

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 1585

#### Chlorinated Biphenyls in 2,2,4-Trimethylpentane (Isooctane)

This Standard Reference Material (SRM) is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of polychlorinated biphenyls. SRM 1585 consists of a set of five sealed ampoules containing a solution of eight chlorinated biphenyl congeners in 2,2,4-trimethylpentane (isooctane).

#### CERTIFIED CONCENTRATIONS OF THE CHLORINATED BIPHENYL CONGENERS

The certified concentrations and estimated uncertainties of eight chlorinated biphenyl congeners are shown in Table 1. Each value is based on the concentration calculated from the mass of the congener added to a known mass of 2,2,4-trimethylpentane and on the analytical results obtained by using capillary gas chromatography with electron capture detection (GC-ECD). Calculations of the values included correction of the measured mass of each congener by its measured purity. The uncertainties listed in Table 1 include estimates of both purity and chromatographic uncertainty components.

The calculated concentrations, the concentrations determined by the GC-ECD method, and the measured purities of the compounds are given in Table 2. The certified concentrations in Table 1 were derived from these data. The concentrations given in Table 2 have been corrected for the purity of the congeners determined by capillary GC equipped with flame ionization detection (GC-FID).

The significance of each of the chlorinated biphenyl compounds present in SRM 1585 is indicated in Table 3.

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

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

Use: Samples of the SRM for analysis should be withdrawn from ampoules immediately after opening and used without delay for the certified values listed in Table 1 to be valid within the stated uncertainties. Certified values are not applicable to material stored in ampoules after opening, even if the ampoules are resealed.

Preparation and analytical determinations were performed by S.N. Chesler, D.P. Enagonio, and R.M. Parris of the Organic Analytical Research Division, NBS Center for Analytical Chemistry.

Consultation on the statistical design of the experimental work and evaluation of data was provided by K.R. Eberhardt of the NBS Statistical Engineering Division.

The coordination of the technical measurements leading to the certification was under the direction of S.N. Chesler and R.M. Parris.

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.

January 30, 1986  
Gaithersburg, MD 20899

Stanley D. Rasberry, Chief  
Office of Standard Reference Materials

(over)

## PREPARATION AND ANALYSIS

The chlorinated biphenyl compounds and 2,2,4-trimethylpentane were obtained from commercial sources. A solution was prepared at NBS by weighing and mixing the individual compounds and 2,2,4-trimethylpentane. This solution was dispensed into 2-mL amber ampoules which were then flame sealed. Aliquots from randomly selected ampoules were analyzed using 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  $^{63}\text{Ni}$  electron capture detector was used for these analyses. Quantitative results were obtained by using 2,2',3,3'-tetrachlorobiphenyl (CB # 40) and 2,2',3,4,5,5'-hexachlorobiphenyl (CB # 141) as internal standards (IS). Calibration solutions consisting of weighted amounts of the compounds and IS compounds in 2,2,4-trimethylpentane were analyzed chromatographically to determine analyte response factors.

The estimated purity of each chlorinated biphenyl component was determined using a gas chromatograph equipped with a 30 m x 0.25 mm non-polar, immobilized phase, wall-coated, open-tubular column and a flame ionization detector. Hexane solutions of each compound were analyzed and the relative response factors of the impurities were determined using the method of Zoller et al.

### Reference

Zoller, W., Schafer, W., Class, T., and Ballschmiter, K., *Fresenius Z. Anal. Chem.* **321**, 247-251 (1985).

Table 1  
Certified Concentrations of Chlorinated Biphenyls in SRM 1585

CB No. <sup>b</sup>	Compound	$\mu\text{g/g}$	Concentration <sup>a</sup>
			$\mu\text{g/mL}^c$ at 23.0 °C
3	4-chlorobiphenyl	43.3 ± 1.0	29.9 ± 0.7
15	4,4'-dichlorobiphenyl	9.53 ± 0.08	6.57 ± 0.06
28	2,4,4'-trichlorobiphenyl	3.70 ± 0.02	2.55 ± 0.01
52	2,2',5,5'-tetrachlorobiphenyl	7.72 ± 0.06	5.32 ± 0.04
77	3,3',4,4'-tetrachlorobiphenyl	6.62 ± 0.05	4.56 ± 0.03
101	2,2',4,5,5'-pentachlorobiphenyl	5.24 ± 0.02	3.61 ± 0.01
138	2,2',3,4,4',5'-hexachlorobiphenyl	2.37 ± 0.02	1.63 ± 0.01
153	2,2',4,4',5,5'-hexachlorobiphenyl	3.06 ± 0.02	2.11 ± 0.01

<sup>a</sup> For each compound, the certified concentration is the mean of the calculated concentration and chromatographic determination. Both calculated and experimental concentrations were corrected for the percent purity of the chlorinated biphenyl components.

The stated uncertainty was computed as a 95% confidence interval for the chromatographic measured value plus an allowance for systematic error. The confidence interval reflects measurement error for both the purity of the CB congener components as well as the concentration measurements for the SRM itself. The allowance for systematic error is the magnitude of the difference between the certified value and the chromatographic determination.

<sup>b</sup> Ballschmiter, K., and Zell, M., *Fresenius Z., Anal. Chem.* **302**, 20-31 (1980).

<sup>c</sup> The concentration and uncertainty expressed in mass/volume units are applicable for use of this material at 23.0 °C. Because the density of 2,2,4-trimethylpentane changes with temperature, the concentration will also change as temperature changes and will be different than the value at 23.0 °C. However, the concentrations will change by less than one percent of the value listed if the SRM is used at temperatures in the 15 to 31 °C range. See "Selected Values of Properties of Hydrocarbons and Related Compounds," American Petroleum Institute Research Project 44, Thermodynamics Research Center, Texas A&M University, Table 3d, page 1, October 1952.

Table 2  
Summary of Results

CB No.	--Concentration <sup>a</sup> , $\mu\text{g/g}$ -		Capillary GC-FID purity, weight percent <sup>d</sup>
	Calculated <sup>b</sup>	GC-ECD <sup>c</sup>	
3	43.23 $\pm$ 0.03	43.45 $\pm$ 0.88	99.93 $\pm$ 0.02
15	9.544 $\pm$ 0.011	9.518 $\pm$ 0.067	96.92 $\pm$ 0.09
28	3.696 $\pm$ 0.003	3.703 $\pm$ 0.020	99.44 $\pm$ 0.02
52	7.714 $\pm$ 0.010	7.720 $\pm$ 0.058	97.74 $\pm$ 0.04
77	6.629 $\pm$ 0.009	6.606 $\pm$ 0.038	97.47 $\pm$ 0.17
101	5.234 $\pm$ 0.004	5.244 $\pm$ 0.019	97.90 $\pm$ 0.09
138	2.373 $\pm$ 0.002	2.362 $\pm$ 0.009	99.54 $\pm$ 0.01
153	3.060 $\pm$ 0.003	3.054 $\pm$ 0.012	97.66 $\pm$ 0.02

<sup>a</sup>Concentrations are corrected for the purity of the compounds.

<sup>b</sup>Uncertainty is expressed as  $\pm 2\sigma$  based on estimates of the accuracy and precision of the weighings performed on the two balances used.

<sup>c</sup>Uncertainty of the gas chromatographic (electron capture detection) results is expressed as 95 percent confidence limits.

<sup>d</sup>The purity values shown here reflect the presence of only the impurities that had a flame ionization detector (FID) response during the capillary GC analysis. Uncertainty of the purity determinations is expressed as the standard error.

Table 3  
Significance of Chlorinated Biphenyl Compounds in SRM 1585

CB No.	Compound	Significance
3	4-chlorobiphenyl	incidentally generated as a by-product of some industrial processes
15	4,4'-dichlorobiphenyl	incidentally generated as a by-product of some industrial processes
28	2,4,4'-trichlorobiphenyl	indicative of the presence of Aroclors 1016, 1242
52	2,2',5,5'-tetrachlorobiphenyl	indicative of the presence of Aroclors 1016, 1242
77	3,3',4,4'-tetrachlorobiphenyl	an especially toxic PCB compound
101	2,2',4,5,5'-pentachlorobiphenyl	indicative of the presence of Aroclors 1254, 1260
138	2,2',3,4,4',5'-hexachlorobiphenyl	indicative of the presence of Aroclors 1254, 1260
153	2,2',4,4',5,5'-hexachlorobiphenyl	indicative of the presence of Aroclors 1254, 1260