National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2720

Sulfur in Di-*n*-Butyl Sulfide

This Standard Reference Material (SRM) is intended for use as an internal standard in X-ray fluorescence spectrometry (XRF) measurements of sulfur in oils and other liquid hydrocarbon matrices. A unit of SRM 2720 consists of five amber ampoules, each containing approximately 4.5 mL of di-*n*-butyl sulfide sealed under an argon atmosphere.

Certified Value: The certified sulfur content is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS) [1]. Homogeneity testing was performed using X-ray fluorescence spectrometry (XRF). 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 [2]. The expanded uncertainty for the certified value for sulfur is calculated as a 95 % confidence interval where $U = ku_c$. The quantity u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO/JCGM and NIST Guides [3]. The coverage factor, $k = 2.26$, corresponds to a t factor obtained from the t-distribution for approximately 9 degrees of freedom.

Certified Value of Sulfur (mass fraction): 21.91 % ± 0.15 %

Expiration of Certification: The certification of this SRM is valid until **31 December 2024**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in the certificate (see "Instructions for Use"). However, the certification will be 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 provided by R.D. Vocke, Jr. of the NIST Chemical Sciences Division, and W.R. Kelly formerly of NIST.

Analytical measurements by ID-TIMS for certification were performed by R.D. Vocke, Jr., J.L. Mann, and W.R. Kelly and homogeneity testing by X-ray fluorescence spectrometry was performed by A.F. Marlow and J.R. Sieber of the NIST Chemical Sciences Division.

Statistical consultation for this SRM was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

Blending and ampouling were performed under the supervision of M.P. Cronise of the NIST Measurement Services Division.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 09 October 2020
See Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR USE

Once an ampoule is opened, it is recommended that the material be used within a period of 8 h to avoid a potential change in the sulfur content. To relate analytical determinations to the certified value in this Certificate of Analysis, a minimum sample mass of 100 mg should be used. The unopened ampoules should be stored under normal laboratory conditions away from direct sunlight.

SOURCE, PREPARATION, AND ANALYSIS[(1)]

SRM 2720 was prepared at NIST by the Meausrement Services Divison by blending and then ampouling the contents of a 19 liter (5 gallon) drum of di-*n*-butyl sulfide (Lot 18656) produced by Penta Manufacturing, West Caldwell, NJ.

REFERENCES

[1] Kelly, W.R.; Paulsen, P.J.; Murphy, K.E.; Vocke, R.D., Jr.; Chen, L.-T.; *Determination of Sulfur in Fossil Fuels by Isotope Dilution Thermal Ionization Mass Spectrometry*; Anal. Chem., Vol. 66, pp. 2505–2513 (1994).

[2] 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; U.S. Government Printing Office: Washington, DC (2000); available at https://www.nist.gov/system/files/documents/srm/SP260-136.PDF (accessed Oct 2020).

[3] 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 (JCGM) (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Oct 2020); 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 2020).

Certificate Revision History: 09 October 2020 (Change of expiration date; editorial changes); 09 September 2008 (Editorial changes); 08 May 2007 (Original certificate date).

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

[(1)]Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify 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.