



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1623c

Sulfur in Residual Fuel Oil (0.3%)

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 in fuel oils or materials of similar matrix. The certified sulfur content is based on analyses using isotope dilution thermal ionization mass spectrometry (ID-TIMS) [1]. Homogeneity testing was performed using X-ray fluorescence (XRF) spectrometry and no significant bottle to bottle variability was observed. The certified value for the sulfur content, reported as a mass fraction expressed in percent, is given below. A unit of SRM 1623c consists of 100 mL of commercial “No. 4 (light)” residual fuel oil as defined by ASTM D 396-95 Standard Specification for Fuel Oils [2], in an amber bottle.

Certified Sulfur Content: 0.3806 % $\pm$ 0.0024 %

The uncertainty in the certified value is calculated as

$$U = ku_c$$

where u_c is the combined standard uncertainty calculated according to the ISO Guide [3] and k is a coverage factor. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with material inhomogeneity and ID-TIMS measurement uncertainty. In the absence of Type B uncertainties (which are negligible here in comparison with Type A), the expanded uncertainty (U) given is for a 95 % prediction interval. The coverage factor, $k = 2.31$, is Student's t -value for a 95 % prediction interval with eight degrees of freedom.

Expiration of Certification: The certification of **SRM 1623c** is valid, within the measurement uncertainty specified, until **13 June 2015**, provided the SRM is handled and stored in accordance with the instructions given in the certificate (see “Instructions for Handling, Storage, and Use”). However, the certification will be nullified if the SRM is damaged, contaminated, or 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 overall direction and coordination of the technical measurements leading to certification of this SRM were performed by W.R. Kelly, R.D. Vocke, and R.L. Watters, Jr. of the NIST Analytical Chemistry Division.

Analytical measurements were performed by W.R. Kelly, R.D. Vocke, A.F. Marlow, P.A. Pella, and J.R. Sieber of the NIST Analytical Chemistry Division.

Statistical calculations for this SRM were carried out by S.D. Leigh of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM was coordinated through the NIST Measurement Services Division.

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Gaithersburg, MD 20899
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INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

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

SUPPLEMENTAL INFORMATION

The physical properties of SRM 1623c are listed in the table below. These properties were determined using ASTM methods by a commercial firm under contract to NIST. The results are **NOT** certified and are provided as additional information on the matrix.

Physical Property Test	ASTM Standard	Result
Density @ 15 °C	D 1298-85 (1990)	899.6 kg/m ³
@ 60 °F	D 1250-80 (1990)	25.7 API
Flash Point, PMCC	D 93-94	52 °C
Pour Point	D 97-93	21 °C
Heat of Combustion, Gross	D 240-92 ^{e1}	43.98 MJ • kg ⁻¹ (18 907 Btu • lb ⁻¹)
Kinematic Viscosity @ 40 °C	D 445-94 ^{e1}	5.015 • 10 ⁻⁶ m ² /s (5.015 cSt)
@ 50 °C	D 445-94 ^{e1}	4.002 • 10 ⁻⁶ m ² /s (4.002 cSt)
@ 100 °C	D 445-94 ^{e1}	1.801 • 10 ⁻⁶ m ² /s (1.801 cSt)
Carbon	D 5291-92	87.8 %
Hydrogen	D 5291-92	11.7 %

ASTM Standards Used

D 1298-85 (1990) ^{e1}	Standard Practice for Density, Relative Density (Specific Gravity) or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D 1250-80 (1990) ^{e1}	Standard Guide for Petroleum Measurement Tables
D 93-94	Standard Test Methods for Flash Point by Pensky-Martens Closed Tester
D 97-93	Standard Test Methods for Pour Point of Petroleum Products
D 240-92 ^{e1}	Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter
D 445-94 ^{e1}	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
D 5291-92	Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants

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] ASTM D396-95; *Standard Specification for Fuel Oils*; Annual Book of Standards, Vol. 05.01, West Conshohocken, PA.
- [3] 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 (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2012); 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/tn1297/index.cfm> (accessed Apr 2012).

Certificate Revision History: 17 April 2012 (Extension of the certification period; editorial changes); 01 March 2006 (Editorial change); 05 May 2004 (This revision reflects an extension of the certification date); 13 June 2000 (This revision reflects a change in the certification expiration date); 30 July 1996 (Original certificate date).
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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) 975-6776; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.