



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1818a

Chlorine in Lubricating Base Oils

This Standard Reference Material (SRM) is intended for use as an analytical standard for the determination of total chlorine in lubricating base oils or materials of similar composition. SRM 1818a consists of a series of five 20 mL ampules of lubricating base oils at different chlorine concentrations.

Total Chlorine Concentration (in mg/kg)

Level	Certified Value	Expanded Uncertainty
1818a-I	31.6	0.9
1818a-II	60.0	2.3
1818a-III	78.2	2.5
1818a-IV	154.4	5.3
1818a-V	234.0	12.4

The chlorine certified values in SRM 1818a were established using instrumental neutron activation analysis (INAA). Homogeneity testing performed using X-ray fluorescence spectroscopy (XRF) did not show significant material variance, while INAA certification analyses did detect possible material heterogeneity.

The uncertainty was calculated according to NIST Technical Note 1297, "Guidelines for Evaluating and Expressing Uncertainty of NIST Measurement Results". [1] The expanded uncertainty given includes both analytical uncertainty and possible material heterogeneity, and is at the 95% level of confidence.

Stability and Storage: The certification of SRM 1818a is valid for three years from date of purchase. The ampules should be stored in a cool and dark place. Any ampule observed to contain sediment should be discarded.

INAA analyses for certification were performed by L. Tandon and D.A. Becker, of the NIST Inorganic Analytical Research Division and XRF analyses were performed by P.T. Pei, of the NIST Ceramic Division. The production of this SRM was coordinated by P.T. Pei.

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

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

Gaithersburg, MD 20899
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Thomas E. Gills, Chief
Standard Reference Materials Program

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SUPPLEMENTAL INFORMATION

I. Detailed standard uncertainties (in mg/kg), and Degrees of Freedom (DF), assuming material heterogeneity based on INAA data

Table 1. Components of Uncertainty (mg/kg)

Source of Uncertainty						Degrees of Freedom ¹	Uncertainty Type ²
	I	II	III	IV	V		
Measurement Replication and Material Variability	0.061	0.0810	0.799	2.052	4.861	5	A
Standard Concentration	0.285	0.545	0.703	0.770	1.170	3	A
Irradiation Position	0.047	0.091	0.117	0.232	0.351	5	A
Weighing	0.001	0.002	0.002	0.005	0.007	9	A
Counting Geometry	0.046	0.094	0.134	0.196	0.304	∞	B
Combined Uncertainty:	0.299	0.985	1.079	2.213	5.021		
Certified Value (mg/kg)	31.6	60.0	78.2	154.4	234.0		
Degrees of Freedom:	3.62	8.15	8.32	6.44	5.65		
Coverage Factor:	2.90	2.30	2.29	2.41	2.48		
Expanded Uncertainty:	0.87	2.3	2.45	5.3	12.4		

¹Two degrees of freedom for standard concentration in Levels IV & V

²Uncertainty Type: A: By statistical methods
B: By other means

II. XRF Results

The uncertainty of the certified value does not reflect the expected precision of a future measurement by X-ray fluorescence spectroscopy (XRF) [2]; therefore, a table providing the expected precision of a single 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 in mg/kg	Uncertainty of a single future XRF measurement
Level I	31.6	7.5
Level II	60.0	6.1
Level III	78.2	5.7
Level IV	154.4	8.1
Level V	234.0	14.1

The following physical properties have been measured on SRM 1818a to provide additional information on the nature of the oils, the values are reported, but are not certified.

Table 3

Physical Properties of SRM 1818a Lubricating Base Oils

Oil	Flash ^a Point (°C)	Kinematic ^b Viscosity (cSt)		Pour ^c Point (°C)	Density ^d at 15 °C (kg/L)	Refractive ^e Index (n_D^{20})
		40°C	100°C			
I	216	58.6	8.38	-12	0.8773	1.4825
II	193	28.52	5.01	-12	0.8752	1.4805
III	210	25.37	4.67	-9	0.8730	1.4790
IV	204	31.49	5.34	-9	0.8773	1.4815
V	196	31.02	5.30	-9	0.8790	1.4835

Methods Used for Physical Tests

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

The measurements for Table 3 were performed by G. Plocek, Saybolt, Inc., Corpus Christi, TX.

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).
- [2] Pei, P.T., Fleming, R., and Hsu, S.M., "Test Methods for Total Chlorine in Lubricating Base Oils", NIST Special Publication 674, p. 271 (1984).