National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2262

Chlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane
(Nominal Concentration 2 μg/mL)

This Standard Reference Material (SRM) is a solution of 28 chlorinated biphenyl congeners, also known as 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 2262 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

Certified Mass Fraction Values: Certified mass fractions 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 milligrams 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 milligrams per kilogram.

Expiration of Certification: The certification of **SRM 2262** is valid, within the measurement uncertainty specified, until **30 September 2036**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use). The 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.A. Wise 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: 06 July 2017
See Certification History on Last Page

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Preparation and analytical measurements of the SRM were performed by 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 2262 IS INTENDED FOR RESEARCH USE.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: This SRM contains chlorinated biphenyls, 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 then 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 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 103 and PCB 185 were added to each sample as internal standards (IS) for quantification purposes. Calibration solutions, consisting of weighed amounts of SRM 1493 *Polychlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane*, the eight PCBs in SRM 2262 that are not components of SRM 1493, 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 2262

PCB No.[a]	CAS Nomenclature	CAS Registry No.
1	2-Monochlorobiphenyl	2051-60-7
8	2,4′-Dichlorobiphenyl	34883-43-7
18	2,2′,5-Trichlorobiphenyl	37680-65-2
28	2,4,4′-Trichlorobiphenyl	7012-37-5
29	2,4,5-Trichlorobiphenyl	15862-07-4
44	2,2′,3,5′-Tetrachlorobiphenyl	41464-39-5
50	2,2',4,6-Tetrachlorobiphenyl	62796-65-0
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
87	2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8
101	2,2′,4,5,5′-Pentachlorobiphenyl	37680-73-2
104	2,2',4,6,6'-Pentachlorobiphenyl	56558-16-8
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
154	2,2',4,4',5,6'-Hexachlorobiphenyl	60145-22-4
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
188	2,2',3,4',5,6,6'-Hepatachlorobiphenyl	74487-85-7
195	2,2′,3,3′,4,4′,5,6-Octachlorobiphenyl	52663-78-2
201[a]	2,2',3,3',4,5',6,6'-Octachlorobiphenyl	40186-71-8
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 [2] with revised numbering sequence as noted by Schulte and Malisch [3] 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 except for 2,2',3,3',4,5',6,6'-octachlorobiphenyl which is PCB 200 per the Ballschmiter system and PCB 201 in Schulte's scheme.

Table 2. Certified Mass Fraction Values for PCB Congeners in SRM 2262

PCB No.[a]	Compound	Mass Fraction (mg/kg)[b]	Concentration (μg/mL)[c]
1	2-Monochlorobiphenyl	2.997 ± 0.041	2.067 ± 0.029
8	2,4′-Dichlorobiphenyl	3.11 ± 0.26	2.14 ± 0.180
18	2,2′,5-Trichlorobiphenyl	2.983 ± 0.028	2.058 ± 0.019
29	2,4,5-Trichlorobiphenyl	2.98 ± 0.11	2.057 ± 0.076
44	2,2′,3,5′-Tetrachlorobiphenyl	2.977 ± 0.054	2.054 ± 0.037
52	2,2′,5,5′-Tetrachlorobiphenyl	2.996 ± 0.034	2.067 ± 0.024
66	2,3′,4,4′-Tetrachlorobiphenyl	2.973 ± 0.056	2.051 ± 0.039
77	3,3′,4,4′-Tetrachlorobiphenyl	3.04 ± 0.10	2.097 ± 0.069
87	2,2',3,4,5'-Pentachlorobiphenyl	3.000 ± 0.024	2.069 ± 0.017
101	2,2′,4,5,5′-Pentachlorobiphenyl	2.950 ± 0.041	2.035 ± 0.029
104	2,2',4,6,6'-Pentachlorobiphenyl	3.007 ± 0.024	2.074 ± 0.017
105	2,3,3′,4,4′-Pentachlorobiphenyl	2.960 ± 0.092	2.042 ± 0.063
118	2,3′,4,4′,5-Pentachlorobiphenyl	2.992 ± 0.095	2.064 ± 0.066
126	3,3′,4,4′,5-Pentachlorobiphenyl	3.01 ± 0.12	2.076 ± 0.086
128	2,2′,3,3′,4,4′-Hexachlorobiphenyl	2.985 ± 0.024	2.059 ± 0.017
138	2,2′,3,4,4′,5′-Hexachlorobiphenyl	2.939 ± 0.035	2.027 ± 0.024
153	2,2′,4,4′,5,5′-Hexachlorobiphenyl	2.957 ± 0.057	2.040 ± 0.039
170	2,2′,3,3′,4,4′,5-Heptachlorobiphenyl	2.964 ± 0.049	2.045 ± 0.034
180	2,2′,3,4,4′,5,5′-Heptachlorobiphenyl	2.986 ± 0.029	2.060 ± 0.020
187	2,2′,3,4′,5,5′,6-Heptachlorobiphenyl	2.967 ± 0.027	2.047 ± 0.019
188	2,2',3,4',5,6,6'-Hepatachlorobiphenyl	3.008 ± 0.050	2.075 ± 0.035
195	2,2′,3,3′,4,4′,5,6-Octachlorobiphenyl	2.974 ± 0.059	2.052 ± 0.041
201[a]	2,2',3,3',4,5',6,6'-Octachlorobiphenyl	3.001 ± 0.031	2.070 ± 0.022
206	2,2′,3,3′,4,4′,5,5′,6-Nonachlorobiphenyl	2.900 ± 0.054	2.001 ± 0.037
209	2,2′,3,3′,4,4′,5,5′,6,6′-Decachlorobiphenyl	2.989 ± 0.041	2.062 ± 0.028

[a] 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 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 except for 2,2',3,3',4,5',6,6'-octachlorobiphenyl which is PCB 200 per the Ballschmiter system and PCB 201 in Schulte's scheme.

[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 microgram per milliliter units were obtained by multiplying the certified values in milligrams 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 2262

PCB No.[a]	Compound	Mass Fraction (mg/kg)[b]	Concentration (μg/mL)[c]
28	2,4,4′-Trichlorobiphenyl	3.00 ± 0.10	2.07 ± 0.10
50	2,2',4,6-Tetrachlorobiphenyl	3.01 ± 0.12	2.08 ± 0.08
154	2,2',4,4',5,6'-Hexachlorobiphenyl	2.95 ± 0.10	2.03 ± 0.07

[a] 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 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 except for 2,2',3,3',4,5',6,6'-octachlorobiphenyl which is PCB 200 per the Ballschmiter system and PCB 201 in Schulte's scheme.

[b] 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.

[c] The concentrations listed in microgram per milliliter units were obtained by multiplying the certified values in milligrams 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 Jul 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: 06 July 2017 (Change of expiration date; removal of value for PCB 194; editorial changes); **31 March 2008** (Change of expiration date); **17 March 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; or via the Internet at http://www.nist.gov/srm.