National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2723a

Sulfur in Diesel Fuel Oil (Nominal Mass Fraction 10 mg/kg)

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 of Sulfur: 10.90 mg/kg ± 0.31 mg/kg

A Bayesian statistical analysis with non-informative prior distributions was used to establish the certified value and its expanded uncertainty, U, which are given as a symmetric 95 % probability interval for the certified sulfur mass fraction [2]. Although the expanded uncertainty of the certified value was not computed using the methods outlined in the ISO Guide [3], the results of the Bayesian analysis can be interpreted in the same way as results from the ISO approach. For this purpose, the expanded uncertainty can be expressed as $U = ku_c$, where $u_c = 0.156$ mg/kg is the combined standard uncertainty, and the coverage factor, $k = 2.008$, is determined from the Student's t-distribution with $v = 50.31$ degrees of freedom. Alternatively, a discrete approximation to the posterior probability distribution for the certified sulfur mass fraction is provided for subsequent Bayesian uncertainty calculations associated with the use of this material. This probability distribution for the certified value can be found as Supplementary Information on the SRM Website at https://www-s.nist.gov/srmors/view_detail.cfm?srm=2723A.

Certified Mass Fraction Value: The certified mass fraction value for sulfur is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS) [4]. 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 [5]. The certified value is reported in mass fraction units [6]. Homogeneity testing was performed using X-ray fluorescence (XRF) spectrometry and indicated no detectable heterogeneity [7]. A NIST XRF gravimetric standard additions procedure yielded a sulfur value that is consistent with the certified value [8].

Information Values: Information values for additional properties of SRM 2723a are provided in Table 1. A NIST information value is a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value, therefore no uncertainty is provided [4].

Expiration of Certification: The certification of this SRM is valid, within the measurement uncertainty specified, until **31 December 2015**, provided the SRM is handled and stored in accordance with the instructions given in the certificate (see "Instructions for Handling, Storage, and 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 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) will facilitate notification.

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Gaithersburg, MD 20899
Certificate Issue Date: 12 September 2012
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Robert L. Watters, Jr., Chief
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The overall direction and coordination of the technical measurements leading to certification of this SRM were performed by G.C. Turk and J.L. Molloy 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, Jr. of the NIST Analytical Chemistry Division. Statistical analysis for this SRM was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

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

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

INSTRUCTIONS FOR HANDLING, STORAGE, AND 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 1. Information Values for Selected Properties

Physical Property Test[a]	ASTM Standard Used	Result
Density @ 15 °C	D 1250-80 (1990)	818.0 kg/m^3
@ 60 °F	D 4052-96	41.4 API
Flash Point	D 93 (A)-94	88.9 °C
Kinematic Viscosity @ 40 °C	D 445-94	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	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)
D 1250-80 (1990)	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 Stand., Vol. 05.01, West Conshohocken, PA (1998).

[2] Gelman, A.; Carlin, J.B.; Stern, H.S.; Rubin, D.B.; *Bayesian Data Analysis*; Chapman and Hall: London, (1995).

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[5] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://www.nist.gov/srm/upload/SP260-136.PDF (accessed Sept 2012).

[6] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at http://www.nist.gov/pml/pubs/sp811/index.cfm (accessed Sept 2012).

[7] Marlow, A.F.; Sieber J.R.; *Acceptance Testing of Proposed SRM 2723a, Sulfur in Diesel Fuel, Report of Analysis*; 6 pages, November 14, 2001, 839.01-02-003.

[8] Barker, L.R.; Kelly, W.R.; Guthrie, W.F.; *Determination of Sulfur in Biodiesel and Petroleum Diesel by X-ray Fluorescence (XRF) Using the Gravimetric Standard Addition Method – II*; Energy & Fuels, Vol. 22, pp. 2488–2490 (2008).

Certificate Revision History: 12 September 2012 (Updated certified sulfur value and uncertainty to reflect additional ID-TIMS sulfur data; editorial changes); **24 February 2006** (Editorial changes); **12 February 2004** (Editorial changes about ampouling under an argon atmosphere); **08 December 2003** (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.