National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2274

Polychlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane

This Standard Reference Material (SRM) is a solution of 11 polychlorinated biphenyl (PCB) congeners in 2,2,4-trimethylpentane (isooctane) intended primarily for use in the calibration of chromatographic instrumentation. A unit of SRM 2274 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Mass Fraction Values: The certified mass fraction values for PCB congeners are given in Table 1. These values are based on results obtained from the gravimetric preparation of the 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 1, and metrological traceability is to the SI derived units for mass fraction, expressed as milligrams per kilogram.

Supplemental Information: A summary of the gravimetric and GC-ECD measurements for SRM 2274 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. A representative chromatogram from the GC-ECD analysis is shown in Figure 1.

Expiration of Certification: The certification **SRM 2274** is valid, within the measurement uncertainty specified, until **31 January 2030**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage, Handling, 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 M.M. Schantz and S.A. Wise of the NIST Chemical Sciences Division.

Statistical consultation was 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 Office of Reference Materials and R.M. Parris and D.L. Poster of the NIST Chemical Sciences Division.

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

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Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

NOTICE AND WARNING TO USERS: SRM 2274 IS INTENDED FOR RESEARCH USE.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: This SRM contains 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.

Use: 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 an ampoule 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 PCB congeners used in the preparation of this SRM were obtained from AccuStandard (New Haven, CT). The solution was prepared at NIST by weighing and mixing the individual PCB congeners and 2,2,4-trimethylpentane. 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 component that was determined using capillary gas chromatography with flame ionization detection, differential scanning calorimetry, and the purity assay information from the component suppliers. 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 were taken from nine ampoules which were selected by random-stratified sampling. The aloquots were analyzed in duplicate by using GC-ECD employing an immobilized non-polar stationary phase column. An internal standard solution containing PCB 103 and PCB 198 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 also are given in Table 2.

Table 1. Certified Mass Fractions Values for PCB Congeners in SRM 2274

PCB No.[a]	Compound	Mass Fraction (mg/kg)[b]	Concentration (µg/mL)[c]
PCB 31	2,4',5-Trichlorobiphenyl	2.929 ± 0.074	2.021 ± 0.051
PCB 49	2,2',4,5'-Tetrachlorobiphenyl	2.916 ± 0.072	2.012 ± 0.050
PCB 95	2,2',3,4',6-Pentachlorobiphenyl	2.925 ± 0.063	2.018 ± 0.043
PCB 99	2,2',4,4',5-Pentachlorobiphenyl	2.933 ± 0.062	2.023 ± 0.043
PCB 110	2,3,3',4',6-Pentachlorobiphenyl	2.911 ± 0.059	2.008 ± 0.041
PCB 149	2,2',3,4',5',6-Hexachlorobiphenyl	2.911 ± 0.068	2.008 ± 0.047
PCB 151	2,2',3,5,5',6-Hexachlorobiphenyl	2.904 ± 0.064	2.003 ± 0.044
PCB 156	2,3,3',4,4',5-Hexachlorobiphenyl	2.917 ± 0.059	2.012 ± 0.041
PCB 169	3,3',4,4',5,5'-Hexachlorobiphenyl	2.902 ± 0.059	2.002 ± 0.041
PCB 183	2,2',3,4,4',5',6-Heptachlorobiphenyl	2.879 ± 0.059	1.986 ± 0.041
PCB 194	2,2',3,3',4,4',5,5'-Octachlorobiphenyl	2.889 ± 0.063	1.993 ± 0.043

[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 PCB congeners 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] 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 and NIST Guides [4] 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 fgravimetric preparation and chromatographic measurements.

[c] The concentrations in microgram per milliliter units were obtained by multiplying the certified values in milligrams per kilogram (prior to rounding) 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 PCB Congeners in SRM 2274[a]

PCB No.	Gravimetric[b] (mg/kg)	GC-ECD[c] (mg/kg)
PCB 31	2.936	2.923 ± 0.005
PCB 49	2.929	2.904 ± 0.008
PCB 95	2.929	2.921 ± 0.005
PCB 99	2.929	2.936 ± 0.006
PCB 110	2.911	2.911 ± 0.010
PCB 149	2.901	2.922 ± 0.011
PCB 151	2.911	2.898 ± 0.009
PCB 156	2.915	2.918 ± 0.013
PCB 169	2.901	2.903 ± 0.011
PCB 183	2.881	2.878 ± 0.011
PCB 194	2.895	2.885 ± 0.007

[a] Results presented for use **only** as background information. Supplimental information values cannot be used to establish metrological traceability.

[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] Concentrations determined by using GC-ECD corrected for the purity of the compounds. The listed uncertainties represent one standard deviation of a single measurement for these results and recognize only the within-method variability.

Table 3. Chemical Abstracts Service Nomenclature and Registry Numbers

PCB No.[a]	Compound	CAS Numbers[b]
PCB 31	2,4',5-Trichlorobiphenyl	16606-02-3
PCB 49	2,2',4,5'-Tetrachlorobiphenyl	41464-40-8
PCB 95	2,2',3,4',6-Pentachlorobiphenyl	38379-99-6
PCB 99	2,2',4,4',5-Pentachlorobiphenyl	38380-01-7
PCB 110	2,3,3',4',6-Pentachlorobiphenyl	38380-03-9
PCB 149	2,2',3,4',5',6-Hexachlorobiphenyl	38380-04-0
PCB 151	2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5
PCB 156	2,3,3',4,4',5-Hexachlorobiphenyl	38380-08-4
PCB 169	3,3',4,4',5,5'-Hexachlorobiphenyl	32774-16-6
PCB 183	2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1
PCB 194	2,2',3,3',4,4',5,5'-Octachlorobiphenyl	35694-08-7

[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 PCB congeners 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, Ohio, 1996.

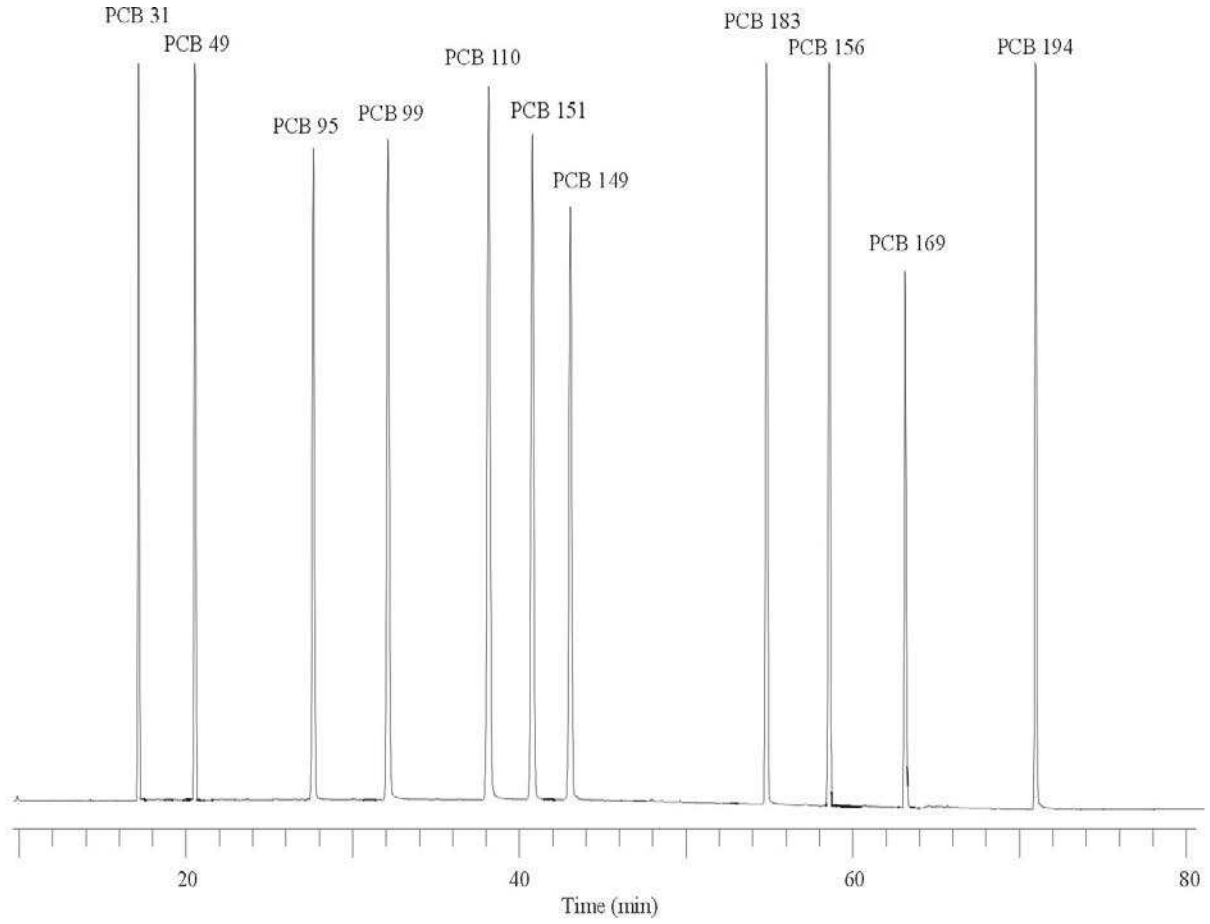

Figure 1. Example of the gas chromatographic elution order of analytes in SRM 2274 on a 5 % phenyl-substituted methylpolysiloxine capillary column (60 m x 0.25 mm i.d., 0.25 μm film thickness) with electron capture detection.

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 Oct 2019).

[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 https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Oct 2019); 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 Oct 2019).

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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 https://www.nist.gov/srm.