



## National Institute of Standards & Technology

# Certificate of Analysis

## Standard Reference Materials 1819a

### Sulfur in Lubricating Base Oil

This Standard Reference Material (SRM) is intended for use as an analytical standard in the determination of total sulfur in lubricating base oils or materials of similar composition. SRM 1819a consists of five lubricating base oils at different sulfur concentrations.

#### Total Sulfur Concentration (mg/kg) in SRM 1819a

Level	Certified Value	Expanded Uncertainty
Oil I	423.5	2.2
Oil II	741.1	4.3
Oil III	4022	17
Oil IV	4689	21
Oil V	6135	23

The sulfur contents in SRM 1819a were certified using isotope dilution thermal ionization mass spectrometry (ID-TIMS). The expanded uncertainty of the certified values was computed according to the NIST uncertainty policy, as described in NIST Technical Note 1297 [1], and is at the 95% level of confidence.

Oils I to IV were purchased from suppliers and Oil V was provided by Mobil Corp., Paulsboro, NJ.

**Stability and Storage:** The certification of SRM 1819a is valid for three years from the date of shipment from NIST. The standards should be stored in a cool, dark place when not in use. Please discard the standards immediately if any sediment is observed.

ID-TIMS analyses for certification were performed by W.R. Kelly and R. Voeck, Inorganic Analytical Research Division. X-ray fluorescence analyses were performed by P.T. Pei, Ceramic Division and by J.R. Sieber, Texaco Inc., Beacon, NY, to test for homogeneity, according to modified ASTM D 4294-90 and D 4927-39. The production of this SRM was coordinated by P.T. Pei.

The statistical analysis of the certification data was performed by S.B. Schiller, Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills and J.S. Kane.

Gaithersburg, MD 20899  
April 11, 1994

Thomas E. Gills, Chief  
Standard Reference Materials Program

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## SUPPLEMENTAL INFORMATION

The uncertainty of the certified value does **not** reflect the expected precision of a future measurement by X-ray fluorescence spectroscopy (modified ASTM D 4294-90); therefore, a table providing the expected precision of a single future measurement (at the 95% confidence level) is given. Assuming the measurement process is under control, a future measurement should fall within this uncertainty of the certified value.

	Certified Value (mg/kg)	Uncertainty of a Single Future XRF Measurement
Level I	423.5	11
Level II	741.1	14
Level III	4022	66
Level IV	4689	67
Level V	6135	92

The table below describes the sources of uncertainty combined to determine the expanded uncertainties of the certified values. The combined standard uncertainty is the root sum of squares of the individual standard uncertainties, and the expanded uncertainty is the product of the combined standard uncertainty and a t-multiplier based on the degrees of freedom reported in the table.

Components of Uncertainty (mg/kg)						Degrees of Freedom	Uncertainty Type <sup>1</sup>
Level	I	II	III	IV	V		
Sample Measurement	0.60	1.58	6.21	7.83	8.59	5	A
Spike Calibration	0.23	0.40	2.20	2.56	3.35	11	A
Sample Blank	0.81	0.93	0.98	0.94	0.93	169	A
Spike Blank	0.12	0.21	1.16	1.36	1.77	∞	B
Mass Fractionation	0.35	0.61	3.28	3.83	5.01	∞	B
Combined:	1.10	1.98	7.51	9.23	10.68		
Certified Value of S (mg/kg):	423.5	741.1	4022	4689	6135		
Expanded Uncertainty:	2.2	4.3	17	21	23		
Degrees of Freedom:	730	12	11	10	12		

<sup>1</sup> A: Estimated by statistical methods

B: Estimated by scientific judgment

The following physical properties of SRM 1819a have been measured to provide additional information on the nature of the oils; the values are reported, but are not certified. Tests were performed at Saybolt, Inc., Corpus Christi, TX.

Table 3

Physical Properties of SRM 1819a

Oil	Flash* Point (°C)	Kinematic <sup>b</sup> Viscosity (cSt)		Pour <sup>c</sup> Point (°C)	Density <sup>d</sup> at 15 °C (kg/L)	Refractive <sup>e</sup> Index ( $n_D^{20}$ )
		40 °C	100 °C			
I	232	49.93	7.14	-15	0.8703	1.4770
II	207	24.85	4.64	-6	0.8650	1.4775
III	207	30.85	5.36	-9	0.8725	1.4800
IV	249	63.00	8.39	-6	0.8773	1.4820
V	235	38.84	6.17	-6	0.8768	1.4830

Methods Used for Physical Tests

- ASTM D 92-90 Flash and Fire Points by Cleveland Open Cup.
- ASTM D 445-88 Kinematic Viscosity of Transparent and Opaque Liquids.
- ASTM D 97-87 Pour Point of Petroleum Oils.
- ASTM D 1298-90 Density, Relative Density or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer method.
- ASTM D 1218-82 Test for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids.

REFERENCE

- [1] Taylor, B.N., and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results", NIST Tech Note 1297, (1993).