



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2723a

Sulfur in Diesel Fuel Oil

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and the calibration of instruments used in the determination of total sulfur in fuel oils or materials of similar matrix. SRM 2723a is a commercial "No. 2-D" distillate fuel oil as defined by ASTM D 975-97 *Standard Specification for Diesel Fuel Oils* [1]. A unit of SRM 2723a consists of 10 amber ampoules, each containing approximately 10 mL of diesel fuel sealed under an argon atmosphere.

Certified Value: The certified sulfur content provided 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 spectrometry. The expanded uncertainty for the certified value for sulfur is calculated as a 95 % confidence interval where $U = ku_c$. The quantity u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO Guide [3]. The coverage factor, k , is determined from the Student's t -distribution for the approximate degrees of freedom and has a value of 4.30.

Table 1. Certified Value (mass fraction)

Sulfur: 11.0 mg/kg \pm 1.1 mg/kg

Information Values: Information values are provided in Table 2 for additional properties of SRM 2723a. The results are not certified and no uncertainty is reported. The results are given to provide additional information on the matrix.

Expiration of Certification: The certification of this SRM is valid until **31 December 2015**, within the uncertainty specified, 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 otherwise modified.

Maintenance of SRM Certification: 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, J.L. Mann, A.F. Marlow, J.R. Sieber, and R.D. Vocke of the NIST Analytical Chemistry Division.

Statistical consultation for this SRM was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

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See Certificate Revision History on Last Page

Blending and ampouling were performed under the supervision of M.P. Cronise of the NIST Measurement Services 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 of the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

Each SRM ampoule should only be opened for the minimum time required to dispense the material. **Once an ampoule is opened, the material must be used within a period of 8 h to avoid a significant change in the sulfur content.** To relate analytical determinations to the certified value in this Certificate of Analysis, a minimum sample mass of 150 mg should be used. The unopened ampoules should be stored under normal laboratory conditions away from direct sunlight.

Table 2. Information Values for Selected Properties

Physical Property Test ^a	ASTM Standard Used	Result
Density @ 15 °C @ 60 °F	D 1250-80 (1990) ^{e1} D 4052-96	818.0 kg/m ³ 41.4 API
Flash Point	D 93 (A)-94	88.9 °C
Kinematic Viscosity @ 40 °C	D 445-94 ^{e1}	3.176 × 10 ⁻⁶ m ² /s (3.176 cSt)
Oxidation Stability	D 2274-94	<0.1 mg/100 mL Filterable 0.1 mg/100 mL Adherent 0.1 mg/100 mL Total
Carbon	D 5291-92	84.8 %
Hydrogen	D 5291-92	14.1 %

^a 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.

ASTM Standards

D 93-94	Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
D 4052-96	Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter
D 445-94 ^{e1}	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)
D 1250-80 (1990) ^{e1}	Standard Guide for Petroleum Measurement Tables
D 2274-94	Standard Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
D 5291-92	Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants

REFERENCES

- [1] ASTM D 975-97, *Standard Specification for Diesel Fuel Oils*; Annual Book of ASTM Standards, Vol. 05.01, West Conshohocken, PA (1998).
- [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] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland (1993); 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/>.

Certificate Revision History: 12 February 2004 (Editorial changes about ampouling under an argon atmosphere); 08 December 2003 (Original certificate date).
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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; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.