



Designation: D6160 – 21

Standard Test Method for Determination of Polychlorinated Biphenyls (PCBs) in Waste Materials by Gas Chromatography¹

This standard is issued under the fixed designation D6160; 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 method² 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 mg/kg 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 Aroclors³ 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 E355, E697, and E1510.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0L on Gas Chromatography Methods.

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² 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.

³ Aroclor Standards may be purchased as 1000 µg/mL in *isooctane*. Aroclor is a registered trademark of the Monsanto Company, 800 N. Lindbergh Blvd., St. Louis, MO 63167.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.10 *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:⁴

D4059 Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

E203 Test Method for Water Using Volumetric Karl Fischer Titration

E288 Specification for Laboratory Glass Volumetric Flasks

E355 Practice for Gas Chromatography Terms and Relationships

E697 Practice for Use of Electron-Capture Detectors in Gas Chromatography

E969 Specification for Glass Volumetric (Transfer) Pipets

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

⁴ 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.

*A Summary of Changes section appears at the end of this standard

2.2 U.S. EPA Standards:

Method 608 Organochlorine Pesticides and PCBs⁵

Method 680 Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry⁶

Method 3620 Florisil Column Clean-Up⁷

Method 3630 Silica Gel Clean-Up⁷

Method 3660 Sulfur Clean-Up⁷

Method 8082 Determination of PCB in Water and Soil/Sediment by Gas Chromatography: Capillary Column Technique⁷

3. Terminology

3.1 Definitions:

3.1.1 This test method makes reference to common gas chromatographic procedures, terms, and relationships. Detailed definitions of these can be found in Practice **E355** and Terminology **D4175**. In addition, definition of terms specific to this standard include:

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

3.1.2.1 *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).

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

3.1.3.1 *Discussion*—All polychlorinated biphenyls (PCBs) share the same C₁₂ structure and vary only by the number and position of the chlorine atoms attached to the aromatic rings.

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

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

3.1.5.1 *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:

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

3.1.6 *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.

3.1.7 *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.

3.1.7.1 *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.

3.1.8 *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 µL 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 µg/mL (each) and for higher level work (10 ppm), the instrument is checked with a 0.1 µg/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 **D4059** 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,

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

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

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