



National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material[®] 2258

BDE 209 in 2,2,4-Trimethylpentane

This Standard Reference Material (SRM) is a solution of decabromodiphenyl ether (BDE 209: Chemical Abstracts Service [CAS] Registry Number 1163-19-5) in 2,2,4-trimethylpentane. SRM 2258 is intended primarily for use in the calibration of instruments and techniques used for the determination of BDE 209. A unit of SRM 2258 consists of five 2 mL ampoules each containing approximately 1.2 mL of solution.

Certified Concentration of BDE 209: The certified concentration value given below is based on results obtained from the gravimetric preparation of the solution and from the analytical results determined 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]. The measurand is the total concentration of BDE 209. Metrological traceability is to the SI derived units for mass fraction expressed as milligrams per kilogram.

BDE 209 Certified Concentration Value: 6.93 mg/kg \pm 0.17 mg/kg

The result is expressed as the certified value \pm the expanded uncertainty. The certified value is the mean 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 [2].

Expiration of Certification: The certification of **SRM 2258** is valid, within the measurement uncertainty specified, until **31 July 2028**, provided the SRM is handled, and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, 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 under the direction of M.M. Schantz and L.C. Sander of the NIST Chemical Sciences 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.

Analytical measurements on the SRM were performed by D.L. Poster of the NIST Materials Measurement Science Division and M.M. Schantz.

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

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Steven J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: The solution contains BDE 209 in 2,2,4-trimethylpentane at the stated concentration. Use proper disposal methods.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures between 10 °C and 30 °C.

Use: Sample aliquots for analysis should be withdrawn **immediately** after opening the ampoules and should be processed without delay for the certified concentration value to be valid within the stated uncertainty. Because of the volatility of 2,2,4-trimethylpentane, the certified concentration value is **NOT** applicable to material stored in ampoules that have been opened for more than 5 min, even if they are resealed.

PREPARATION AND ANALYSIS⁽¹⁾

The solution was prepared at NIST by weighing and mixing known masses of BDE 209 and 2,2,4-trimethylpentane. The solution was mixed overnight (a minimum of 16 h). The total mass of the solution was measured, and the concentration was calculated from this gravimetric procedure. The gravimetric concentration was adjusted for the purity estimation of the BDE 209, 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 mL glass ampoules, which were then flame sealed.

Aliquots from nine ampoules, selected using a random stratified 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). The internal standard added to each sample for quantification purposes was decabromobiphenyl (BB 209). Calibration solutions consisting of weighed amounts of BDE 209 and the internal standard compound 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 used for the GC/NCI-MS analysis was carbon-13 labeled BDE 209.

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 (2000); available at <https://www.nist.gov/sites/default/files/documents/srm/SP260-136.PDF> (accessed Aug 2018).
- [2] 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/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2018); 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 Aug 2018).

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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) 948-3730; e-mail srminfo@nist.gov; or via the Internet at <https://www.nist.gov/srm>.

⁽¹⁾ Certain 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.