National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1623d

Sulfur in Residual Fuel Oil

(Nominal Mass Fraction 0.2 %)

This Standard Reference Material (SRM) is intended primarily for use in the determination of total sulfur in fuel oils or materials of similar matrix. SRM 1623d consists of 100 mL of commercial "No. 6" residual fuel oil [1], in an amber glass bottle.

The certified mass fraction value for the sulfur content, expressed in percent, is given below.

Certified Sulfur Mass Fraction: 0.2070 % ± 0.0054 %

The certified sulfur mass fraction is based on analyses using Isotope Dilution Inductively Coupled Plasma Mass Spectrometry (ID-ICP-MS), Prompt Gamma-ray Activation Analysis (PGAA), and Wavelength Dispersive X-ray Fluorescence Spectrometry (WDXRF). The certified value is metrologically traceable to the derived SI unit for mass fraction, expressed as a percentage. 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]. Homogeneity testing was performed using WDXRF.

A Bayesian statistical analysis was used to establish the sulfur certified value and its expanded uncertainty, *U*, from the results of the analyses, producing a symmetric 95 % probability interval for the certified sulfur mass fraction [3] that is consistent with the analytic methods described in the Simple Guide for Evaluating and Expressing the Uncertainty of NIST Measurement Results [4]. Although the expanded uncertainty of the certified value was not computed using the methods outlined in the ISO/JCGM Guide [5], the uncertainty from the Bayesian analysis can be interpreted similarly to results from the ISO/JCGM approach. For this purpose, the expanded uncertainty can be expressed as $U = ku_c$, where $u_c = 0.0027$ % is the combined standard uncertainty, and the coverage factor, $k = 2$, is determined from the normal distribution.

Expiration of Certification: The certification of **SRM 1623d** is valid, within the measurement uncertainty specified, until **31 March 2025**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). However, the certification will be nullified if the SRM is damaged, contaminated, or 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 online) will facilitate notification.

Overall direction and coordination of the technical measurements leading to certification of this SRM were performed by J.L. Molloy of the NIST Chemical Sciences Division.

Analytical measurements were performed by S.J. Christopher, J.L. Molloy and R.L. Paul of the NIST Chemical Sciences Division.

Statistical calculations for this SRM were carried out by S. Lund of the NIST Statistical Engineering Division.

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Gaithersburg, MD 20899
Certificate Issue Date: 25 June 2018

Steven J. Choquette, 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 HANDLING, STORAGE, AND USE

Because of the viscosity of SRM 1623d, it is recommended that the SRM unit be warmed slowly to between 40 °C and 60 °C and then shaken, or stirred with a clean stirrer, before sampling. Care must be exercised to not introduce entrapped air, which could affect gravimetric measurements and XRF responses. A detailed study to determine if the sulfur components of SRM 1623d will segregate has not been performed.

The SRM bottle should only be opened for the minimum time required to dispense the material. To relate analytical determinations to the certified value in this Certificate of Analysis, a minimum sample mass of 150 mg should be used. After use, the bottle should be tightly recapped and stored under normal laboratory conditions away from direct sunlight.

SUPPLEMENTAL INFORMATION

The physical properties of SRM 1623d are listed in the table below. These properties were determined using ASTM methods by a commercial firm under contract to NIST. The results are **NOT** certified and are provided as additional information on the matrix.

Physical Property Test		ASTM Standard	Result
Density	@ 15 ºC	D 4052-11	0.9520 g/cm^3
	@ 60 ºF	D 287-92 (2006)	17ºAPI
Flash Point, PMCC		D 93-12, Method B	>110 °C
Pour Point		D 97-12	27 °C
Kinematic Viscosity	@ 40 °C	D 445-12	2.061 x 10^{-3} m^2/s (2061 cSt)
	@ 50 °C	D 445-12	7.039 x 10^{-4} m^2/s (703.9 cSt)
Carbon		D 5291-10	87.4 %
Hydrogen		D 5291-10	11.9 %

ASTM Standards Used

D93-12	Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
D97-12	Standard Test Method for Pour Point of Petroleum Products
D287-92 (2006)	Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)
D445-12	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
D4052-11	Standard Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
D5291-10	Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

REFERENCES

[1] ASTM D396-17 *Standard Specification for Fuel Oils*, ASTM International, West Conshohocken, PA, 2017, https://doi.org/10.1520/D0396-17 (accessed Jun 2018).

[2] 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; U.S. Government Printing Office: Washington, DC (2000); available at https://www.nist.gov/srm/upload/SP260-136.PDF (accessed Jun 2018).

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

[4] Possolo, A.; Simple Guide for Evaluating and Expressing the Uncertainty of NIST Measurement Results; NIST Technical Note 1900; National Institute of Standards and Technology: Gaithersburg, MD (2015); available at https://nvlpubs.nist.gov/nistpubs/TechnicalNotes/NIST.TN.1900.pdf (accessed Jun 2018)

[5] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jun 2018); 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 https://www.nist.gov/pml/pubs/tn1297/index.cfm (accessed Jun 2018).

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 https://www.nist.gov/srm.