



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 2724b

#### 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 and mercury in fuel oils or materials of similar matrix. SRM 2724b is a commercial "No. 2-D" distillate fuel oil as defined by ASTM D 975-96a Standard Specification for Diesel Fuel Oils [1]. A unit of SRM 2724b consists of 10 amber ampoules, each containing approximately 10 mL of diesel fuel.

**Certified Values:** The certified sulfur and mercury values are provided in Table 1. The certified sulfur content is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS) [2]. The certified mercury content is based in cold vapor isotope dilution inductively coupled plasma mass spectrometry (CV-ID-ICP-MS) [3]. Homogeneity testing was performed using X-ray fluorescence spectrometry. The uncertainty in the certified values is expressed as an expanded uncertainty and is calculated according to the method in the NIST/ISO Guide [4]. The expanded uncertainty is based on a 95 % prediction interval.

Table 1. Certified Values (Mass Fraction)

Sulfur:	0.04265 %	± 0.00057 %
Mercury:	0.034 ng/kg	± 0.026 ng/kg

**Information Values:** Information values are provided in Table 2 for additional properties of SRM 2724b. 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 2008**, 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 overall direction and coordination of the technical measurements leading to certification of this SRM were performed by J.D. Fassett and G.C. Turk of the NIST Analytical Chemistry Division.

Analytical measurements were performed by W.R. Kelly, R.D. Vocke, S.E. Long, A.F. Marlow, J.R. Sieber, and J.L. Mann of the NIST Analytical Chemistry Division.

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

Willie E. May, Chief  
Analytical Chemistry Division

Robert L. Watters, Jr., Chief  
Measurement Services Division

Gaithersburg, MD 20899  
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*See Certificate Revision History on Last Page*

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 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*	ASTM Standard Used	Result
Density @ 15 °C	D 1250-80 (1990)	822.3 kg/m <sup>3</sup>
@ 60 °F	D 287-92 (1995)	40.5 API
Flash Point	D 93-94	73.0 °C
Pour Point	D 97-93	- 18.0 °C
Heat of Combustion, Gross	D 240-92 <sup>e1</sup>	45.99 MJ/kg (19 771 Btu/lb)
Kinematic Viscosity @ 40 °C	D 445-94 <sup>e1</sup>	2.344 × 10 <sup>-6</sup> m <sup>2</sup> /s (2.344 cSt)
Color	D 1500-96	L 0.5
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	85.5 %
Hydrogen	D 5291-92	11.9 %

\* 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 Standard Test Methods

D 93-94	Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
D 97-93	Standard Test Method for Pour Point of Petroleum Products
D 240-92 <sup>e1</sup>	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 <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 1500-96	Standard Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
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>e1</sup>Indicates that only editorial changes were made to the previous issuance of the ASTM standard.

## REFERENCES

- [1] ASTM D 975-96a *Standard Specification for Diesel Fuel Oils*; Annual Book of ASTM Standards, Vol. 05.01, West Conshohocken, PA (1997).
- [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] Christopher, S.J.; Long, S.E.; Rearick, M.S.; *Development of High Accuracy Vapor Generation ICP-MS and its Application to the Certification of Mercury in Standard Reference Materials*; Anal. Chem., Vol. 73, pp. 2190-2199 (2001).
- [4] *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/>.

<b>Certificate Revision Date History:</b> 29 November 2004 (This revision reflects the addition of a certified mercury value); 28 March 2001 (Certified sulfur value and uncertainty revised); 09 September 1999 (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](mailto:srminfo@nist.gov); or via the Internet <http://www.nist.gov/srm>.