

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1581

Polychlorinated Biphenyls in Oils

This Standard Reference Material (SRM) is intended primarily for calibrating instrumentation and validating methodology used in the determination of polychlorinated biphenyl mixtures (PCB's) in motor and transformer oils. These PCB's are present as Aroclor 1242 and Aroclor 1260. Certified concentrations are shown in Table 1.

Table 1. Certified Concentrations of Aroclor 1242 and Aroclor 1260 in
Motor and Transformer Oils:

<u>Matrix</u>	<u>Aroclor Type</u>	<u>Concentration, $\mu\text{g/g}^*$</u>
Motor Oil	1242	100 ± 1
Motor Oil	1260	100 ± 2
Transformer Oil	1242	100 ± 1
Transformer Oil	1260	100 ± 3

*Uncertainty is expressed at the 95% confidence level.

NOTICE AND WARNING TO USERS

Handling: PCB-containing materials are reported to be toxic and should be handled with care. Contact your regional office of the U.S. Environmental Protection Agency for information regarding proper disposal.

Expiration of Certification: This certification is valid within the specified uncertainty limits for two years from the date of purchase. In the event that the certification should become invalid before then, purchasers will be notified by NBS.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures between 10 to 30° C.

Preparation and analytical determinations were performed at the Center for Analytical Chemistry, Organic Analytical Research Division, by S.N. Chesler, F.R. Guenther, and R.M. Parris.

Consultation on the statistical design and analysis of the experimental work was provided by K. Kafadar and K.E. Eberhardt of the Statistical Engineering Division.

The coordination of the technical measurements leading to certification was under the direction of S.N. Chesler and H.S. Hertz.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234
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George A. Uriano, Chief
Office of Standard Reference Materials

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Preparation and Analysis

The Aroclors were obtained from the U.S. Food and Drug Administration Industrial Chemical Repository (Aroclor 1242, FDA 399-DCT-71696; Aroclor 1260, FDA 371-DCT-71699). The virgin motor oil base stock and the mineral base transformer oil were obtained from commercial sources. The PCB solutions were prepared by weighing and mixing the individual Aroclors and oils. The four solutions were then dispensed into 5-mL amber ampoules and flame sealed. Samples representing early, middle, and final stages of ampouling were analyzed using gas chromatography. Prior to gas chromatography, the oil solutions were separated into fractions using a high performance liquid chromatographic (HPLC) procedure. In this HPLC procedure, an aminosilane semi-preparative scale HPLC column was used. The mobile phase was 100 percent hexane and the effluent was monitored at a wavelength of 254 nm with a UV detector.

The HPLC fractionated samples were analyzed with a gas chromatograph equipped with an injector splitter and a 30 m x 0.25 mm nonpolar, immobilized phase, wall coated, open-tubular column. A constant current Ni⁶³ electron capture detector was used for these analyses. Quantitative results were obtained by using, as internal standards (IS), PCB isomers that were not detected in the analyte Aroclors. Calibration standards consisting of weighed amounts of the Aroclor and IS compounds in the PCB-free diluent transformer or motor oil were chromatographed to measure analyte response factors. The results of Aroclor 1260 are based on the areas of ten selected Aroclor PCB peaks and three IS peaks; for Aroclor 1242, on areas of nine Aroclor PCB and two IS peaks.

Table 2 lists the calculated and analytically determined concentrations of the Aroclors in the Oils.

Table 2. Calculated and Gas Chromatographic Results Used for Certification

<u>Matrix</u>	<u>Aroclor Type</u>	<u>Concentration, $\mu\text{g/g}$</u>	
		<u>Calculated^a</u>	<u>Gas Chromatography</u>
Motor Oil	1242	100.00 \pm 0.04 ^b	100.1 \pm 1.4 ^b
Motor Oil	1260	100.00 \pm 0.04	100.7 \pm 2.0
Transformer Oil	1242	99.96 \pm 0.04	100.4 \pm 1.2
Transformer Oil	1260	99.98 \pm 0.04	100.6 \pm 2.7

^aThe calculated concentration is based on the total mass of the Aroclor added to the oil.

^bUncertainty is given as 95% confidence limits.