ASTM D4059-00(2010) Standard Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography

Significance and Use

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.

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.

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. Relevant chromatograms are reproduced in Fig. 1, Fig. 2, and Fig. 3 , 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. 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.

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.

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

2. Referenced Documents *(purchase separately)*

ASTM Standards

D923 Practices for Sampling Electrical Insulating Liquids

3. Practical Application

Instrumentation: **Agilent 6890 GC with ECD** **(Ctrl + Click to follow link)**

Column: Agilent HP-5MS, 30 m × 0.25 mm X 0.25 µm

Injection Mode: Splitless

Carrier Gas: Helium at 1.9 mL/min constant flow

Col./Oven Temp: 70 °C hold 2 min, to 150 °C at 25 °C/min, then to 200 °C at 3 °C/min, then to 280 °C at 8 °C/min.

Inlet temperature: 250 °C

Injection volume: 1 µL

GC Detection: ECD at 270 °C

Results: The Aroclors identified have typical reporting limits ranging from 0.033 mg/Kg in soils to a range roughly ten times lower (2 µg/Kg) using advanced analytical techniques. In water samples, typical reporting limits are 0.01 – 1.0 µg/L with low level limits ranging around 0.005 µg/L**.** Mixtures of standards obtained as Aroclors from Alltech prepared in a 1:1 relationship (A1254:A1260), as well as a standard of 1,3,5-trichlorobenzene (1,3,5-TCB) can be used for this method. 39 PCB's can be quantified along with the 1,3,5-trichlorobenzene (1,3,5-TCB) peak.

References: http://www.astm.org/Standards/D4059.htm
http://www.jmcs.org.mx/PDFS/V49/N3/01-Robles.pdf
Larsen, B.; Bowadt, S.; Tilio, R., in: Environmental Analytical Chemistry of PCBs, Vol. 16, Albaigés J., Ed., Gordon and Breach Sci. Pub., Singapore 1993, 3-24
Erickson, M., in: Analytical Chemistry of PCBs; Second Ed., Lewis Pub., CRC Pres. 1997, 1-97.