.2.5 *laboratory fortified blank (LFB)*—an aliquot of reagent water to which known quantities of 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)*—an aliquot of an environmental sample to which known quantities of 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 analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations (see 3.2.4.1).

3.2.7 *laboratory reagent blank (LRB)*—an aliquot of reagent water that is treated exactly like a sample, including exposure to all glassware, equipment, solvents, and reagents that are used with other samples. The LRB is used to determine whether method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.

3.2.8 *quality control sample (QCS)*—a sample containing analytes or a solution of analytes in a water-miscible solvent that is 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 and is analyzed exactly like a sample.

3.2.9 *spike*—an addition of a known quantity of a component of known identity to a known volume of a sample in order to determine the efficiency with which the added component is recovered. Spike components should be prepared from a different source than that used for calibration standards. Refer to Guide D 5810 for guidance on spiking organics into aqueous samples.

3.2.10 *standard solution, secondary dilution*—a solution of several analytes prepared in the laboratory from stock analyte solutions and diluted as necessary to prepare calibration solutions and other needed analyte solutions.

3.2.11 *standard solution, stock*—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.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Available from U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268.

⁶ U.S. EPA CLP Statement of Work for Organics Analysis, Document OLM01.1.1, Available from U.S. EPA Contracts Management Division (MD33), Administration Building Lobby, Alexander Drive, Research Triangle Park, NC 27711.

⁷ Available from Association of Official Analytical Chemists, Suite 400, 2200 Wilson Boulevard, Arlington, VA 22201.