



# Certificate of Analysis

## Standard Reference Material<sup>®</sup> 1624d

### 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 a similar matrix. SRM 1624d 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 1624d 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-TI-MS) [2]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [3]. The expanded uncertainty,  $U$ , of the certified value for the sulfur concentration was computed using the formula  $U = ku_c$ , where  $u_c$  is the combined standard uncertainty of the certified value and  $k$  is a coverage factor used to control the confidence level associated with the expanded uncertainty interval. The combined standard uncertainty,  $u_c = 9.4$  mg/kg, was computed using the methods outlined in the ISO and NIST guides [4] and accounts for random measurement uncertainty, fluctuations in chemical blank, instrument calibration, and certification of the calibrant. Homogeneity of this material was better than 0.76 % relative as determined using X-ray fluorescence spectrometry (XRF). The coverage factor,  $k = 2.12$ , was obtained from a Student's  $t$ -distribution with 16.1 degrees of freedom and a confidence level of 95 %.

Table 1. Certified Value (mass fraction)

Sulfur: 3882 mg/kg  $\pm$  20 mg/kg

**Information Values:** Information values are provided in Table 2 for additional properties of SRM 1624d. The values are not certified and are given to provide additional information on the matrix, but insufficient information is available to adequately assess the uncertainties associated with the values [3].

**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. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification of this SRM was provided by W.R. Kelly and G.C. Turk of the NIST Analytical Chemistry Division.

Analytical measurements by ID-TIMS for certification were performed by W.R. Kelly, J.L. Mann, and R.D. Vocke and homogeneity testing by X-ray fluorescence spectrometry was performed by A.F. Marlow and J.R. Sieber of the NIST Analytical Chemistry Division.

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

Ampouling was performed under the supervision of M.P. Cronise of the NIST Measurement Services Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

The diesel fuel for this SRM was donated by Haltermann Products, Channel View, TX.<sup>1</sup>

## INSTRUCTIONS FOR USE

Each SRM ampoule should only be opened for the minimum time required to dispense the material. Once an ampoule is opened, it is recommended that the material be used within a period of 8 h to avoid a potential 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 <sup>(a)</sup>	ASTM Standard Used	Result
Density @ 15 °C	D 1250-80 (1990) <sup>e1</sup>	849.9 kg/m <sup>3</sup>
@ 60 °F	D 4052-96	34.9 API
Flash Point	D 93 (A)-94	73.3 °C
Kinematic Viscosity @ 40 °C	D 445-94 <sup>e1</sup>	3.239 × 10 <sup>-6</sup> m <sup>2</sup> /s (3.239 cSt)
Oxidation Stability	D 2274-94	0.1 mg/100 mL Filterable 0.1 mg/100 mL Adherent 0.2 mg/100 mL Total
Carbon	D 5291-92	86.5 %
Hydrogen	D 5291-92	13.3 %

<sup>(a)</sup> 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 <sup>e1</sup>	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)
D 1250-80 (1990) <sup>e1</sup>	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

<sup>1</sup> Certain commercial organizations, services, equipment, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the organizations, services, materials or equipment identified are necessarily the best available for the purpose.

## 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] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, p. 16, U.S. Government Printing Office: Washington, DC (2000).
- [4] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: 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/>.

*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](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*