National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2259

Polychlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane

This Standard Reference Material (SRM) is a solution of 80 polychlorinated biphenyl (PCB) congeners in 2,2,4-trimethylpentane (isooctane). 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 2259 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 1 along with the Chemical Abstract Service (CAS) Registry Numbers. 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 mass spectrometric detection (GC-MS). 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 1, and metrological traceability is to the SI derived units for mass fraction, expressed as micrograms per gram.

Expiration of Certification: The certification of **SRM 2259** is valid, within the measurement uncertainties 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"). 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 M.M. Schantz and L.C. Sander of the NIST Chemical Sciences Division.

Statistical consultation was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Preparation and analytical measurements of the SRM were performed by J.R. Kucklick and M.M. Schantz of the NIST Chemical Sciences Division and M.P. Cronise and C.N. Fales of the NIST Office of Reference Materials.

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

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

Steven J. Choquette, Director
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NOTICE AND WARNING TO USERS: SRM 2259 IS INTENDED FOR RESEARCH USE.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: This SRM contains 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.

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 1 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-trimethlpentane. 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 flame ionization capillary gas chromatography with two stationary phases of different polarities. From the bulk solution, 1.2 mL aliquots were dispensed into 2 mL amber glass ampoules, which were then flame sealed.

Aliquots from nine ampoules selected using a stratified, random sampling scheme were analyzed in duplicate by using GC-MS with two stationary phases, a non-polar 5 % (mole fraction) phenyl methylpolysiloxane phase and a non-polar proprietary phase. The seven internal standards added to each sample for quantification purposes were carbon-13 labeled PCB congeners. Calibration solutions, consisting of weighed amounts of the compounds (adjusted for the purity estimation) and the internal standard compounds in 2,2,4-trimethylpentane, were chromatographically analyzed to determine analyte response factors.

Table 1. Certified Mass Fraction Values for Polychlorinated Biphenyl (PCB) Congeners in SRM 2259

PCB No.[a]	BZ No.[a]	Compound	CAS Registry No.	Mass Fraction (μg/g)[b]
PCB 8		2,4'-Dichlorobiphenyl	34883-43-7	2.21 ± 0.18
PCB 18		2,2',5-Trichlorobiphenyl	37680-65-2	2.00 ± 0.14
PCB 28		2,4,4'-Trichlorobiphenyl	7012-37-5	1.99 ± 0.14
PCB 29		2,4,5-Trichlorobiphenyl	15862-07-4	0.363 ± 0.025
PCB 31		2,4',5-Trichlorobiphenyl	16606-02-3	4.20 ± 0.24
PCB 44		2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	2.53 ± 0.11
PCB 45		2,2',3,6-Tetrachlorobiphenyl	70362-45-7	0.574 ± 0.025
PCB 49		2,2',4,5'-Tetrachlorobiphenyl	41464-40-8	3.41 ± 0.14
PCB 52		2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	5.05 ± 0.47
PCB 56		2,3,3',4'-Tetrachlorobiphenyl	41464-43-1	4.18 ± 0.28
PCB 63		2,3,4',5-Tetrachlorobiphenyl	74472-34-7	1.039 ± 0.023
PCB 66		2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	6.34 ± 0.24
PCB 70		2,3',4',5-Tetrachlorobiphenyl	32598-11-1	2.18 ± 0.11
PCB 74		2,4,4',5-Tetrachlorobiphenyl	32690-93-0	4.00 ± 0.20
PCB 77		3,3',4,4'-Tetrachlorobiphenyl	32598-13-3	0.351 ± 0.015
PCB 79		3,3',4,5'-Tetrachlorobiphenyl	41464-48-6	0.610 ± 0.034
PCB 82		2,2',3,3',4-Pentachlorobiphenyl	52663-62-4	0.845 ± 0.018
PCB 87		2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	3.65 ± 0.17

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Table 1 Continued

PCB No.[a]	BZ No.[a]	Compound	CAS Registry No.	Mass Fraction (μg/g)[b]
PCB 92		2,2',3,5,5'-Pentachlorobiphenyl	52663-61-3	2.22 ± 0.12
PCB 95		2,2',3,5',6-Pentachlorobiphenyl	38379-99-6	5.84 ± 0.42
PCB 99		2,2',4,4',5-Pentachlorobiphenyl	38380-01-7	12.61 ± 0.66
PCB 101		2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	7.45 ± 0.56
PCB 105		2,3,3',4,4'-Pentachlorobiphenyl	32598-14-4	5.90 ± 0.26
PCB 106		2,3,3',4,5-Pentachlorobiphenyl	70424-69-0	0.314 ± 0.044
PCB 109	107	2,3,3',4',5-Pentachlorobiphenyl	70424-68-9	1.814 ± 0.068
PCB 110		2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	8.45 ± 0.58
PCB 112		2,3,3',5,6-Pentachlorobiphenyl	74472-36-9	0.527 ± 0.026
PCB 114		2,3,4,4',5-Pentachlorobiphenyl	74472-37-0	0.667 ± 0.043
PCB 118		2,3',4,4',5-Pentachlorobiphenyl	31508-00-6	20.79 ± 0.60
PCB 119		2,3',4,4',6-Pentachlorobiphenyl	56558-17-9	0.402 ± 0.045
PCB 121		2,3',4,5',6-Pentachlorobiphenyl	56558-18-0	0.355 ± 0.008
PCB 126		3,3',4,4',5-Pentachlorobiphenyl	57465-28-8	0.317 ± 0.011
PCB 127		3,3',4,5,5'-Pentachlorobiphenyl	39635-33-1	0.811 ± 0.050
PCB 128		2,2',3,3',4,4'-Hexachlorobiphenyl	38380-07-3	5.39 ± 0.14
PCB 130		2,2',3,3',4,5'-Hexachlorobiphenyl	52663-66-8	1.181 ± 0.078
PCB 132		2,2',3,3',4,6'-Hexachlorobiphenyl	38380-05-1	2.91 ± 0.22
PCB 137		2,2',3,4,4',5-Hexachlorobiphenyl	35694-06-5	0.964 ± 0.048
PCB 138		2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	31.2 ± 1.8
PCB 146		2,2',3,4',5,5'-Hexachlorobiphenyl	51908-16-8	7.04 ± 0.15
PCB 149		2,2',3,4',5',6-Hexachlorobiphenyl	38380-04-0	9.30 ± 0.91
PCB 151		2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	4.30 ± 0.22
PCB 153		2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	53.3 ± 3.0
PCB 154		2,2',4,4',5,6'-Hexachlorobiphenyl	60145-22-4	2.43 ± 0.20
PCB 156		2,3,3',4,4',5-Hexachlorobiphenyl	38380-08-4	1.740 ± 0.045
PCB 157		2,3,3',4,4',5'-Hexachlorobiphenyl	69782-90-7	1.263 ± 0.135
PCB 158		2,3,3',4,4',6-Hexachlorobiphenyl	74472-42-7	1.459 ± 0.052
PCB 159		2,3,3',4,5,5'-Hexachlorobiphenyl	39635-35-3	0.370 ± 0.016
PCB 163		2,3,3',4',5,6-Hexachlorobiphenyl	74472-44-9	7.43 ± 0.65
PCB 165		2,3,3',5,5',6-Hexachlorobiphenyl	74472-46-1	0.487 ± 0.013
PCB 166		2,3,4,4',5,6-Hexachlorobiphenyl	41411-63-6	0.746 ± 0.030
PCB 167		2,3',4,4',5,5'-Hexachlorobiphenyl	52663-72-6	1.874 ± 0.047
PCB 169		3,3',4,4',5,5'-Hexachlorobiphenyl	32774-16-6	0.285 ± 0.016
PCB 170		2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	6.73 ± 0.30
PCB 172		2,2',3,3',4,5,5'-Heptachlorobiphenyl	52663-74-8	1.513 ± 0.036
PCB 174		2,2',3,3',4,5,6'-Heptachlorobiphenyl	38411-25-5	2.52 ± 0.11
PCB 175		2,2',3,3',4,5',6-Heptachlorobiphenyl	40186-70-7	0.998 ± 0.038
PCB 176		2,2',3,3',4,6,6'-Heptachlorobiphenyl	52663-65-7	0.738 ± 0.038
PCB 177		2,2',3,3',4',5,6-Heptachlorobiphenyl	52663-70-4	3.09 ± 0.14
PCB 178		2,2',3,3',5,5',6-Heptachlorobiphenyl	52663-67-9	2.412 ± 0.086
PCB 180		2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	20.3 ± 1.2
PCB 183		2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	3.26 ± 0.11
PCB 185		2,2',3,4,5,5',6-Heptachlorobiphenyl	52712-05-7	0.369 ± 0.010

Table 1 Continued on Page 4

Table 1 Continued

PCB No.[a]	BZ No.[a]	Compound	CAS Registry No.	Mass Fraction (μg/g)[b]
PCB 187		2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	16.39 ± 0.74
PCB 188		2,2',3,4',5,6,6'-Hepatachlorobiphenyl	74487-85-7	0.399 ± 0.032
PCB 189		2,3,3',4,4',5,5'-Heptachlorobiphenyl	39635-31-9	0.498 ± 0.021
PCB 191		2,3,3',4,4',5',6-Heptachlorobiphenyl	74472-50-7	0.410 ± 0.009
PCB 193		2,3,3',4',5,5',6-Heptachlorobiphenyl	69782-91-8	1.00 ± 0.11
PCB 194		2,2',3,3',4,4',5,5'-Octachlorobiphenyl	35694-08-7	3.22 ± 0.28
PCB 195		2,2',3,3',4,4',5,6-Octachlorobiphenyl	52663-78-2	1.111 ± 0.084
PCB 196		2,2',3,3',4,4',5,6'-Octachlorobiphenyl	42740-50-1	6.28 ± 0.14
PCB 197		2,2',3,3',4,4',6,6'-Octachlorobiphenyl	33091-17-7	0.475 ± 0.026
PCB 199	201	2,2',3,3',4,5,5',6'-Octachlorobiphenyl	52663-75-9	2.857 ± 0.082
PCB 200	199	2,2',3,3',4,5,6,6'-Octachlorobiphenyl	52663-73-7	10.37 ± 0.23
PCB 201	200	2,2',3,3',4,5',6,6'-Octachlorobiphenyl	40186-71-8	0.385 ± 0.023
PCB 202		2,2',3,3',5,5',6,6'-Octachlorobiphenyl	2136-99-4	2.667 ± 0.085
PCB 205		2,3,3',4,4',5,5',6-Octachlorobiphenyl	74472-53-0	0.429 ± 0.013
PCB 206		2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	3.01 ± 0.22
PCB 207		2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl	52663-79-3	1.030 ± 0.021
PCB 208		2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl	52663-77-1	1.96 ± 0.11
PCB 209		2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl	2051-24-3	2.89 ± 0.25

[a] The PCB congener numbering scheme used here is published by Ballschmiter [2] and later revised 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 (BZ) numbers correspond to those of the Schulte and Malisch except for the PCB congeners annotated with a BZ numbering in the table

[b] The certified values are the equally weighted means of the mass fractions determined gravimetrically and by using GC-MS. The expanded uncertainty of each certified value represents a 95 % confidence interval for the mean using a coverage factor *k* of 2 and includes both correction for estimated purity and allowance for differences between the methods [4].

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).

[4] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed May 2017); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at https://www.nist.gov/pml/nist-technical-note-1297 (accessed May 2017).

Certificate Revision History: 17 May 2017 (Change of expiration date; editorial changes); 0**8 April 2009** (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.