National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 2716

Sulfur in Gasoline

(< 1 mg/kg)

This Standard Reference Material (SRM) is intended for use in the evaluation of methods, calibration of instruments, and preparation of calibration mixes [1,2] used in the determination of total sulfur in gasoline or materials of a similar matrix. SRM 2716 is a blend of volume fractions of 91 % iso-octane and 9 % *n*-heptane. A unit of SRM 2716 consists of five amber ampoules each containing approximately 20 mL of gasoline.

Certified Values and Uncertainties: The certified value for the sulfur mass fraction is given in Table 1. 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 taken into account [3]. A certified value is the present best estimate of the true value based on the results of isotope dilution thermal ionization mass spectrometry analyses performed at NIST. The uncertainty in the certified value is calculated according to the ISO Guide [4] and is expressed as an expanded uncertainty, $U = ku_c$, where k is a coverage factor and u_c is the combined standard uncertainty of the certified value. The value of $k = 2.306$ based on a Student's t-distribution with a nominal confidence level of 95 % and 8 degrees of freedom. The calculated value of $u_c = 0.131$ mg/kg.

Table 1. Certified Value (Mass Fraction)

Sulfur: 0.44 mg/kg ± 0.30 mg/kg

Information Values: An information value for the density at 23 °C determined by a calibrated pycnometer is provided in Table 2. A NIST information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [3].

Table 2. Information Value

Density: 0.689 01 g/mL

Expiration of Certification: The certification of **SRM 2716** is valid, within the measurement uncertainty specified, until **01 January 2015**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for 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) will facilitate notification.

The coordination of the technical measurements leading to the certification of this SRM was provided by W.R. Kelly and G.C. Turk of the NIST Analytical Chemistry Division.

Analyses were performed by W.R. Kelly, J.L. Mann, J.R Moody, and R.D. Vocke, Jr. of the NIST Analytical Chemistry Division.

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

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 14 June 2011

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

Support aspects involved with the issuance of this SRM were coordinated through the NIST Measurement Services Division.

INSTRUCTIONS FOR STORAGE AND USE

Storage: The SRM should be stored at temperatures between 10 °C and 30 °C. It should not be exposed to intense sources of radiation, including ultraviolet light or sunlight.

Use: The ampoules in the SRM unit should be opened only at the time of use. No attempt should be made to store the material in opened ampoules for future use. To relate analytical determinations to the certified value in this certificate, a minimum sample mass of 200 mg is recommended.

COLLECTION, PREPARATION, AND ANALYSIS[(1)]

Source and Preparation of Material: The material for SRM 2716 was acquired from Phillips Chemical Company, Bartlesville, OK, and is a mixture of volume fractions of 91 % (0.899 mole fraction) 2,2,4-trimethlypentane (iso-octane) and 9 % *n*-heptane giving it a nominal octane rating of 91. The ampouling of this SRM was performed by Aqua Air Corporation in Charlotte, NC.

Analytical Methods: The certified sulfur value listed in Table 1 was obtained by isotope dilution thermal ionization mass spectrometry using a highly enriched (atom fraction of 99.82 %) ^{34}S spike and Carius tube digestion [5]. Data reduction was by the method of least squares [6]. The information value for density listed in Table 2 was based on determinations using a pycnometer calibrated against distilled water.

Homogeneity: This material is a clear colorless liquid that is expected to be homogenous due to its chemical form and nature. The sulfur content of this material is low enough that assessment of homogeneity for sulfur is made difficult by measurement variation caused primarily by blank control. The data collected for analysis of this material does not indicate any evidence of heterogeneity.

REFERENCES

[1] Kelly, W.R.; MacDonald, B.S; Leigh S.D.; *A Method for the Preparation of NIST Traceable Fossil Fuel Standards with Concentrations Intermediate to SRM Values*; J. ASTM International, Vol. 4, No. 2 (2007).

[2] Kelly, W.R.; MacDonald, B.S.; Leigh, S.D.; *A Method for the Preparation of NIST Traceable Fossil Fuel Standards with Concentrations Intermediate to SRM Values (DVD)*; NIST Special Publication SP260-167; free and available at: http://www.nist.gov/srm/publications.cfm (accessed June 2011).

[3] 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://www.nist.gov/srm/publications.cfm (accessed June 2011).

[4] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed June 2011); 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://www.nist.gov/pml/pubs/index.cfm (accessed June 2011).

[5] Paulsen, P.J.; Kelly, W.R.; *The Determination of Sulfur as Arsenic Monosulfide Ion by Isotope Dilution Thermal Ionization Mass Spectrometry*; Anal. Chem. Vol. 56, pp. 708-713 (1984).

[6] Guthrie, W.F.; Vocke, R.D.; Mann, J.L.; Kelly, W.R.; *Chemical Blank Corrections Using a Linear Regression Approach*; Abstracts of Papers of the American Chemical Society, Vol. 230, U423 (2005).

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) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

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