Designation: D 4059 – 00

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

This standard is issued under the fixed designation D 4059; 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 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 923 Test Method for Sampling Electrical Insulating Liquids[3]

D 2233 Specification for Chlorinated Aromatic Hydrocarbons (Askarels) for Capacitors[3]

D 2283 Specification for Chlorinated Aromatic Hydrocarbons (Askarels) for Transformers[3]

D 3534 Test Method for Polychlorinated Biphenyls (PCBs) in Water[4]

E 260 Practice for Packed Column Gas Chromatography[5]

E 355 Practice for Gas Chromatography Terms and Relationships[5]

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 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

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

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[2] Registered trademark of Monsanto Co.

[3] *Annual Book of ASTM Standards*, Vol 10.03.

[4] *Annual Book of ASTM Standards*, Vol 11.02.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

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