



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1621e

Sulfur in Residual Fuel Oil

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 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 consists of 100 mL of commercial "No. 6" residual fuel oil as defined by ASTM D 396-95 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 this SRM is valid until **01 July 2006**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in the certificate (see Use and Handling). However, the certification will be nullified if the SRM is damaged, contaminated, or modified.

Stability: This material is considered to be stable during the period of certification. The National Institute of Standards and Technology (NIST) will monitor this material and will report any significant changes in certification to the purchaser. Return of the attached registration card will facilitate notification.

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

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

Willie E. May, Chief
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Gaithersburg, MD 20899
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See Certificate Revision History on Last Page

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.

Use and Handling: 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 ³
@ 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 ^{e1}	42.37 MJ·kg ⁻¹ (18 214 Btu·lb ⁻¹)
Kinematic Viscosity @ 40 °C	D 445-94 ^{e1}	80.76 × 10 ⁻⁵ m ² /s (807.6 cSt)
@ 50 °C	D 445-94 ^{e1}	37.07 × 10 ⁻⁵ m ² /s (370.7 cSt)
@ 100 °C	D 445-94 ^{e1}	3.31 × 10 ⁻⁵ m ² /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) ^{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

^{e1} 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., and Chen, L.-T., "Determination of Sulfur in Fossil Fuels by Isotope Dilution-Thermal Ionization Mass Spectrometry," *Anal. Chem.* **66**, pp. 2505-2513, (1994).
- [2] ASTM D 396-95, "Standard Specification for Fuel Oils," **05.01**, Annual Book of ASTM Standards, West Conshohocken, PA, (1996).
- [3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st ed., ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and 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/>).

Certificate Revision History: 9 Sept 99 (update expiration date); 30 Jul 96 (original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet <http://ts.nist.gov/srm>.