



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1619b

Sulfur in Residual Fuel Oil (0.7 %)

This Standard Reference Material (SRM) is intended for use in the calibration of instruments and the evaluation of methods used in the determination of total sulfur and mercury in fuel oils or materials of similar matrix. A unit of SRM 1619b consists of 100 mL of commercial “No. 6” residual fuel oil as defined by ASTM D396-95 Standard Specification for Fuel Oils [1].

Table 1. Certified Mass Fraction Values

Sulfur	0.6960 %	±	0.0077 %
Mercury	3.46 ng/g	±	0.74 ng/g

Certified Mass Fraction Values: The certified sulfur and mercury mass fraction values are provided in Table 1 [2]. 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]. The certified sulfur content value is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS) [4]. The certified mercury content is based in cold vapor isotope dilution inductively coupled plasma mass spectrometry (CV-ID-ICP-MS) [5]. Homogeneity testing was performed using X-ray fluorescence (XRF) spectrometry. The uncertainty in the certified value is expressed as an expanded uncertainty and is calculated according to the method in the ISO/JCGM Guide [6]. The expanded uncertainty is based on a 95 % prediction interval [7].

Expiration of Certification: The certification of **SRM 1619b** is valid, within the measurement uncertainty specified, until **01 July 2021**, provided the SRM is handled and stored in accordance with 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 expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of the technical measurements leading to certification of this SRM were performed by J.D. Fassett and G.C. Turk of the NIST Chemical Sciences Division.

Analytical measurements were performed by W.R. Kelly, R.D. Vocke, Jr., S.E. Long, A.F. Marlow, J.R. Sieber, and J.L. Mann of the NIST Chemical Sciences Division.

Statistical consultation for this SRM was provided by K.R. Eberhardt of the NIST Statistical Engineering Division.

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

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INSTRUCTIONS FOR HANDLING, STORAGE AND USE

Because of the viscosity of SRM 1619b, it is recommended that the SRM unit be warmed slowly to between 40 °C and 60 °C and then shaken, or stirred with a clean stirrer, before sampling. Care must be exercised to not introduce entrapped air, which could affect gravimetric measurements and XRF responses. A detailed study to determine if the sulfur components of SRM 1619b will segregate has not been performed at this time.

The SRM bottle should only be opened for the minimum time required to dispense the material. To relate analytical determinations to the certified value in this Certificate of Analysis, a minimum sample mass of 140 mg should be used. After use, the bottle should be tightly recapped and stored under normal laboratory conditions away from direct sunlight.

SUPPLEMENTAL INFORMATION

Additional properties of SRM 1619b are listed below. These properties were determined by a commercial firm under contract to NIST using ASTM methods. The results are **NOT** certified and are provided only as additional information on the matrix.

Physical Property Test	ASTM Standard Used	Result
Density @ 15 °C @ 60 °F	D1250-80 (1990) D287-92 (1995)	1010.1 kg/m ³ 8.5 °API
Flash Point, PMCC	D93-94	93 °C
Pour Point	D97-93	0 °C
Heat of Combustion, Gross	D240-92 ^{C1}	41.74 MJ/kg (17947 Btu/lb)
Kinematic Viscosity @ 40 °C	D445-94 ^{C1}	322.9 × 10 ⁻⁶ m ² /s (322.9 cSt)
@ 50 °C	D445-94 ^{C1}	151.1 × 10 ⁻⁶ m ² /s (151.1 cSt)
@ 100 °C	D445-94 ^{C1}	16.17 × 10 ⁻⁶ m ² /s (16.17 cSt)
Carbon	D5291-92	88.0 %
Hydrogen	D5291-92	10.0 %

ASTM Standard Test Methods

D93-94	Standard Test Methods for Flash Point by Pensky-Martens Closed Tester
D97-93	Standard Test Methods for Pour Point of Petroleum Products
D240-92 ^{C1}	Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter
D287-92 (1995)	Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)
D445-94 ^{C1}	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
D1250-80 (1990) ^{C1}	Standard Guide for Petroleum Measurement Tables
D5291-92	Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants

^{C1} Indicates that only editorial changes were made to the previous issuance of the ASTM standard.

REFERENCES

- [1] ASTM D396-95, *Standard Specification for Fuel Oils*; Annu. Book ASTM Stand., Vol. 05.01, West Conshohocken, PA.
- [2] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at <http://www.nist.gov/pml/pubs/index.cfm/> (accessed June 2013).
- [3] 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 <http://www.nist.gov/srm/publications.cfm> (accessed June 2013).
- [4] 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).
- [5] Christopher, S.J.; Long, S.E.; Rearick, M.S.; *Development of High Accuracy Vapor Generation ICP-MS and Its Application to the Certification of Mercury in Standard Reference Materials*; Anal. Chem., Vol. 73, pp. 2190–2199 (2001).
- [6] 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 http://www.bipm.org/utls/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed June 2013); 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 2013).
- [7] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: NY (1991).

Certificate Revision History: 28 June 2013 (Certification period extended; editorial changes); 24 February 2010 (Corrected the unit for the certified value of mercury from ng/kg to ng/g; editorial changes); 22 July 2008 (Editorial changes); 28 January 2008 (Certification period extended; editorial changes); 16 May 2006 (Editorial changes); 01 March 2006 (Editorial changes); 24 January 2006 (Editorial changes); 29 November 2004 (Certified mercury value added); 27 July 1998 (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 at: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.