



# Standard Test Method for Determination of Polychlorinated Biphenyls (PCBs) in Waste Materials by Gas Chromatography<sup>1</sup>

This standard is issued under the fixed designation D 6160; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method<sup>2</sup> covers a two-tiered analytical approach to PCB screening and quantitation of liquid and solid wastes, such as oils, sludges, aqueous solutions, and other waste matrices.

1.2 Tier I is designed to screen samples rapidly for the presence of PCBs.

1.3 Tier II is used to determine the concentration of PCBs, typically in the range of from 2 to 50 mg/kg. PCB concentrations greater than 50 mg/kg are determined through analysis of sample dilutions.

1.4 This is a pattern recognition approach, which does *not* take into account individual congeners that might occur, such as in reaction by-products. This test method describes the use of Aroclors<sup>3</sup> 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268, as reference standards, but others could also be included. Aroclors 1016 and 1242 have similar capillary gas chromatography (GC) patterns. Interferences or weathering are especially problematic with Aroclors 1016, 1232, and 1242 and may make distinction between the three difficult.

1.5 This test method provides sample clean up and instrumental conditions necessary for the determination of Aroclors. Gas chromatography (GC) using capillary column separation technique and electron capture detector (ECD) are described. Other detectors, such as atomic emission detector (AED) and mass spectrometry (MS), may be used if sufficient performance (for example, sensitivity) is demonstrated. Further details about the use of GC and ECD are provided in Practices E 355, E 697, and E 1510.

1.6 Quantitative results are reported on the dry weights of waste samples.

1.7 Quantification limits will vary depending on the type of waste stream being analyzed.

1.8 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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 regulator limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>4</sup>

D 4059 Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography

E 203 Test Method for Water Using Volumetric Karl Fischer Titration

E 288 Specification for Laboratory Glass Volumetric Flasks

E 355 Practice for Gas Chromatography Terms and Relationships

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

E 969 Specification for Glass Volumetric (Transfer) Pipets

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

### 2.2 U.S. EPA Standards:

Method 608 Organochlorine Pesticides and PCBs<sup>5</sup>

Method 680 Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry<sup>6</sup>

Method 3620 Florisil Column Clean-Up<sup>7</sup>

Method 3630 Silica Gel Clean-Up<sup>7</sup>

Method 3660 Sulfur Clean-Up<sup>7</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04.0L on Gas Chromatography Methods.

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<sup>2</sup> This test method is based largely on EPA 8080 (and the proposed modification for the use of capillary columns, EPA 8081) and EPA Report 600/4-81-045 by Bellar, T. and J. Lichtenberg, reported in 1981. The report is titled, "The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils," and provides significant support to the protocol in this standard.

<sup>3</sup> Aroclor Standards may be purchased as 1000  $\mu\text{g/mL}$  in *isooctane*. Aroclor is a registered trademark of the Monsanto Company, 800 N. Lindbergh Blvd., St. Louis, MO 63167.

<sup>4</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>5</sup> EPA Report 600/4/82-057, Environmental Monitoring and Support Laboratory, Cincinnati, OH.

<sup>6</sup> Alford-Stevens, Ann, et al, Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory Office of Research and Development, USEPA, Cincinnati, OH.

<sup>7</sup> U.S. EPA. "Test Methods for Evaluating Solid Waste," *Physical/Chemical Methods*, SW-846.

Method 8082 Determination of PCB in Water and Soil/  
Sediment by Gas Chromatography: Capillary Column  
Technique<sup>7</sup>

### 3. Terminology

#### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *Aroclors, n*—commercial mixtures of polychlorinated biphenyl congeners marketed and trademarked by Monsanto prior to 1977.

3.1.1.1 *Discussion*—Specific Aroclors are usually designated by a four-digit number, with the first two digits usually designating the number of carbon atoms and the last two digits providing the chlorine content (for example, Aroclor 1260 is 60 % (weight) chlorine).

3.1.2 *congeners, n*—compounds related by structural similarities.

3.1.2.1 *Discussion*—All polychlorinated biphenyls (PCBs) share the same C<sub>12</sub> structure and vary only by the number and position of the chlorine atoms attached to the aromatic rings.

3.1.3 *continuing calibration standard (CCS)*—a known blend of one or more Aroclors at a fixed concentration that is injected into the gas chromatograph to demonstrate the validity of the calibration.

3.1.4 *dry weight, n*—concentration of PCBs after factoring out the water content.

3.1.4.1 *Discussion*—This correction assumes that all PCBs originated from nonaqueous sources and any water present has been added subsequently, diluting the original concentration. This correction can be described using the formula:

$$\text{Aroclor (mg/Kg) (dry)} = \frac{\text{Aroclor (mg/Kg) (wet)}}{(100 - \% \text{ water})/100} \quad (1)$$

3.1.5 *instrument performance standard (IPS), n*—a known low level of an Aroclor in a clean solvent used as a comparator to determine which qualitative (screening) results are of sufficient magnitude to require quantitative analyses.

3.1.6 *surrogate, n*—compound or compounds that are similar to analytes of interest in chemical composition, extraction, and chromatography, but that are not normally found at significant levels in the matrices of interest.

3.1.6.1 *Discussion*—Surrogates may be spiked into blanks, standards, samples, or matrix spikes prior to analysis to allow a determination of a quantitative recovery rate. Surrogates are also used to document matrix effects and method control.

3.1.7 *waste material, n*—any matter, within the scope of this test method, that is in the process of being recycled or disposed.

### 4. Summary of Test Method

4.1 The sample is extracted with solvent and the extract is treated to remove interfering substances, if needed. The sample extract is injected into a gas chromatograph. The components are separated as they pass through the capillary column and polychlorinated biphenyl compounds, if present, are detected by an ECD.

NOTE 1—Portions of this test method are similar to EPA Methods 608, 680, and 8082.

4.2 For screening (Tier I), instrument performance is monitored by a 2- $\mu$ L injection of a standard containing Aroclors

1016 and 1260. For low level work (1 ppm) the instrument is checked with a standard concentration of 0.01  $\mu$ g/mL (each) and for higher level work (10 ppm), the instrument is checked with a 0.1  $\mu$ g/mL standard.

4.3 Identification involves a pattern comparison of the chromatograms of an unknown sample with that of a standard obtained under identical instrumental conditions.

4.4 When quantification is required (Tier II), an external standards method (ESTD) is used. The quantitation technique typically requires a comparison of five peaks (minimum of three) between the chromatograms of an unknown sample and that of standard Aroclor obtained under identical conditions. Quantitation of either Aroclors 1016 or 1260 is performed using a five-point calibration of a mixed Aroclor standard containing Aroclors 1016 and 1260. All remaining Aroclors are quantitated from single point calibrations. Calibration is verified daily by comparison of results obtained for analysis of the midpoint calibration standard of Aroclor 1016 and 1260 to the five-point calibration curve. (See [Appendix X1](#) for an example chromatogram and calibration table.)

### 5. Significance and Use

5.1 This test method provides sufficient PCB data for many regulatory requirements. While the most common regulatory level is 50 ppm (dry weight corrected), lower limits are used in some locations. Since sensitivities will vary for different types of samples, one shall demonstrate a sufficient method detection limit for the matrix of interest.

5.2 This test method differs from Test Method [D 4059](#) in that it provides for more sample clean-up options, utilizes a capillary column for better pattern recognition and interference discrimination, and includes both a qualitative screening and a quantitative results option.

### 6. Interferences

6.1 The ECD has selective sensitivity to alkyl halides, conjugated carbonyls, nitrogen compounds, organometallics, and sulfur. Therefore, the chromatogram obtained for each sample shall be carefully compared to chromatograms of standards to allow proper interpretation.

6.2 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts or interferences, or both, to standard analysis. All these materials shall be demonstrated to be free from interferences under the conditions of analysis by analyzing method blanks.

6.3 Interferences from phthalate esters may pose a major problem in Aroclor determinations when using ECD. Phthalates generally appear in the chromatogram as broad late eluting peaks. Since phthalates are commonly used as plasticizers and are easily extracted from plastic, all contact of samples and extracts with plastic should be avoided.

6.4 While general clean-up techniques are provided as part of this test method, some samples may require additional clean-up beyond the scope of this test method before proper instrumental analysis may be performed.

## 7. Apparatus

7.1 *Gas Chromatograph*, a temperature programmable gas chromatograph suitable for splitless injections; equipped with an ECD.

7.2 *Data System*, a data system capable of measuring peak areas.

7.3 *Regulator (Make-up Gas)*—N<sub>2</sub> or Ar:Methane (95:5); two stage regulator rated at 20 MPa (3000 psi) inlet and 35 to 860 kPa (5 to 125 psi) outlet.

7.4 *Regulator (Carrier Gas)*—H<sub>2</sub>, two-stage regulator rated at 20 MPa (3000 psi) inlet and 35 to 860 kPa (5 to 125 psi) outlet.

7.5 *Gas Purifiers*, to remove moisture and particulates. Depending on the levels and types of interferences encountered, these might involve molecular sieves (moisture), activated carbon (organics), or other commercially-available media.

7.6 *Flow Meter*, to measure gas flow. Typical range is from 0.5 to 50 mL/min. ± 0.1 mL/min.

7.7 *Column*, crosslinked 5 % phenyl methyl silicone, 30 m by 0.32 mm id by 0.25 μm film thickness.

7.7.1 It is possible that other columns will provide sufficient separating power, but this shall be demonstrated before use.

7.8 *Analytical Balance*, capable of weighing to 0.0001 g.

7.9 *Volumetric Flasks*, 10, 50, 100, 200 mL, (see Specification E 288) Class A with ground-glass stoppers.

7.10 *Vortex Mixer*:

7.11 *Vials*, glass, 20 mL and 40 mL capacity with TFE-fluorocarbon-lined caps.

7.12 *Septum Inserts*—Inserts shall be treated with a silylation reagent before use or after cleaning. (See Annex A2 for possible procedure.) They may be purchased already treated.

7.13 *Volumetric Pipette*, 1, 5, 10 mL (see Specification E 969), Class A.

7.14 *Syringe*, 500 μL, mechanical guide.

## 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 conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>8</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Acetone*—(Warning—Extremely flammable. Vapors may cause flash fire.)

8.3 *Activated Magnesium Silicate (Florisol)*, Pesticide residue (PR) grade (60/100 mesh); store in glass containers with ground glass stoppers or foil lined screw caps.

8.3.1 Just before use, activate each batch at least 4 h at 130°C in a glass container loosely covered with aluminum foil. Alternatively, store the magnesium silicate in an oven at 130°C. Cool the magnesium silicate in a desiccator for 30 min before use.

8.4 *Hexane*—(Warning—Extremely flammable. Harmful if inhaled. May produce nerve cell damage. Vapors may cause flash fire.)

8.5 *Isooctane*—(Warning—Extremely flammable. Harmful if inhaled. Vapors may cause flash fire.)

8.6 *Methanol*—(Warning—Flammable. Vapor harmful. May be fatal or cause blindness if swallowed or inhaled. Cannot be made nonpoisonous.)

8.7 *Silylation Reagent* (for example, 5 % dimethyldichlorosilane in toluene). See Annex A2 for instructions.

8.8 *Sodium Sulfate*, granular, anhydrous (maintained at 130°C for at least 24 h prior to use). Cool the sodium sulfate in a desiccator for 30 min before use.

8.9 *Sulfuric Acid (concentrated)*:

8.10 *Acetone/Hexane*, 10 % acetone/90 % hexane (v/v).

8.11 *Gases*, Hydrogen (zero grade; 99.995 % purity) and nitrogen (zero grade; 99.998 % purity) or argon/methane (95:5; ECD grade).

8.11.1 Care shall be given to ensure purity of the carrier gas. For example, an in-line filter may be required.

8.12 *Aroclor Standards*<sup>3</sup>, Aroclor 1016, 1221, 1232, 1242, 1254, 1260, 1262, 1268.

8.13 *Decachlorobiphenyl (DCB) (surrogate) Optional*:

8.13.1 *Surrogate Stock Standard (15 μg/mL) Preparation*—Accurately dilute 1.5 mL of 1000 μg/mL DCB concentrate in 100 mL volumetric flask and fill to the mark with methanol, yielding a 15 μg/mL solution.

8.13.2 *Surrogate Working Standard (1.5 μg/mL) Preparation*—Accurately dilute 10 mL of the 15 μg/mL DCB stock standard in a 100 mL volumetric flask and fill to the mark with methanol, yielding a 1.5 μg/mL working DCB standard.

NOTE 2—Sample preparations will normally use 0.1 mL of this solution. The resulting concentration in the sample extract is 0.005 μg/mL before any further dilutions. The following calculations show this.

$$\frac{1.5 \mu\text{g/mL} \times 0.1 \text{ mL} = 0.15 \mu\text{g}}{(3.0 \text{ mL sample} + 27 \text{ mL})} = 0.005 \mu\text{g/mL} \quad (2)$$

8.14 *Calibration Standards*:

8.14.1 *Intermediate Stock Standard (50 μg/mL)*:

If high level standards (for example, commercially available standards at 2000 to 5000 μg/mL) have been purchased, prepare solutions of 50 μg/mL concentration.

8.14.1.1 The surrogate calibration standard may be added (optional) to the Aroclor 1016/1260 intermediate stock standard at a concentration of 2.5 μg/mL. For preparation of the standard, add 500 μL of 50 μg/mL surrogate to a 10 mL volumetric flask containing 3.0 mL of *isooctane*. Add the Aroclor 1016/1260 standard (5.0 mL at 100 μg/mL) to the flask. Dilute to 10 mL volume with *isooctane* and mix well.

8.14.1.2 To prepare the continuing CCS, dilute 200 μL of the intermediate stock standard to 100 mL.

<sup>8</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual 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.