



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 (CB) [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 Concentration Values: The certified concentrations and estimated uncertainties for the 25 CB 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 three CB congeners and one CB congener that was identified as an impurity in one of the CB congeners used to prepare SRM 2262, 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 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 modified. NIST reserves the right to withdraw, amend, or extend this certification at anytime.

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.

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Certificate Issue Date: 31 March 2008
See Certification History on Last Page

The coordination of the technical measurements leading to the certification of this SRM was under the direction of R.M. Parris and S.A. Wise 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.

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 R.M. Parris and R.E. Rebert of the NIST Analytical Chemistry 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 CB 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 CB components were based on NIST analyses using capillary gas chromatography with flame ionization detection and, where appropriate, differential scanning calorimetry. A major impurity in CB 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. 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). PCBs 103 and 185 were added to each sample as internal standards (IS) for quantification purposes. Calibration solutions consisting of weighed amounts of NIST SRM 1493 (Polychlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane), the eight CBs 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 Number

CB No. ^a	CAS Nomenclature ^b	CAS Registry Number ^b
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	62796-65-8
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'-heptachlorobiphenyl	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 CB 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 CBs 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 CB 200 according to the Ballschmiter system and CB 201 in Schulte's scheme.

^b Chemical Abstracts, Eleventh Collective Index, Index Guide, American Chemical Society, Columbus, Ohio, 1986.

Table 2. Certified Concentrations of Chlorinated Biphenyl (CB) Congeners in SRM 2262

CB No. ^a	Compound	Mass Fraction ^b			
		mg/kg		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'-heptachlorobiphenyl	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	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 CB 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 CBs 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 CB 200 according to the Ballschmiter system and CB 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 CB material.

^c The concentrations listed in µg/mL units were obtained by multiplying the certified values in mg/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 °C 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 CB Congeners in SRM 2262

CB No.	Compound	Mass Fraction ^a					
		mg/kg			μg/mL ^b		
28	2,4,4'-trichlorobiphenyl	3.00	±	0.1	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
194 ^c	2,2',3,3',4,4',5,5'-octachlorobiphenyl	0.32	±	0.02	0.22	±	0.02

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

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

^c PCB 194 was a contaminant in the PCB 206 material used to prepare SRM 2262.

REFERENCES

- [1] 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).
- [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).

Certificate Revision History: 31 March 2008 (Extension of the certification period; 17 March 1995(Original certification date)

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