



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 5 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 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 % \pm 0.15 %

Expiration of Certification: The certification of this SRM is valid until **31 December 2020**, 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: This material is considered to be stable during the period of certification. NIST will monitor this material and will report any significant changes in certification to the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification of this SRM was provided by R.D. Vocke, Jr. and W.R. Kelly of the NIST Analytical Chemistry Division.

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 Analytical Chemistry 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.

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

Stephen A. Wise, Chief
Analytical Chemistry Division

Robert L. Watters, Jr., Chief
Measurement Services Division

Gaithersburg, MD 20899
Certificate Issue Date: 09 September 2008
See Certificate Revision History on Last Page

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 ANALYSIS¹

SRM 2720 was prepared at NIST by the Measurement Services Division 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.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, p. 16; U.S. Government Printing Office: Washington, DC (2000).
- [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: 09 September 2008 (Editorial changes); 08 May 2007 (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 at <http://www.nist.gov/srm>.

¹Certain 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.