



# Certificate of Analysis

## Standard Reference Material<sup>®</sup> 1493

### Polychlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane

This Standard Reference Material (SRM) is a solution of 20 polychlorinated biphenyl (PCB) congeners in 2,2,4-trimethylpentane (isooctane). The Chemical Abstracts Service (CAS) Nomenclature and Registry Number for each component are listed in Table 1. This SRM is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of the certified compounds. A unit of SRM 1493 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

**Certified Mass Fraction Values:** Certified mass fraction values and estimated uncertainties for PCB congeners are given in Table 2. The certified mass fraction values are based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography with electron capture detection (GC-ECD). A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [1]. The measurands are the PCB congeners listed in Table 2, and metrological traceability is to the SI derived units for mass fraction, expressed as micrograms per kilogram.

**Reference Mass Fraction Values:** Reference mass fraction values and estimated uncertainties for additional PCB congeners are given in Table 3. A NIST reference value is a noncertified value that is the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1]. The measurands are the PCB congeners listed in Table 3, as determined by the indicated methods, and metrological traceability is to the SI derived units for mass fraction, expressed as micrograms per kilogram.

**Expiration of Certification:** The certification of **SRM 1493** is valid, within the measurement uncertainty specified, until **30 September 2036**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

**Maintenance of SRM Certification:** NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was performed by R.M. Parris and S.N. Chesler of the former NIST Analytical Chemistry Division. Coordination of the Certificate update was performed by M.M. Schantz and L.C. Sander of the NIST Chemical Sciences Division.

Partial support for the preparation and certification of this Standard Reference Material was provided by the National Oceanographic and Atmospheric Administration, National Ocean Service, Office of Ocean Resources Conservation and Assessment, and by the Environmental Protection Agency, Cincinnati Environmental Monitoring and Support Laboratory.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

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Certificate Issue Date: 17 May 2017  
*Certificate Revision History on Last Page*

Preparation and analytical measurements of the SRM were performed by S.N. Chesler, R.G. Christensen, F.R. Guenther, R.M. Parris and R.E. Rebbert of the former NIST Analytical Chemistry Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

**NOTICE AND WARNING TO USERS:** SRM 1493 IS INTENDED FOR RESEARCH USE.

### **INSTRUCTIONS FOR HANDLING, STORAGE, AND USE**

**Handling:** This SRM contains polychlorinated biphenyl (PCB) congeners, many of which have been reported to have toxic, mutagenic, and/or carcinogenic properties. Therefore, this material should be handled with care. Use proper methods for disposal of wastes.

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

**Use:** Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening an ampoule and should be processed without delay for the certified values in Table 2 to be valid within the stated uncertainties. Because of the volatility of 2,2,4-trimethylpentane, certified values are not applicable to material stored in ampoules that have been opened for more than 5 minutes, even if they are resealed.

### **PREPARATION AND ANALYSIS**

All chemicals used in the preparation of this SRM were obtained from commercial sources. The solution was prepared at NIST by weighing and mixing the individual compounds and 2,2,4-trimethylpentane. The total mass of this solution was measured, and the mass fractions were calculated from this gravimetric procedure. The gravimetric mass fractions were adjusted for the purity estimation of each component, as determined at NIST using capillary gas chromatography with flame ionization detection and, where appropriate, differential scanning calorimetry. A major impurity in PCB 206 was tentatively identified as PCB 194 (2,2',3,3',4,4',5,5'-octachlorobiphenyl). For the ampouling process, this bulk solution was then chilled to approximately -5 °C. From the bulk solution, 1.2 mL aliquots were dispensed into argon-flushed 2 mL amber glass ampoules, which were then flame sealed.

Aliquots from 12 randomly selected ampoules were analyzed in duplicate by GC-ECD on a 0.25 mm i.d. x 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness). PCB 116 (2,3,4,5,6-pentachlorobiphenyl) was added to each sample as an internal standard (IS) for quantification purposes. Calibration solutions, consisting of weighed amounts of the PCBs and the IS in 2,2,4-trimethylpentane, were chromatographically analyzed to determine analyte response factors.

Table 1. Chemical Abstracts Service (CAS) Nomenclature and Registry Numbers for PCBs in SRM 1493

PCB No. <sup>(a)</sup>	CAS Nomenclature	CAS Registry No.
8	2,4'-Dichlorobiphenyl	34883-43-7
18	2,2',5-Trichlorobiphenyl	37680-65-2
28	2,4,4'-Trichlorobiphenyl	7012-37-5
44	2,2',3,5'-Tetrachlorobiphenyl	41464-39-5
52	2,2',5,5'-Tetrachlorobiphenyl	35693-99-3
66	2,3',4,4'-Tetrachlorobiphenyl	32598-10-0
77	3,3',4,4'-Tetrachlorobiphenyl	32598-13-3
101	2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2
105	2,3,3',4,4'-Pentachlorobiphenyl	32598-14-4
118	2,3',4,4',5-Pentachlorobiphenyl	31508-00-6
126	3,3',4,4',5-Pentachlorobiphenyl	57465-28-8
128	2,2',3,3',4,4'-Hexachlorobiphenyl	38380-07-3
138	2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2
153	2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1
170	2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6
180	2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3
187	2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0
195	2,2',3,3',4,4',5,6-Octachlorobiphenyl	52663-78-2
206	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9
209	2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl	2051-24-3

<sup>(a)</sup> The PCB congener numbering scheme used here is as published by Ballschmiter and Zell [2] with revised numbering sequence as noted by Schulte and Malisch [3] in which the PCB congeners are numbered in accordance with IUPAC rules. For the specific congeners in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch.

Table 2. Certified Mass Fraction Values for Polychlorinated Biphenyl (PCB) Congeners in SRM 1493

PCB No. <sup>(a)</sup>	Compound	Mass Fraction (µg/kg) <sup>(b)</sup>	Concentration (ng/mL) <sup>(c)</sup>
PCB 18	2,2',5-Trichlorobiphenyl	290.8 ± 6.8	200.6 ± 4.7
PCB 28	2,4,4'-Trichlorobiphenyl	288.0 ± 1.7	198.7 ± 1.2
PCB 44	2,2',3,5'-Tetrachlorobiphenyl	289 ± 16	199 ± 11
PCB 52	2,2',5,5'-Tetrachlorobiphenyl	285.9 ± 6.0	197.2 ± 4.1
PCB 66	2,3',4,4'-Tetrachlorobiphenyl	291.9 ± 7.0	201.4 ± 4.8
PCB 77	3,3',4,4'-Tetrachlorobiphenyl	284.3 ± 2.6	196.2 ± 1.8
PCB 101	2,2',4,5,5'-Pentachlorobiphenyl	287.8 ± 5.3	198.5 ± 3.6
PCB 105	2,3,3',4,4'-Pentachlorobiphenyl	286 ± 25	197 ± 17
PCB 126	3,3',4,4',5-Pentachlorobiphenyl	287.4 ± 3.3	198.3 ± 2.3
PCB 128	2,2',3,3',4,4'-Hexachlorobiphenyl	290.0 ± 1.9	200.1 ± 1.3
PCB 138	2,2',3,4,4',5'-Hexachlorobiphenyl	287.1 ± 1.4	198.1 ± 1.0
PCB 153	2,2',4,4',5,5'-Hexachlorobiphenyl	287.5 ± 5.0	198.4 ± 3.5
PCB 170	2,2',3,3',4,4',5-Heptachlorobiphenyl	285.3 ± 6.6	196.8 ± 4.6
PCB 180	2,2',3,4,4',5,5'-Heptachlorobiphenyl	289.2 ± 5.4	199.5 ± 3.8
PCB 187	2,2',3,4',5,5',6-Heptachlorobiphenyl	285.3 ± 2.0	196.8 ± 1.4
PCB 195	2,2',3,3',4,4',5,6-Octachlorobiphenyl	289.0 ± 3.3	199.4 ± 2.3
PCB 206	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	259 ± 12	179.0 ± 8.5
PCB 209	2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl	289.6 ± 9.4	199.8 ± 6.5

<sup>(a)</sup> The PCB congener numbering scheme used here is as published by Ballschmitter and Zell [2] with revised numbering sequence as noted by Schulte and Malisch [3] in which the PCB congeners are numbered in accordance with IUPAC rules. For the specific congeners in this SRM, the Ballschmitter-Zell numbers correspond to those of Schulte and Malisch.

<sup>(b)</sup> The certified value is the equally weighted mean of the mass fractions determined gravimetrically and by using GC-ECD. The uncertainty of the certified value is the half-width of a 95 % confidence interval for the mean, with an allowance for systematic error between the methods. A significant portion of the total uncertainty is due to the uncertainty in the purity determination of the PCB material.

<sup>(c)</sup> The concentrations listed in nanogram per milliliter units were obtained by multiplying the certified values in micrograms per kilogram (prior to rounding) by the density of the SRM solution at 22.5 °C (0.6899 kg/L). These concentrations are for use in the temperature range of 20 °C to 25 °C, and an allowance for the change in density over this temperature range is included in the uncertainties.

Table 3. Reference Mass Fraction Values for PCB Congeners in SRM 1493

PCB No. <sup>(a)</sup>	Compound	Mass Fraction (µg/kg) <sup>(b)</sup>	Concentration (ng/mL) <sup>(c)</sup>
PCB 8	2,4'-Dichlorobiphenyl	277 ± 24	191 ± 17
PCB 118	2,3',4,4',5-Pentachlorobiphenyl	289 ± 30	199 ± 20

<sup>(a)</sup> The PCB congener numbering scheme used here is as published by Ballschmitter and Zell [2] with revised numbering sequence as noted by Schulte and Malisch [3] in which the PCB congeners are numbered in accordance with IUPAC rules. For the specific congeners in this SRM, the Ballschmitter-Zell numbers correspond to those of Schulte and Malisch.

<sup>(b)</sup> The reference value is the equally weighted mean of the mass fractions determined gravimetrically and by using GC-ECD. The uncertainty of the certified value represents a 95 % confidence interval for the mean.

<sup>(c)</sup> The concentrations listed in nanogram per milliliter units were obtained by multiplying the certified values in micrograms per kilogram (prior to rounding) by the density of the SRM solution at 22.5 °C (0.6899 kg/L). These concentrations are for use in the temperature range of 20 °C to 25 °C, and an allowance for the change in density over this temperature range is included in the uncertainties.

## REFERENCES

- [1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at <https://www.nist.gov/sites/default/files/documents/srm/SP260-136.PDF> (accessed May 2017).
- [2] Ballschmiter, K.; Zell, M.; *Analysis of Polychlorinated Biphenyls (PCB) by Glass Capillary Gas Chromatography – Composition of Technical Aroclor- and Clophen-PCB Mixtures*; *Fresenius Z. Anal. Chem.*, Vol. 302, pp. 20–31 (1980).
- [3] Schulte, E.; Malisch, R. *Calculation of the Real PCB Content in Environmental Samples. I. Investigation of the Composition of Two Technical PCB Mixtures*; *Fresenius Z. Anal. Chem.*, Vol. 314, pp. 545–551 (1983).

**Certificate Revision History:** 17 May 2017 (Change of expiration date; editorial changes); 29 January 2008 (Change of expiration date; editorial changes); 28 February 1995 (Original certificate date).

*Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*