National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1622e

Sulfur in Residual Fuel Oil
(Nominal Mass Fraction 2 %)

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. A unit of SRM 1622e consists of 100 mL of commercial No. 6 residual fuel oil as defined by ASTM D396-95 Standard Specification for Fuel Oils [1].

Certified Value: The certified sulfur content reported in Table 1 is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS) [2]. Homogeneity testing was performed using X-ray fluorescence (XRF) spectrometry. No significant bottle to bottle variability was observed.

Table 1. Certified Value

Certified Value of Sulfur (mass fraction): 2.1468 % ± 0.0041 %

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/JCGM and NIST Guides [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) is given for a 95 % confidence interval. The coverage factor, k = 2.36, is the Student's t-value for a 95 % confidence interval with seven degrees of freedom. The measurand is the total mass fraction of sulfur. The certified value is metrologically traceable to the SI derived unit of mass fraction (expressed as a percent).

Expiration of Certification: The certification of **SRM 1622e** is valid, within the uncertainty specified, until **31 August 2025**, 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 modified.

Maintenance of 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 certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of the technical measurements leading to certification of this SRM were performed by G.C. Turk of the NIST Chemical Sciences Division.

Analytical measurements were performed by W.R. Kelly, R.D. Vocke, A.F. Marlow, P.A. Pella, and T.L. Quinn 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 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: 05 October 2015
Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
Office of Reference Materials

INSTRUCTIONS FOR 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 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

The additional properties of SRM 1622e are listed in Table 2 below. These properties were determined by a commercial firm under contract to NIST using ASTM methods. The results are **NOT** certified and are provided as additional information on the matrix.

Table 2. Supplemental Information

Physical Property Test	ASTM Standard Used	Result
Density @ 15 °C	D1250-80 (1990)	1005.1 kg/m^3
@ 60 ° F	D287-92 (1995)	9.2°API
Flash Point, PMCC	D93-94	77 °C
Pour Point	D97-93	14 °C
Heat of Combustion, Gross	D240-92^{C1}	42.49 MJ/kg (18 268 Btu/lb)
Kinematic Viscosity @ 40 °C	D445-94^{C1}	1763 × 10^{-6} m^2/s (1763 cSt)
@ 50 °C	D445-94^{C1}	776.9 × 10^{-6} m^2/s (776.9 cSt)
@ 100 °C	D445-94^{C1}	54.7 × 10^{-6} m^2/s (54.74 cSt)
Carbon	D5291-92	86.5 %
Hydrogen	D5291-92	10.1 %

ASTM Standards

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

REFERENCES

[1] ASTM D396-95, *Standard Specification for Fuel Oils*; Annu. Book ASTM Stand.; Vol. 05.01, West Conshohocken, PA.

[2] 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).

[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 (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2015); 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/guidelines/TN1297/tn1297s.pdf (accessed Sep 2015).

Certificate Revision History: 05 October 2015 (Change of expiration date; editorial changes); 20 February 2014 (Correction of density result at 15°C; editorial changes); 16 May 2006 (Editorial changes); 01 March 2006 (Editorial changes); 02 April 2003 (This revision reflects an extension in the expiration date); 10 April 1997 (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 http://www.nist.gov/srm.