



Certificate of Analysis

Standard Reference Material[®] 1624d

Sulfur in Diesel Fuel Oil (Nominal Mass Fraction 0.4 %)

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 mass fraction value for sulfur is provide below. 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 taken into account [2]. The certified value is based on measurements by isotope dilution thermal ionization mass spectrometry (ID-TI-MS) [3].

Certified Mass Fraction Value for Sulfur: 3882 mg/kg \pm 20 mg/kg

The uncertainty associated with each certified value is an expanded uncertainty, U , and was evaluated in accordance with the ISO/JCGM Guides [4]. The expanded uncertainty is calculated as $U = ku_c$, where $u_c = 9.4$ mg/kg and is intended to represent random measurement uncertainty, fluctuations in chemical blank, instrument calibration, and certification of the calibrant. 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 %. The measurand is the sulfur content. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram).

Information Values: Information values for selected properties in SRM 1624d are provided in Table 1. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [2]. Information values may not be used to assess metrological traceability.

Expiration of Certification: The certification of **SRM 1624b** is valid, within the measurement uncertainty specified, until **31 January 2022**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Storage, Handling, and 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 or register) will facilitate notification.

Coordination of technical measurements for the certification of this SRM was performed by W.R. Kelly and G.C. Turk formerly of the NIST Chemical Sciences Division.

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

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
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Certificate Revision History on Last Page

Steven J. Choquette, Acting Director
Office of Reference Materials

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR STORAGE, HANDLING, AND USE

Each SRM bottle 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 SRM should be stored under normal laboratory conditions away from direct sunlight.

SOURCE MATERIAL AND ANALYSIS⁽¹⁾

Source Material: The diesel fuel for SRM 1624d was donated by Haltermann Products (Channel View, TX).

Homogeneity Assessment: Homogeneity of this material was better than 0.76 % relative as determined using X-ray fluorescence spectrometry (XRF).

SUPPLEMENTAL INFORMATION

The physical property values given below are NOT certified but are provided as additional information on the diesel fuel matrix.

Table 1. Information Values for Selected Properties of SRM 1624d

Physical Property Test	ASTM Method	Value
Density @ 15 °C	D 1250-80 (1990) ^{E1}	849.9 kg/m ³
@ 60 °F	D 4052-96	34.9 API
Flash Point	D 93 (A)-94	73.3 °C
Kinematic Viscosity @ 40 °C	D 445-94 ^{E1}	3.239 × 10 ⁻⁶ m ² /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 %

ASTM METHODS USED FOR PHYSICAL TESTS

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

⁽¹⁾ Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify 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 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] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at www.nist.gov/srm/publications.cfm (accessed Apr 2016).
- [3] 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).
- [4] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utls/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2016); 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/pml/pubs/index.cfm> (accessed Apr 2016).

Certificate Revision History: 04 April 2016 (Change of expiration date;editorial changes); 24 February 2006 (Editorial changes); 26 April 2005 (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) 948-3730; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.