



Report of Investigation

Reference Material 8771

Sulfur in Diesel Fuel Blend Stock

This Reference Material (RM) is intended for use in the evaluation of methods and the calibration of instruments used in the determination of total sulfur in diesel fuel oils or materials of a similar matrix. RM 8771 is a commercial diesel fuel blend stock. A unit of RM 8771 consists of an amber bottle containing approximately 100 mL of diesel fuel blend stock.

Reference Value: A NIST reference value is a noncertified value that is the best estimate of the true value; however, the value does not meet the NIST criteria for certification and is provided with an associated uncertainty that may not include all sources of uncertainty [1]. The reference sulfur content, provided in Table 1, was estimated using a standard addition procedure with gravimetric mixtures of RM 8771 with SRM 1616b *Sulfur in Kerosene* and SRM 2770 *Sulfur in Diesel Fuel Oil* prepared at NIST and measured according to a NIST experimental plan at ConocoPhillips Company (Bartlesville, OK) using thermal oxidation with a UV fluorescence detector [2]. Homogeneity was evaluated by NIST using measurements by the same instrument. No evidence of significant heterogeneity was observed. The two NIST SRMs used as calibrants in this work were previously certified by isotope dilution thermal ionization mass spectrometry (ID-TIMS) [3]. The reference value for sulfur is given as a 95 % expanded uncertainty interval with an expanded uncertainty of $U = ku_c$, where the quantity u_c is the combined standard uncertainty of the estimated sulfur content and k is a coverage factor used to control the confidence level [4]. The value of the coverage factor, $k = 2.074$, was obtained from the Student's t -distribution with 22 effective degrees of freedom and a confidence level of 95 % [4]. A Bayesian statistical analysis with non-informative prior distributions [5] was used to verify that the uncertainty in the standards was not significant relative to the other sources of uncertainty accounted for in the reference value.

Table 1. Reference Value (mass fraction)

Sulfur: 0.071 mg/kg \pm 0.014 mg/kg

Information Values: A NIST information value is considered to be a value that will be of interest and use to the RM user, but insufficient information is available to assess adequately the uncertainty associated with the value or only a limited number of analyses were performed [3]. Information values are provided in Table 2 for additional properties of RM 8771.

Expiration of Reference Value: The reference value of RM 8771 is valid, within the measurement uncertainty specified, until **01 January 2015**, provided the RM is handled in accordance with instructions given in this report (see "Instructions for Use").

Maintenance of Reference Value: NIST will monitor this RM over the period of its validity. If substantive changes occur that affect the reference value before expiration, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification of this RM was provided by W.R. Kelly and G.C. Turk of the NIST Analytical Chemistry Division.

The gravimetric mixes were prepared by W.R. Kelly and J.L. Mann of the NIST Analytical Chemistry Division.

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Collection of the material used for RM 8771 was performed under the direction of J.A. Bennett and R.R. Robinson ConocoPhillips Company (Westlake, LA). Sulfur measurements at ConocoPhillips Company (Bartlesville, OK) using the Analytik Jena multi EA 3100 analyzer¹ were performed by R.D. Williams under the direction of K.F. Dahnke.

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

Bottling was performed under the supervision of M.P. Cronise of the NIST Measurement Services Division.

The support aspects involved in the issuance of this RM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

Source and Preparation of Material: The material for RM 8771 was donated by ConocoPhillips Company (Westlake, LA). The ultra low sulfur diesel fuel blend stock used for RM 8771 is a hydro-treated, straight-cut fraction collected within the diesel boiling range. This material has a higher viscosity and lower API gravity (density) than commercial diesel fuel oil.

INSTRUCTIONS FOR USE

Each RM 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 RM. The contents of the RM bottle do **NOT** need to be mixed or stirred. To relate analytical determinations to the reference value in this report, a minimum sample mass of 200 mg should be used. The capped bottle should be stored under normal laboratory conditions away from direct sunlight and sulfur containing fumes.

Table 2. Information Values for Selected Properties

Physical Property Test ^(a)	ASTM Standard Used	Result
Density @ 15 °C @ 60 °F	D 1250-80 (1990) ^{C1} D 4052-96	835.4 kg/m ³ 37.8 °API
Flash Point	D 93 (A)-94	105.6 °C
Kinematic Viscosity @ 37.8 °C (100 °F)	D 445-94 ^{C1}	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. The results are **NOT** certified and are provided only 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 ^{C1}	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)
D 1250-80 (1990) ^{C1}	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

¹Certain commercial equipment, instruments, or materials are identified in this report to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

^{C1}Indicates that only editorial changes were made to the previous issuance of the ASTM standard.

REFERENCES

- [1] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, p. 16, U.S. Government Printing Office: Washington, DC (2000).
- [2] ASTM D 5453-05; *Standard Test Method for Determination of Total Sulfur in Light Hydrocarbons, Motor Fuels and Oils by Ultraviolet Fluorescence*; Annu. Book of ASTM Stand., Vol. 05.03 (2005).
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- [4] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: 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/>.
- [5] Gelman, A.; Carlin, J.B.; Stern, H.S.; Rubin, D.B.; *Bayesian Data Analysis*; Chapman and Hall: London, (1995).

Users of this RM should ensure that the report 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; or via the Internet at <http://www.nist.gov/srm>.