National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 2276

Three Planar Polychlorinated Biphenyl (PCB) Congeners in Isooctane

This Standard Reference Material (SRM) is a solution of three planar or "non-ortho" polychlorinated biphenyl (PCB) congeners in 2,2,4-trimethylpentane (isooctane) intended primarily for use in the calibration of chromatographic instrumentation. A unit of SRM 2276 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentrations of Constituent PCB Congeners: The certified concentration values for three planar PCB congeners are given in Table 1. These 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.

Supplemental Information: A summary of the gravimetric and gas chromatographic measurements for SRM 2276 is provided in Table 2. This information is **NOT** to be used as a substitute for NIST certified values. Chemical Abstracts Service (CAS) Nomenclature and Registry Numbers of the certified components are listed in Table 3.

Expiration of Certification: The certification **SRM 2276** is valid, within the measurement uncertainty specified, until **31 January 2020**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for 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) will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was under the direction of R.M. Parris and M.M. Schantz of the NIST Analytical Chemistry Division.

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

Partial support for the preparation and certification of this SRM was provided by the National Oceanic and Atmospheric Administration, National Ocean Service, Center for Coastal Monitoring and Assessment, Silver Spring, MD.

Preparation and analytical measurements of the SRM were performed by M.P. Cronise of the NIST Standards Reference Materials Program and R.M. Parris and L.K. Walton of the NIST Analytical Chemistry Division.

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

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Analytical Chemistry Division

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Certificate Issue Date: 06 November 2009
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
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INSTRUCTIONS FOR USE

Handling: This material contains polychlorinated biphenyls, many of which have been reported to have toxic, mutagenic, and/or carcinogenic properties, 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.

Opening of Ampoule: Open ampoules carefully to prevent contamination and injury. The ampoules are pre-scored and should **NOT** be opened using a file. 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

SRM Preparation: The PCB congeners used in the preparation of this SRM were obtained from AccuStandard, New Haven, CT and Ultra Scientific, North Kingston, RI. The solution was prepared at NIST by weighing and mixing the individual PCB congeners and 2,2,4-trimethylpentane. The weighed components were added to the 2,2,4-trimethylpentane and mixed until completely dissolved and homogenized. The total mass of this solution was measured and the concentrations calculated for the components (see Table 2). These gravimetric concentrations were adjusted for the consensus purity estimation of each of the components that were determined by using capillary gas chromatography with flame ionization detection, differential scanning calorimetry, and the purity assay information from the component suppliers. The bulk solution was then chilled to approximately –5 °C and 1.2 mL aliquots were dispensed into 2 mL amber glass ampoules that were then flame sealed.

SRM Analysis: Aliquots from nine ampoules selected using a random stratified sampling scheme were analyzed in duplicate by using capillary gas chromatography with electron capture detection employing an immobilized non-polar stationary phase column. An internal standard solution containing PCB 153 and PCB 201 was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of the PCB congeners (adjusted for the consensus purity estimation) and internal standard compounds in 2,2,4-trimethylpentane were chromatographically analyzed to determine analyte response factors. The analytical values determined for the compounds are given in Table 2.

Table 1. Certified Concentrations of the PCB Congeners in SRM 2276

PCB Number[a]	Compound	Concentration mg/kg[b]	Concentration µg/mL[c]
PCB 77	3,3',4,4'-Tetrachlorobiphenyl	2.927 ± 0.067	2.019 ± 0.046
PCB 126	3,3',4,4',5-Pentachlorobiphenyl	2.879 ± 0.064	1.986 ± 0.044
PCB 169	3,3',4,4',5,5'-Hexachlorobiphenyl	2.971 ± 0.067	2.050 ± 0.046

[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] Each result is expressed as the certified value ± the expanded uncertainty. The certified value is the unweighted average of the concentrations determined by gravimetric and chromatographic measurements. The expanded uncertainty, at the 95 % level of confidence, is calculated as $U = ku_c$, where u_c is a combined standard uncertainty calculated according to the ISO Guide [3] and $k = 2$ is the coverage factor. The value of u_c includes both a correction for estimated purity and an allowance for differences between the concentration determined by gravimetric preparation and chromatographic measurements.

[c] The certified concentrations, in µg/mL units, were obtained by multiplying the certified mass values by the measured density of the SRM solution at 22 °C (0.6899 g/mL). These concentrations are for use over 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 2. Supplemental Information for Three Planar PCB Congeners in SRM 2276[a]

PCB Number	Concentration	
	Gravimetric[b] mg/kg	GC-ECD[c] mg/kg
PCB 77	2.919	2.936 (± 0.005)
PCB 126	2.873	2.885 (± 0.005)
PCB 169	2.964	2.979 (± 0.010)

[a] Results are presented for use **only** as background information.

[b] Calculated concentration based on the mass of the PCB congener added to the total mass of the solution corrected for the chemical purity.

[c] Measured concentrations determined by using gas chromatography with electron capture detection (GC-ECD) corrected for the purity of the components. The listed uncertainties in parentheses represent one standard deviation of a single measurement for these results and recognize only the within-method variability.

Table 3. Chemical Abstracts Service (CAS) Nomenclature, and CAS Registry Number

PCB Number[a]	CAS Nomenclature[b]	CAS Registry Number[b]
PCB 77	3,3',4,4'-Tetrachlorobiphenyl	32598-13-3
PCB 126	3,3',4,4',5-Pentachlorobiphenyl	57465-28-8
PCB 169	3,3',4,4',5,5'-Hexachlorobiphenyl	32774-16-6

[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, Thirteenth Collective Index, Index Guide, American Chemical Society, Columbus, OH, 1996.

REFERENCES

[1] Ballschmiter, K.; Zell, M.; *Analysis of Polychlorinated Biphenyls* (*PCBs*) *by Glass Capillary Gas Chromatography – Composition of Technical Aroclor– and Clophen–PCB Mixtures*; Fresenius' J. 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' J. Anal. Chem., Vol. 314, pp. 545–551, (1983).

[3] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); 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 http://physics.nist.gov/Pubs/.

Certificate Revision History: 06 November 2009 (This revision reflects at extension of the certification period and editorial changes); 02 July 2001 (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) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.