National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 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 instruments 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 Concentration Values: The certified concentrations and estimated uncertainties for the 18 PCB congeners, expressed as mass fraction, are given in Table 2. The certified concentration values are based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography. 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.

Reference Concentration Values: The reference concentrations and estimated uncertainties for the remaining two PCB congeners, expressed as mass fraction, are given in Table 3. Reference values are noncertified values that are 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.

Expiration of Certification: The certification of SRM 1493 is valid, within the measurement uncertainty specified, until **31 December 2017**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for 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) will facilitate notification.

The coordination of the technical measurements leading to the certification of this SRM was under the direction of R.M.Parris and S.N.Chesler of the NIST Analytical Chemistry Division. The coordination of the update to the Certificate was under the direction of M.M. Schantz and L.C. Sander of the NIST Analytical Chemistry 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.

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See Certificate Revision History on Last Page

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Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.B. Schiller of the NIST Statistical Engineering Division.

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 NIST Analytical Chemistry Division.

Support aspects involved in the preparation of this SRM were coordinated through the NIST Measurement Services Division.

NOTICE AND WARNING TO USERS

Handling: This SRM contains polychlorinated biphenyls (PCBs), 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.

Instructions for Use: Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C immediately after opening the ampoules and should be processed without delay for the certified values in Table 2 to be valid within the stated uncertainty. 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 min, even if they are resealed.

PREPARATION AND ANALYSIS

All chemicals used in the preparation of this SRM were obtained from commercial sources. The PCB solution was prepared at NIST by weighing and mixing the individual CB congeners and 2,2,4-trimethylpentane until completely dissolved and homogenized. The total mass of this solution was then measured. The consensus purity estimations of the PCB components were based on NIST analyses 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. Each 2-mL amber ampoule was purged with argon just prior to the addition of approximately 1.2 mL of solution to the ampoule that was then flame-sealed.

Aliquots from 12 randomly selected ampoules were analyzed in duplicate by gas chromatography with electron capture detection (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 Number

PCB No.[a]	CAS Nomenclature[b]	CAS Registry Number[b]
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

[a] The PCB congener numbering scheme used here is as published by Ballschmiter and Zell [1] with revised numbering sequence as noted by Schulte and Malisch [2] in which the PCBs 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.

[b] Chemical Abstracts, Eleventh Collective Index, Index Guide, American Chemical Society, Columbus, OH, 1986.

Table 2. Certified Concentrations of Polychlorinated Biphenyl (PCB) Congeners in SRM 1493

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

[a] The PCB congener numbering scheme used here was first proposed by Ballschmiter and Zell [1] and later revised by Schulte and Malisch [2] in order to conform with IUPAC rules. For the specific congeners in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch.

[b] The certified value is the equally weighted mean of the gravimetric and average chromatographic concentration. 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.

[c] The concentrations listed in ng/mL units were obtained by multiplying the certified values in µg/kg (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 to 25 °C and an allowance for the change in density over this temperature range is included in the uncertainties.

Table 3. Reference Concentrations of PCB Congeners in SRM 1493

PCB No.	Compound	Mass Fraction[a] µg/kg	ng/Ml[b]
8	2,4′-dichlorobiphenyl	277 ± 24	191 ± 17
118	2,3′,4,4′,5-pentachlorobiphenyl	289 ± 30	199 ± 20

[a] The listed uncertainties are at the 95% level of confidence.

[b] The concentrations listed in ng/mL units were obtained by multiplying the values in µg/kg (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 to 25 °C and an allowance for the change in density over this temperature range is included in the uncertainties.

REFERENCES

[1] 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).

[2] 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: 29 January 2008 (Update of expiration date and editorial changes); 28 February 1995 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.