



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 1621e

#### Sulfur in Residual Fuel Oil (1 %)

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 by 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 1621e consists of 100 mL of commercial “No. 6” residual fuel oil as defined by ASTM D 396-97 Standard Specification for Fuel Oils [2].

Sulfur Content: 0.9480 %  $\pm$  0.0057 %

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 the Student's  $t$ -value for a 95 % prediction interval with eight degrees of freedom.

**Expiration of Certification:** The certification of **SRM 1621e** is valid, within the measurement uncertainty specified, until **01 July 2016**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see “Instructions for 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 overall direction and coordination of the technical measurements leading to certification of this SRM were performed by W.R. Kelly, R.D. Vocke, A.F. Marlow, P.A. Pella, and R.L. Watters, Jr. 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 were coordinated through the NIST Measurement Services Division.

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*See Certificate Revision History on Last Page*

## 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 capped and stored under normal laboratory conditions away from direct sunlight.

## SUPPLEMENTAL INFORMATION

The physical properties of SRM 1621e 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 1250-80 (1990)	1005.1 kg/m <sup>3</sup>
@ 60 °F	D 287-92 (1995)	9.2 API
Flash Point, PMCC	D 93-94	62 °C
Pour Point	D 97-93	1.7 °C
Heat of Combustion, Gross	D 240-92 <sup>e1</sup>	42.37 MJ•kg <sup>-1</sup> (18 214 Btu•lb <sup>-1</sup> )
Kinematic Viscosity @ 40 °C	D 445-94 <sup>e1</sup>	80.76 × 10 <sup>-5</sup> m <sup>2</sup> /s (807.6 cSt)
@ 50 °C	D 445-94 <sup>e1</sup>	37.07 × 10 <sup>-5</sup> m <sup>2</sup> /s (370.7 cSt)
@ 100 °C	D 445-94 <sup>e1</sup>	3.31 × 10 <sup>-5</sup> m <sup>2</sup> /s (33.10 cSt)
Carbon	D 5291-92	88.1 %
Hydrogen	D 5291-92	10.3 %

### ASTM Standards Used

D 287-92 (1995)	Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method).
D 1250-80 (1990) <sup>e1</sup>	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 <sup>e1</sup>	Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter.
D 445-94 <sup>e1</sup>	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.

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<sup>e1</sup> indicates that only editorial changes were made to the previous issuance of the ASTM standard.

## 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 D 396-97, *Standard Specification for Fuel Oils*, Annual Book of ASTM Standards, Vol. 05.01 (1996).
- [3] JCGM 100:2008; *Evaluation of Measurement Data – Guide to the Expression of 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](http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf) (accessed Sep 2010); 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/physlab/pubs/index.cfm> (accessed Sep 2010).

**Certificate Revision History:** **16 September 2010** (Extension of the certification period; editorial changes); **01 March 2006** (Editorial changes); **16 February 2006** (Update of expiration date and editorial changes); **09 September 1999** (Update expiration date); **30 July 1996** (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) 926-4751; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*