



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material® 1617b

#### Sulfur in Kerosene (High Level)

This Standard Reference Material (SRM) is a high sulfur kerosene as described in ASTM Specification for Kerosine [1] intended for use in the determination of total sulfur in fuel oils or materials of similar matrix. A unit of SRM 1617b consists of 100 mL of kerosene in an amber bottle.

**Certified Mass Fraction Value:** The certified value for the sulfur content of SRM 1617b is reported as a mass fraction [2] 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 [3]. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories using instrumental test methods.

**Reference Values:** Reference values for kinematic viscosity, speed of sound, and density of SRM 1617b are given in Table 2. A NIST reference value is a noncertified value that is the best estimate of the true value based on available data; however, the value does not meet the NIST criteria for certification [3] and is provided with associated uncertainties that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple measurement methods.

**Information Values:** Information values for additional parameters are given in Table 3 and Table 4. An 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 the uncertainty associated with the value [3]. Information values cannot be used to establish metrological traceability.

**Expiration of Certification:** The certification of **SRM 1617b** is valid, within the uncertainty specified, until **01 January 2023** provided the SRM is handled and stored in accordance with the 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 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.

The coordination of technical measurements for certification was performed by J.L. Molloy of the NIST Chemical Sciences Division.

Analyses leading to the certification of this SRM were performed by J.L. Molloy, J.R. Sieber, and S.J. Christopher of the NIST Chemical Sciences Division and A. Laesecke and T.M. Lovestead of the NIST Applied Chemicals and Materials Division. Analytical results were also provided by the ASTM International Committee D-2 Interlaboratory Crosscheck Program and SGS North America Inc.

Statistical consultation for this SRM was provided by S. Lund and J.D. Splett of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Gaithersburg, MD 20899  
Certificate Issue Date: 11 June 2014

Robert. L Watters, Jr., Director  
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## INSTRUCTIONS FOR STORAGE AND USE

Open the SRM bottle 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. Store the SRM under normal laboratory conditions away from direct sunlight.

## PREPARATION AND ANALYSIS

Determination of sulfur content of SRM 1617b was performed using wavelength dispersive X-ray fluorescence spectrometry (XRF) and isotope dilution inductively coupled plasma mass spectrometry (ID-ICP-MS). Kinematic viscosity values shown in Table 2 were measured using an open gravitational capillary viscometer. Density and speed of sound values shown in Table 2 were measured using a density and speed of sound analyzer. Hydrocarbon type (Table 3) was measured using gas chromatography with mass spectrometry detection. Flashpoint and mercaptan sulfur (Table 4) were determined in an ASTM International Committee D-2 Interlaboratory Crosscheck Program using the methods shown in Table 4. Hydrogen and carbon content of SRM 1617b were determined by SGS North America Inc. using the methods shown in Table 4.

**Homogeneity Assessment:** The homogeneity was assessed for sulfur in the bottled material using wavelength dispersive XRF. Results from duplicate portions from individual bottles indicated that the uncertainty contribution attributable to inhomogeneity did not contribute significantly to the overall uncertainty of the sulfur mass fraction value.

**Certified Mass Fraction Value:** The measurand is the mass fraction of sulfur in kerosene. The certified value is metrologically traceable to the SI unit of mass, expressed as percent. A Bayesian statistical analysis was used to establish the sulfur certified value and its expanded uncertainty,  $U$ , from results of the analyses described above, resulting in a symmetric 95 % probability interval for the certified sulfur mass fraction [4]. Although the expanded uncertainty of the certified value was not computed using the methods outlined in the ISO/JCGM Guide [5,6], 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  $u_c = 0.0012$  % is the combined standard uncertainty, and the coverage factor,  $k = 2$ , is determined from the normal distribution.

Table 1. Certified Mass Fraction Value for Sulfur in SRM 1617b

Constituent	Mass fraction (%)
Sulfur	0.1250 ± 0.0025

**Reference Values for Kinematic Viscosity, Density, and Speed of Sound:** The reference values for kinematic viscosity, density, and speed of sound are the results from a single method [7,8], measured at ambient atmospheric pressure (0.083 MPa). Kinematic viscosity was measured for temperatures from 20 °C to 100 °C and density and speed of sound were measured for temperatures from 5 °C to 70 °C. The expanded uncertainties ( $U$ ) were calculated using a  $k$  factor associated with a 95 % confidence level based on degrees of freedom from the Welch-Satterthwaite approximation [5,6]. Reference values are based on the method used for each property. The measurands are listed as headings in Table 2 and are metrological traceability to SI Units of length per unit of time, density and velocity, respectively.

Table 2. Reference Values for Kinematic Viscosity, Density, and Speed of Sound in SRM 1617b

Temperature (°C)	Kinematic Viscosity (mm <sup>2</sup> /s)		Density (kg/m <sup>3</sup> )		Speed of Sound (m/s)	
	value	$U$	value	$U$	value	$U$
5	-	-	816.75	0.06	1380.2	0.6
10	-	-	813.02	0.06	1360.6	0.6
15	-	-	809.28	0.06	1340.8	0.6
20	1.957	0.007	805.57	0.06	1321.6	0.6
25	1.794	0.007	801.88	0.06	1302.3	0.6
30	1.652	0.007	798.2	0.06	1283.3	0.6
35	1.527	0.006	794.52	0.06	1264.3	0.6
40	1.419	0.006	790.83	0.06	1245.5	0.6
45	1.323	0.006	787.14	0.06	1226.9	0.6
50	1.237	0.006	783.44	0.06	1208.3	0.6
55	1.159	0.006	779.73	0.06	1189.9	0.6
60	1.091	0.007	776.01	0.06	1171.6	0.6
65	1.029	0.007	772.27	0.06	1153.7	0.6
70	0.9732	0.0070	768.52	0.06	1136	0.6
75	0.9222	0.0074	-	-	-	-
80	0.8757	0.0079	-	-	-	-
85	0.8335	0.0084	-	-	-	-
90	0.7949	0.0091	-	-	-	-
95	0.7592	0.0099	-	-	-	-
100	0.7263	0.0108	-	-	-	-

**Information Values:** The values in Table 3 were generated using ASTM method D2789 *Standard Test Method for Hydrocarbon Types in Low Olefinic Gasoline by Mass Spectrometry* to classify various hydrocarbon types. The uncertainties and potential pitfalls of this method have been discussed previously [9,10]. The methods used to generate the values in Table 4 are denoted in the table.

Table 3. Information Values for Hydrocarbon Types Present in SRM 1617b

Hydrocarbon Type	Mass Fraction (%)
Paraffins	39
Monocycloparaffins	30
Dicycloparaffins	9.5
Alkylbenzenes	14
Indanes and Tetralins	4
Naphthalenes	2

Table 4. Information Values for Additional Properties of SRM 1617b

Property	ASTM Method	Value
Flash Point	D56 <sup>(a)</sup>	55.4 °C
Hydrogen	D5291 <sup>(b)</sup>	13.9 % (mass fraction)
Carbon	D5291 <sup>(b)</sup>	86.3 % (mass fraction)
Mercaptan Sulfur	D3227 <sup>(c)</sup>	7.3 mg/kg

- <sup>(a)</sup> D56-05 Test Method for Flash Point by Tag Closed Tester  
<sup>(b)</sup> D5291-10 Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants  
<sup>(c)</sup> D3227-04a Test Method for Thiol (Mercaptan) Sulfur in Gasoline, Kerosine, Aviation Turbine, and Distillate Fuels (Potentiometric Method)

## REFERENCES

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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](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.