National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1848

Lubricating Oil Additive Package

This Standard Reference Material (SRM) is a typical additive package used in the manufacture of crankcase lubricating oil for gasoline engines. It is intended primarily for evaluation of methods used in the analysis of lubricating oil additive packages, engine lubricating oils, and materials of a similar matrix. It can be used to validate value assignment of in house reference materials. A unit of SRM 1848 consists of an amber, borosilicate glass bottle containing approximately 100 g of material.

Certified Mass Fraction Values: Certified values for constituents in SRM 1848 are listed in Table 1 as mass fractions [1] of the total elements in a lubricant additive matrix. 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 taken into account [2]. The certified values are based on measurements using two or more independent analytical methods or a single NIST primary method (see Table 4). The certified values are metrologically traceable to the SI derived unit of mass fraction (expressed as percent).

Table 1. Certified Mass Fraction Values in SRM 1848 Lubricating Oil Additive Package

Element	Mass Fraction (%)		
Boron (B)	0.137	±	0.019
Calcium (Ca)	0.359	±	0.011
Chlorine (Cl)	0.0927	±	0.0020
Magnesium (Mg)	0.821	±	0.038
Phosphorus (P)	0.788	±	0.028
Sulfur (S)	2.3270	±	0.0043
Zinc (Zn)	0.873	±	0.022

The certified value for each element, except sulfur, is equal to a weighted mean of the results of the cited methods performed by NIST and industry laboratories. The uncertainty listed with each value is an expanded uncertainty based on a 95 % confidence interval [3] calculated as $U = ku_c$, where u_c is the combined standard uncertainty and $k = 2$ is a coverage factor. The expanded uncertainty is calculated by combining a between-method variance [4] with a pooled, within-method variance following the ISO/JCGM Guide [5]. For sulfur, the certified value is based on a single method, with the expanded uncertainty including an estimate of the bias in the method.

Expiration of Certification: The certification of **SRM 1848** is valid, within the measurement uncertainty specified, until **01 June 2025**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage, Handling and Use"). Reference values are expected also to remain valid for the same period. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this material 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 or register online) will facilitate notification.

Coordination and overall direction of the technical measurements leading to certification of this SRM were performed by J.R. Sieber of the NIST Chemical Sciences Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
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Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Statistical consultation for this SRM was performed by S.D. Leigh of the NIST Statistical Engineering Division.

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

INSTRUCTIONS FOR STORAGE, HANDLING AND USE

To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample mass of 30 mg is recommended. The material should be stored in its original container at room temperature. If the material has not been sampled recently, it may be advisable to stir the contents of the bottle using a clean glass or plastic implement.

PREPARATION AND ANALYSIS[(1)]

The material used for this SRM was prepared by a commercial supplier of lubricating oil additives, according to its normal procedures and specifications. The material was bottled at NIST.

NIST analytical measurements were performed by W.R. Kelly, E.A. Mackey, J.L. Mann, A.F. Marlow, J.R. Sieber, R.D. Vocke, Jr., and L.J. Wood of the NIST Chemical Sciences Division. Additional analyses were performed by the following collaborating laboratories: BP Amoco (Naperville, IL); Chevron Research and Technology Company (Richmond, CA); Exxon Research and Engineering Company (Annandale, NJ); Phillips Petroleum Company (Bartlesville, OK); Texaco Technology Ghent (Ghent, Belgium); and The Lubrizol Corporation (Wickliffe, OH).

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 1848.

Reference Values: Reference values [2] for hydrogen, nitrogen, and silicon, expressed as mass fractions, and for total base number as defined by ASTM methods D 2896 [6] and D 4739 [7] are given in Table 2. A reference value is a non-certified 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 and is provided with an associated uncertainty that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [2]. Each reference value is equal to a weighted mean of the results of the cited methods performed by NIST or industry laboratories. The uncertainty listed with each value is an expanded uncertainty [3] based on a 95 % confidence interval calculated as $U = ku_c$, where u_c is the combined standard uncertainty and $k = 2$ is a coverage factor. The expanded uncertainty is calculated by combining a between-method variance [4] with a pooled, within-method variance following the ISO/JCGM Guide [5]. The reference value for hydrogen is the mean from a single method, and the expanded uncertainty includes an estimate of the bias in the method. The reference values in Table 2 are traceable to the units listed in Table 2 as realized using the specific test methods given in Table 4.

Table 2. Reference Mass Fraction Values in SRM 1848 Lubricating Oil Additive Package

Element	Mass Fraction (%)
Hydrogen (H)	12.3 ± 0.4
Nitrogen (N)	0.57 ± 0.03
Silicon (Si)	0.0050 ± 0.0002

Property	Total Base Number (mg KOH/g)
Total Base Number by ASTM D 2896[(a)]	56.7 ± 0.7
Total Base Number by ASTM D 4739[(a)]	49.6 ± 5.6

[(a)] This property is method specific. The value stated applies only to the ASTM method cited using the appropriate quantity of material specified for that test.

[(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.

Information Values: Information values [2] are listed in Table 3 for selected physical characteristics of the material determined using selected ASTM methods. An information value is a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value. Information values cannot be used to establish metrological traceability.

Table 3. Information Values for SRM 1848 Lubricating Oil Additive Package

Property	ASTM Method Used	Value	
COC Flash Point	D 92 [8]	194	°C
Pensky-Martens Flash Point	D 93 [9]	173	°C
Kinematic Viscosity, 100 °C	D 445 [10]	170	cSt
Kinematic Viscosity, 40 °C	D 445 [10]	4000	cSt
Ash	D 482 [11]	5.0	% (mass fraction)
Total Acid Number	D 664 [12]	21	mg KOH/g
Sulfated Ash	D 874 [13]	6.1	% (mass fraction)

Table 4. Methods of Analysis for SRM 1848

Element	Methods Used
Boron	PGAA [14], ICPOES, D 4951 [15], D 5185 [16]
Calcium	XRF, ICPOES, D 5185, D 4927 [17]
Chlorine	XRF, PGAA, DIN 51577 [18]
Hydrogen	PGAA
Magnesium	XRF, ICPOES, D 5185, D 4927
Nitrogen	D 5291 [19], D 5762 [20]
Phosphorus	XRF, ICPOES, D 5185, D 4927, D4047 [21]
Silicon	XRF, D 5185
Sulfur	ID-TIMS [22]
Zinc	PGAA, ICPOES, D 4951, D 5185, D 4927

Methods Key

PGAA	Prompt gamma-ray activation analysis
ICPOES	Inductively coupled plasma optical emission spectrometry
ID-TIMS	Isotope dilution - thermal ionization mass spectrometry
XRF	X-ray fluorescence spectrometry: DIN 51577 Part 2, ASTM D 4927 Method A, and other internal standard procedures

REFERENCES

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Certificate Revision History: 19 July 2018 (Change of expiration date; editorial changes.); **01 October 2010** (Editorial changes to Table 4); **29 April 2010** (Change of expiration date; editorial changes.); **20 September 2000** (Original certificate date).

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; or via the Internet at https:/www.nist.gov/srm.