



Certificate of Analysis

Standard Reference Material[®] 2257

PBDE Congeners in 2,2,4-Trimethylpentane

This Standard Reference Material (SRM) is a solution of 38 polybrominated diphenyl ether (PBDE) congeners in 2,2,4-trimethylpentane. This SRM is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of PBDE congeners. A unit of SRM 2257 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 38 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, within the measurement uncertainties specified, until **31 January 2018**, 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 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.

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 D.L. Poster 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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NOTICE AND WARNING TO USERS

Handling: This material contains PBDE 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 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 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-trimethylpentane. 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 capillary gas chromatography with two stationary phases of different polarities. The bulk solution was chilled slightly, and 1.2 mL aliquots were dispensed into 2-milliliter 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 electron capture detection (GC-ECD) on a relatively non-polar 5% phenyl methylpolysiloxane phase (RXi-5MS, 15 m × 0.25 mm id, 0.25 µm film thickness, Restek, Bellefonte, PA)¹. The internal standard solution added to each sample for quantification purposes contained 4,4'-DDT-d₈, 2,2',4,5,5'-pentabromobiphenyl, and decabromobiphenyl. Calibration solutions consisting of weighed amounts of the 38 PBDE congeners and the internal standard compounds in 2,2,4-trimethylpentane were chromatographically analyzed to determine analyte response factors. The concentrations determined from the GC-ECD analysis were confirmed using a similar non-polar column in a GC equipped with negative chemical ionization mass spectrometric detection (NCI-MS). The internal standard solution used for the GC/NCI-MS analysis contained fluorinated 2,2',4,4'-tetrabromodiphenyl ether and carbon-13 labeled PBDE 209.

¹Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 1. Certified Concentrations of Components in SRM 2257

IUPAC No. (Compound name) ^(a)	CAS Registry No ^(b)	Concentration $\mu\text{g/g}^{(c)}$
PBDE 17 (2,2',4-Tribromodiphenyl ether)	147217-75-2	5.87 \pm 0.35
PBDE 25 (2,3',4-Tribromodiphenyl ether)	147217-77-4	3.19 \pm 0.39
PBDE 28 (2,4,4'-Tribromodipenyl ether)	41318-75-6	1.88 \pm 0.14
PBDE 30 (2,4,6-Tribromodiphenyl ether)	N/A	3.08 \pm 0.10
PBDE 47 (2,2',4,4'-Tetrabromodiphenyl ether)	5436-43-1	2.09 \pm 0.16
PBDE 49 (2,2',4,5'-Tetrabromodiphenyl ether)	243982-82-3	3.95 \pm 0.31
PBDE 66 (2,3',4,4'-Tetrabromodiphenyl ether)	189084-61-5	2.162 \pm 0.089
PBDE 71 (2,3',4',6-Tetrabromodiphenyl ether)	189084-62-6	2.86 \pm 0.10
PBDE 74 (2,4,4',5-Tetrabromodiphenyl ether)	N/A	3.56 \pm 0.10
PBDE 75 (2,4,4',6-Tetrabromodiphenyl ether)	189084-63-7	3.45 \pm 0.15
PBDE 85 (2,2',3,4,4'-Pentabromodiphenyl ether)	182346-21-0	3.359 \pm 0.092
PBDE 97 (2,2',3',4,5-Pentabromodiphenyl ether)	N/A	
118 (2,3',4,4',5-Pentabromodiphenyl ether)	N/A	5.29 \pm 0.19
PBDE 99 (2,2',4,4',5-Pentabromodiphenyl ether)	60348-60-9	2.127 \pm 0.090
PBDE 100 (2,2',4,4',6-Pentabromodiphenyl ether)	189084-64-8	3.05 \pm 0.15
PBDE 101 (2,2',4,5,5'-Pentabromodiphenyl ether)	N/A	3.784 \pm 0.080
PBDE 116 (2,3,4,5,6-Pentabromodiphenyl ether)	N/A	3.042 \pm 0.067
PBDE 119 (2,3',4,4',6-Pentabromodiphenyl ether)	189084-66-0	4.11 \pm 0.14
PBDE 138 (2,2',3,4,4',5'-Hexabromodiphenyl ether)	182677-30-1	2.522 \pm 0.053
PBDE 139 (2,2',3,4,4',6-Hexabromodiphenyl ether)	N/A	3.514 \pm 0.082
PBDE 153 (2,2',4,4',5,5'-Hexabromodiphenyl ether)	68631-49-2	2.048 \pm 0.068
PBDE 154 (2,2',4,4',5,6'-Hexabromodiphenyl ether)	207122-15-4	1.622 \pm 0.057
PBDE 155 (2,2',4,4',6,6'-Hexabromodiphenyl ether)	35854-94-5	0.895 \pm 0.026
PBDE 156 (2,3,3',4,4',5-Hexabromodiphenyl ether)	N/A	2.77 \pm 0.13
PBDE 173 (2,2',3,3',4,5,6-Heptabromodiphenyl ether)	N/A	
190 (2,3,3',4,4',5,6-Heptabromodiphenyl ether)	189084-68-2	3.01 \pm 0.13
PBDE 181 (2,2',3,4,4',5,6-Heptabromodiphenyl ether)	189084-67-1	2.859 \pm 0.079
PBDE 182 (2,2',3,4,4',5,6'-Heptabromodiphenyl ether)	N/A	1.502 \pm 0.060
PBDE 183 (2,2',3,4,4',5',6-Heptabromodiphenyl ether)	207122-16-5	2.183 \pm 0.049
PBDE 185 (2,2',3,4,5,5',6-Heptabromodiphenyl ether)	N/A	2.37 \pm 0.18
PBDE 191 (2,3,3',4,4',5',6-Heptabromodiphenyl ether)	N/A	2.085 \pm 0.055
PBDE 196 (2,2',3,3',4,4',5,6'-Octabromodiphenyl ether)	N/A	2.46 \pm 0.11
PBDE 197 (2,2',3,3',4,4',6,6'-Octabromodiphenyl ether)	N/A	
204 (2,2',3,4,4',5,6,6'-Octabromodiphenyl ether)	N/A	2.823 \pm 0.070
PBDE 198 (2,2',3,3',4,5,5',6-Octabromodiphenyl ether)	N/A	1.898 \pm 0.057
PBDE 203 (2,2',3,4,4',5,5',6-Octabromodiphenyl ether)	N/A	1.373 \pm 0.034
PBDE 206 (2,2',3,3',4,4',5,5',6-Nonabromodiphenyl ether)	N/A	1.982 \pm 0.063
PBDE 208 (2,2',3,3',4,5,5',6,6'-Nonabromodiphenyl ether)	N/A	1.108 \pm 0.026

^(a) PBDEs are numbered based on the IUPAC numbering scheme. When two congeners coelute under the conditions used, the quantitative results are based on the response of both congeners.

^(b) Chemical Abstracts, Fourteenth Collective Index. Index Guide, American Chemical Society, Columbus, Ohio, 2001. The CAS numbers that are not available are indicated by N/A.

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

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 <http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PUBLICATIONS.cfm>.
- [2] *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 (select “Certificates”), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet <http://www.nist.gov/srm>.