



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1624c

Sulfur in Distillate 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. A unit of SRM 1624c consists of 100 mL of commercial "No. 2" fuel oil as defined by ASTM D396-95 Standard Specification for Fuel Oils [1].

Certified Value of Sulfur (mass fraction, in %): 0.3970 % \pm 0.0040 %

The certified sulfur content is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS) [2]. Homogeneity testing was performed using x-ray fluorescence spectrometry.

The uncertainty in the certified value is expressed as an expanded uncertainty, U , at the 95 % level of confidence, and is calculated according to the method in the ISO Guide to the Expression of Uncertainty in Measurement [3]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is the combined standard uncertainty 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 variability and ID-TIMS measurement uncertainty. The expanded uncertainty (U) is based on a 95 % prediction interval [4]. The coverage factor, $k = 2.57$, is the Student's t -value for a 95 % prediction interval with 5 degrees of freedom.

Expiration of Certification: The certification of this SRM is valid until July 1, 2003, 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. 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.

The overall direction and coordination of the technical measurements leading to certification of this SRM were performed by G.C. Turk of the NIST Analytical Chemistry 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 Analytical Chemistry Division.

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

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

Gaithersburg, MD 20899
Certificate Issue Date: June 6, 1997

Thomas E. Gills, Chief
Standard Reference Materials Program

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 150 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 1624c 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 as additional information on the matrix.

Physical Property Test	ASTM Standard Used	Result
Density @ 15 °C	D 1250-80 (1990)	860.2 kg/m ³
@ 60 ° F	D 287-92 (1995)	32.9 API
Flash Point, PMCC	D 93-94	62 °C
Pour Point	D 97-93	-18 °C
Heat of Combustion, Gross	D 240-92 ^{e1}	44.86 MJ/kg (19 285 Btu/lb)
Kinematic Viscosity @ 40 °C	D 445-94 ^{e1}	2.502 · 10 ⁻⁶ m ² /s (2.502 cSt)
Carbon	D 5291-92	85.4 %
Hydrogen	D 5291-92	12.6 %

ASTM Standards

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 287-92 (1995)	Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)
D 445-94 ^{e1}	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
D 1250-80 (1990) ^{e1}	Standard Guide for Petroleum Measurement Tables
D 5291-92	Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants

REFERENCES

- [1] ASTM D 396-95, 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., and Chen, L.-T., Determination of Sulfur in Fossil Fuels by Isotope Dilution Thermal Ionization Mass Spectrometry, *Anal. Chem.* **66**, pp. 2505-2513, (1994).
- [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).
- [4] Hahn, G.J., and Meeker, W.Q., "Statistical Intervals: A Guide for Practitioners," John Wiley & Sons, Inc., New York, (1991).