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.

Designation: D4059 – 00 (Reapproved 2018)

Standard Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography[1]

This standard is issued under the fixed designation D4059; 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 describes a quantitative determination of the concentration of polychlorinated biphenyls (PCBs) in electrical insulating liquids by gas chromatography. It also applies to the determination of PCB present in mixtures known as askarels, used as electrical insulating liquids.

1.2 The PCB mixtures known as Aroclors[2] were used in the formulation of the PCB-containing askarels manufactured in the United States. This test method may be applied to the determination of PCBs in insulating liquids contaminated by either individual Aroclors or mixtures of Aroclors. This technique may not be applicable to the determination of PCBs from other sources of contamination.

1.3 The precision and bias of this test method have been established only for PCB concentrations in electrical insulating mineral oils and silicones. The use of this test method has not been demonstrated for all insulating fluids. Some insulating liquids, such as halogenated hydrocarbons, interfere with the detection of PCBs and cannot be tested without pretreatment.

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

1.5 *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.*

1.6 *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:*[3]

D923 Practices for Sampling Electrical Insulating Liquids

3. Symbols

3.1 The following symbols are used in this test method:

- C —concentration of PCB (ppm by weight) in the insulating test specimen.
- C_i —concentration of PCB (ppm by weight) found for the peak, *i*, in the chromatogram of the insulating liquid test specimen.
- d —density of the test specimen at 25°C, g/mL.
- f_i —relative content of the PCB species associated with each individual peak, *i*, in the chromatogram of the standard Aroclor solution, %.
- M —total amount of PCB in the standard test specimen injected into the chromatograph, g.
- M_i —amount of PCB represented by peak, *i*, in the chromatogram of the standard Aroclor test specimen, g.
- $R_i^{\,s}$ —response of the detector to PCB components with relative retention time, *i*, in the chromatograms of the standard, *s*, solutions, response may be expressed as peak height, peak area, or integrator counts.
- $R_i^{\,x}$ —response of the detector to PCB components with relative retention time, *i*, in the chromatogram of an unknown test specimen, may be expressed as peak height, peak area, or integrator counts.
- $R_p^{\,s}$ —response of the detector to PCB components in the largest or most cleanly separated peaks, *p*, in chromatograms of standard solutions; may be expressed as peak height, peak area, or integrator counts.
- $R_p^{\,x}$ —response of the detector to PCB components in the largest or most cleanly separated peaks, *p*, in the chromatogram of an unknown test specimen contaminated by a single Aroclor; may be expressed in peak height, peak area, or integrator counts.
- $v^{\,s}$ —volume of the standard test specimen injected into the chromatograph, µL.
- $v^{\,x}$ —volume of the unknown test specimen injected into the chromatograph, µL.
- V —original volume of the test specimen to be analyzed, µL.
- V^s —total volume of the diluted standard, mL.
- V^x —total volume of the test specimen to be analyzed, µL.
- W^x —weight of the test specimen to be analyzed, g.
- W^s —weight of the initial standard Aroclor test specimen, g.

4. Summary of Test Method

4.1 The test specimen is diluted with a suitable solvent. The resulting solution is treated by a procedure to remove interfering substances after which a small portion of the resulting solution is injected into a gas chromatographic column. The components are separated as they pass through the column with

[1] This test method is under the jurisdiction of Committee D27 onElectrical Insulating Liquids and Gasesand is the direct responsibility of Subcommittee D27.03 on Analytical Tests.

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[2] Registered trademark of Monsanto Co.

[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.

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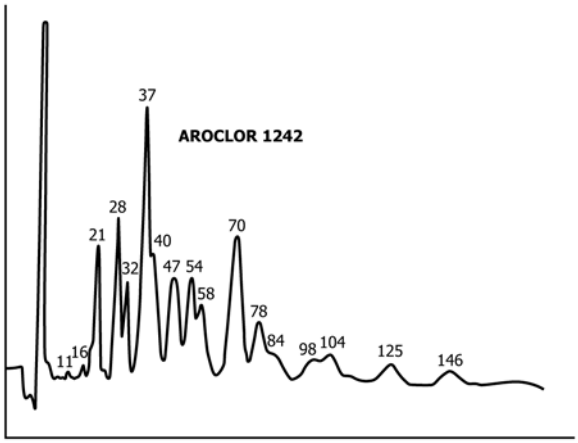

FIG. 1 Column: 3 % OV-1, Carrier Gas: Nitrogen at 60 mL/min, Column Temperature: 170°C, Detector: Electron Capture

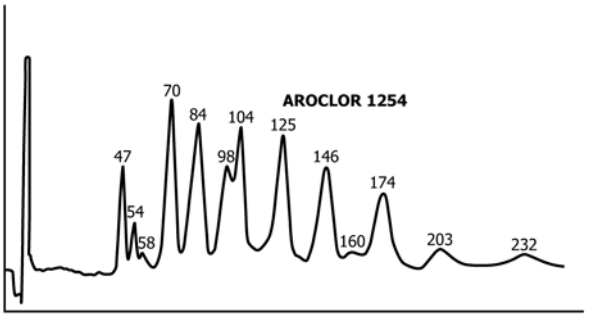

FIG. 2 Column: 3 % OV-1, Carrier Gas: Nitrogen at 60 mL/min, Column Temperature: 170°C, Detector: Electron Capture

carrier gas and their presence in the effluent is measured by an electron capture (EC) detector and recorded as a chromatogram. The test method is made quantitative by comparing the sample chromatogram with a chromatogram of a known quantity of one or more standard Aroclors, obtained under the same analytical conditions.

5. Significance and Use

5.1 United States governmental regulations mandate that electrical apparatus and electrical insulating fluids containing PCB be handled and disposed of through specific procedures. The procedure to be used for a particular apparatus or quantity of insulating fluid is determined by the PCB content of the fluid. The results of this analytical technique can be useful in selecting the appropriate handling and disposal procedure.

5.2 Quantification in this technique requires a peak-by-peak comparison of the chromatogram of an unknown specimen with that of standard Aroclor test specimens obtained under identical conditions. The amount of PCB producing each peak in the standard chromatogram shall be known independently.

5.3 The technique described is based on data for standard chromatograms of Aroclors 1242, 1254, and 1260 obtained using specific chromatographic column packing materials and operating conditions.[4] Relevant chromatograms are reproduced in Fig. 1, Fig. 2, and Fig. 3[5], for isothermal packed columns and in Figs. X4.1 through X4.3) for temperature programmed mega-bore capillary columns. Each peak is identified by its retention time relative to that of a standard. The types and amounts of PCB associated with each peak have been determined by mass spectroscopy and are given in Table 1, Table 2, and Table 3.[4] Other chromatographic operating conditions, and in particular, other column packing materials, may give different separations. The data given in the tables should not be used if chromatograms of the standards differ significantly from those shown in the figures. The peaks in such standard chromatograms shall be independently identified and quantified.

5.4 Different isomers of PCB with the same number of chlorine substituents can cause substantially different responses from EC detectors. Mixtures of PCB containing the same amount of PCB, but with a different ratio of isomers, can give quite different chromatograms. This technique is effective only when the standard PCB mixtures and those found in the unknown test specimen are closely related. Aroclors 1242, 1254, and 1260 are adequate standards because they have been found to be the most common PCB contaminant in electrical insulating oils.

6. Interferences

6.1 Electron capture detectors respond to other chlorine containing compounds and to certain other electrophilic materials containing elements such as other halogens, nitrogen, oxygen, and sulfur. These materials may give peaks with retention times comparable to those of PCBs. Most common interferences will be removed by the simple pre-analysis treatment steps detailed within this test method. The chromatogram of each analyzed test specimen should be carefully compared with those of the standards. The results of an analysis are suspect if major extraneous or unusually large individual peaks are found.

6.1.1 Data acquisition and treatment by electronic integrators or other instrumental means easily permits the unrecognized inclusion of interferences in the quantification of results.

[4] Webb, R. G., and McCall, A. C., *Journal of Chromatographic Science*, Vol 11, 1973, p. 366.

[5] Reproduced from the *Journal of Chromatographic Science* by permission of Preston Publications, Inc.

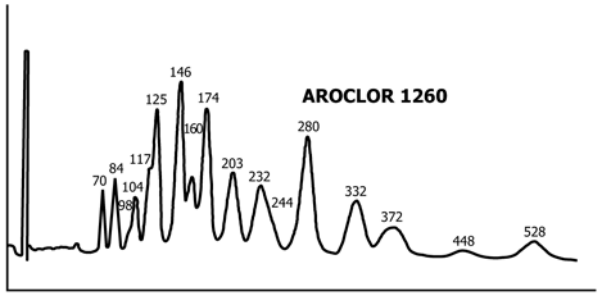

FIG. 3 Column: 3 % OV-1, Carrier Gas: Nitrogen at 60 mL/min, Column Temperature: 170°C, Detector: Electron Capture