National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2771

Sulfur in Diesel Fuel Blend Stock

This Standard Reference Material (SRM) is intended for use in the evaluation of methods, preparation of calibration mixes [1], and calibration of instruments used in the determination of total sulfur in diesel fuel oils or materials of a similar matrix. SRM 2771 is a commercial diesel fuel blend stock that was initially issued as Reference Material (RM) 8771 in 2005, with a reference value assigned for sulfur. With the incorporation of new data, the value has been upgraded to a certified value, and the material is now available as SRM 2771 [2]. A unit of SRM 2771 consists of an amber bottle containing approximately 100 mL of diesel fuel blend stock.

Certified Mass Fraction Value: The certified mass fraction value for sulfur is in Table 1. 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 the present best estimate of the "true" value based on the results of analyses performed at NIST and at LGC, Teddington, UK. Because the dominant source of uncertainty in this very low sulfur material is the variation in the chemical blank during the analysis, a Bayesian statistical analysis with non-informative prior distributions was used to establish the mean and its expanded uncertainty, *U*, which are given as a symmetric 95 % probability interval for the certified sulfur mass fraction [3].

Table 1. Certified Mass Fraction Value for Sulfur

Sulfur:	0.102 mg/kg ± 0.014 mg/kg

The expanded uncertainty can be expressed as $U = ku_c$, where $u_c = 0.00735$ mg/kg is the combined standard uncertainty, and the coverage factor, $k = 1.96$, is determined from the Student's *t*-distribution corresponding to infinite degrees of freedom. Although the expanded uncertainty of the certified value was not computed using the methods outlined in the ISO Guide [4], the results of the Bayesian analysis can be interpreted in the same way as results from the ISO approach. Alternatively, a discrete approximation of the posterior distribution for the certified sulfur mass fraction is provided for subsequent Bayesian uncertainty calculations associated with the use of this material can be found on the SRM Website at https://www-s.nist.gov/srmors/view_detail.cfm?srm=2771.

Information Values: A NIST information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess adequately the uncertainty associated with the value or only a limited number of analyses were performed [2]. Information values are provided in Table 2 for additional properties of SRM 2771.

Expiration of Certification: The certification of **SRM 2771** is valid, within the measurement uncertainty specified, until **01 January 2015**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Storage 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) 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.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 28 September 2010

Robert L. Watters, Jr., Chief
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Samples of SRM 2771 for sulfur determinations were prepared by W.R. Kelly and J.L. Mann of the NIST Analytical Chemistry Division, and determined at LGC by S.E. Long of the NIST Analytical Chemistry Division and R. Hearn of LGC.

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

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

INSTRUCTIONS FOR STORAGE AND USE

Storage: The capped bottle must be stored under normal laboratory conditions away from direct sunlight and sulfur-containing fumes.

Use: Each SRM bottle should only be opened for the minimum time required to dispense the material. Transfer of material from the bottle is to be performed with maximum care not to contaminate the SRM. The contents of the SRM bottle do **NOT** need to be mixed or stirred. To relate analytical determinations to the certified value in this certificate, a minimum test portion of 200 mg should be used.

Source and Preparation of Material[1]: The material for SRM 2771 was produced by ConocoPhillips Company (Westlake, LA). The ultra low sulfur diesel fuel blend stock used for SRM 2771 is a hydro-treated, straight-cut fraction collected within the diesel boiling range. This material has a higher viscosity and lower American Petroleum Institute (API) gravity (density) than commercial diesel fuel oil. Collection of the material used for SRM 2771 was performed under the direction of J.A. Bennett and R.R. Robinson, ConocoPhillips Company in consultation with B.S. MacDonald of the NIST Measurement Services Division.

Analytical Methods: The certified sulfur value was obtained using a high-resolution isotope dilution inductively coupled plasma mass spectrometry (HR-ID-ICP-MS) with a single pulse-counting detector operated in the medium-resolution mode at LGC. The information values listed in Table 2 were obtained by collaborating laboratories using the ASTM methods that are listed in the table.

Homogeneity: This material is a clear colorless liquid with a viscosity similar to that of diesel fuel. Based on experience with similar materials and the experimental data, the observed bottle-to-bottle variability did not indicate an appreciable component of variance attributable to inhomogeneity.

Table 2. Information Values for Selected Properties

Physical Property Test[(a)]	ASTM Standard Used[(b)]	Result
Density at 15 °C	D 1250-80 (1990)[Є1]	835.4 kg/m^3
At 60 °F	D 4052-96	37.8 °API
Flash Point	D 93 (A)-94	105.6 °C
Kinematic Viscosity at 37.8 °C (100 °F)	D 445-94[Є2]	4.590 × 10^{-6} m^2/s (4.590 cSt)
Carbon	D 5291-92	85.0 %
Hydrogen	D 5291-92	14.7 %

[(a)] These properties were determined by a commercial firm under contract to NIST using ASTM methods listed below. The results are **NOT** certified and are provided only as additional information on the matrix.
[(b)] Standards listed with Є1 or Є2 indicate that only editorial changes were made to the previous issuance of the ASTM standard as indicated by ASTM.

[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.

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$^{\epsilon 2}$	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (The Calculation of Dynamic Viscosity)
D 1250-80 (1990)$^{\epsilon 1}$	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] Kelly, W.R.; MacDonald, B.S.; Liegh, S.D.; *A Method for the Preparation of NIST Traceable Fossil Fuel Standards with Concentrations Intermediate to SRM Values*; J. ASTM Int., Vol. 4, No. 2 (2007).

[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 (2000); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PUBLICATIONS.cfm (accessed Sep 2010).

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

[4] 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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2010); 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/physlab/pubs/index.cfm (accessed Sep 2010).

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) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.