National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2259
PCB 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. This SRM is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of PCB congeners. A unit of SRM 2259 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentrations of Constituents: The certified concentration values and estimated uncertainties for the 80 constituents, expressed as mass fractions, are given in Table 1 along with the Chemical Abstract Service (CAS) Registry Numbers. 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 [1].

Expiration of Certification: The certification of this SRM lot is valid until **31 December 2017**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, 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. Return of the attached registration card will facilitate notification.

The coordination of the technical measurements leading to the certification of this SRM was under the direction of M.M. Schantz and L.C. Sander of the NIST Analytical Chemistry Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Measurement Services Division.

Consultation on the statistical design of the experimental work and evaluation of the data were 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 Analytical Chemistry Division and M.P. Cronise and C.N. Fales of the Standard Reference Materials Group.

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Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 8 April 2009

Robert L. Watters, Jr., Chief
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NOTICE AND WARNING TO USERS

Handling: This material contains PCB congeners and should be handled with care. Use proper disposal methods.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures lower than 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

The compounds 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 weighed components were added to the 2,2,4-trimethylpentane and mixed overnight. The total mass of this solution was measured, and the concentrations were calculated from this gravimetric procedure. These gravimetric concentrations were adjusted for the purity estimation of each component, which was determined 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 capillary gas chromatography with mass spectrometric detection and 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 Concentrations of Components in SRM 2259

IUPAC #	(Compound name)[a]	CAS Registry No.[b]	Concentration µg/g[c]
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
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

Table 1 Continued on Page 3

Table 1 Continued:

IUPAC #	(Compound name)[a]	CAS Registry No.[b]	Concentration µg/g[c]
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	(2,3,3',4',5-Pentachlorobiphenyl) (BZ#107)	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
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

Table 1 Continued on Page 4

Table 1 Continued:

IUPAC #	(Compound name)[a]	CAS Registry No.[b]	Concentration µg/g[c]
PCB 197	(2,2',3,3',4,4',6,6'-Octachlorobiphenyl)	33091-17-7	0.475 ± 0.026
PCB 199	(2,2',3,3',4,5,5',6'-Octachlorobiphenyl) (BZ# 201)	52663-75-9	2.857 ± 0.082
PCB 200	(2,2',3,3',4,5,6,6'-Octachlorobiphenyl) (BZ# 199)	52663-73-7	10.37 ± 0.23
PCB 201	(2,2',3,3',4,5',6,6'-Octachlorobiphenyl) (BZ# 200)	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] PCBs are numbered based on the IUPAC numberings scheme [2] which differs from the Ballschmiter and Zell numbering scheme [3] for the PCBs noted in the table.

[b] Chemical Abstracts, Fourteenth Collective Index. Index Guide, American Chemical Society, Columbus, Ohio, 2001.

[c] The results are expressed as the certified value ± the expanded uncertainty. The certified value is the average of the concentrations determined by gravimetric and chromatographic measurements. The expanded 95% uncertainty uses a coverage factor of 2 and includes both correction for estimated purity and allowance for differences between the concentration determined by gravimetric preparation and chromatographic measurements [4].

REFERENCES

[1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260-136, U.S. Government Printing Office, Gaithersburg, MD (2000); available at http://www.cstl.nist.gov/nist839/NIST_special_publications.htm.

[2] Schulte, E. and 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. **314**, pp. 545-551, (1983).

[3] Ballschmiter, K. and Zell, M., "Analysis of Polychlorinated Biphenyls (PCB) by Glass Capillary Gas Chromatography - Composition of Technical Aroclor- and Clophen-PCB Mixtures," Fresenius Z. Anal. Chem. **302**, pp. 20-31, (1980).

[4] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Switzerland, 1993; see also Taylor, B.N., and Kuyatt, C.E., "*Guidelines for Evaluating and Expressing Uncertainty of NIST Measurement Results*," NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994) (available at http://physics.nist.gov/Pubs/).

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) 975-2200, Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://www.nist.gov/srm.