National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1848

Lubricating Oil Additive Package

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

Certified Concentration Values: The certified concentration values [1] reported as mass fractions for seven elements in SRM 1848 are listed 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 accounted for by NIST. The certified values are based on measurements using two or more independent analytical methods or a single NIST primary method (see Table 4).

Reference Values: Reference values [1] for hydrogen, nitrogen, and silicon, expressed as mass fractions, and for total base number as defined by ASTM methods D 2896 [2] and D 4739 [3] are given in Table 2. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty. The reference values for total base number are method specific and apply only to the ASTM methods cited.

Information Values: Information values [1] are provided in Table 3 for selected physical characteristics of the material determined using selected ASTM methods. An information value is considered to be of interest to the SRM user and is provided only as additional information.

Expiration of Certification: The certification of **SRM 1848** is valid, within the measurement uncertainty specified, until **01 May 2018**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "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 technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) 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 Analytical Chemistry Division.

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 Analytical Chemistry Division.

Statistical consultation and evaluation of measurement data were provided by S.D. Leigh of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
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Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

Collaborating Laboratories

The following laboratories contributed to the certification and physical property characterization of this SRM.

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
The Lubrizol Corporation, Wickliffe, OH

Source of Material: The material used for this SRM was prepared by a commercial supplier of lubricant additives according to its normal procedures and specifications. The material was bottled at NIST under the supervision of D.G. Friend of the NIST Measurement Services Division.

STORAGE 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.

Table 1. Certified Concentrations (Mass Fractions, in %) for Selected Elements

Element	Concentration
Boron	0.137 ± 0.019
Calcium	0.359 ± 0.011
Chlorine	0.0927 ± 0.0020
Magnesium	0.821 ± 0.038
Phosphorus	0.788 ± 0.028
Sulfur	2.3270 ± 0.0043
Zinc	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 [4] 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 [5] with a pooled, within-method variance following the ISO Guide [6]. For sulfur, the certified value is based on a single method, with the expanded uncertainty including an estimate of the bias in the method.

Table 2. Reference Values for Selected Elements and Total Base Number

Element or Property	Concentration
Hydrogen	12.3 % ± 0.4 % (mass fraction)
Nitrogen	0.57 % ± 0.03 % (mass fraction)
Silicon	0.0050 % ± 0.0002 % (mass fraction)
Total Base Number by ASTM D 2896[(a)]	56.7 ± 0.7 mg KOH/g
Total Base Number by ASTM D 4739[(a)]	49.6 ± 5.6 mg KOH/g

[(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.

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 [4] 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 [5] with a pooled, within-method variance following the ISO Guide [6]. Reference values for hydrogen and silicon are the mean from a single method for each element. For those elements, the expanded uncertainty includes an estimate of the bias in the method.

Table 3. Information Values for SRM 1848

Property	ASTM Method Used		Value
COC Flash Point	D 92	[7]	194 ºC
Pensky-Martens Flash Point	D 93	[8]	173 ºC
Kinematic Viscosity, 100 ºC	D 445	[9]	170 cSt
Kinematic Viscosity, 40 ºC	D 445	[9]	4000 cSt
Ash	D 482	[10]	5.0 % (mass fraction)
Total Acid Number	D 664	[11]	21 mg KOH/g
Sulfated Ash	D 874	[12]	6.1 % (mass fraction)

Table 4. Methods of Analysis for SRM 1848

Element[(a)]	Methods Used
Boron	PGAA [13], ICPOES, D 4951 [14], D 5185 [15]
Calcium	XRF, ICPOES, D 5185, D 4927 [16]
Chlorine	XRF, PGAA, DIN 51577 [17]
Hydrogen	PGAA
Magnesium	XRF, ICPOES, D 5185, D 4927
Nitrogen	D 5291 [18], D 5762 [19]
Phosphorus	XRF, ICPOES, D 5185, D 4927
Silicon	XRF, D 5185
Sulfur	ID-TIMS [21]
Zinc	PGAA, ICPOES, D 4951, D 5185, D 4927

[(a)]Certified Elements Are in **Bold**

NIST Methods

PGAA	Prompt gamma-ray activation analysis
ICPAES	Inductively coupled plasma atomic 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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[3] ASTM D 4739, *Annual Book of ASTM Standards*, Vol. 05.02 (1999).

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[6] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Oct 2010); 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://www.nist.gov/physlab/pubs/index.cfm (accessed Oct 2010).

[7] ASTM D 92-97, *Annual Book of ASTM Standards*, Vol. 05.01 (1999).

[8] ASTM D 93-97, *Annual Book of ASTM Standards*, Vol. 05.01 (1999).

[9] ASTM D 445-97, *Annual Book of ASTM Standards*, Vol. 05.01 (1999).

[10] ASTM D 482-95, *Annual Book of ASTM Standards*, Vol. 05.01 (1999).

[11] ASTM D 664-95, *Annual Book of ASTM Standards*, Vol. 05.01 (1999).

[12] ASTM D 874-96, *Annual Book of ASTM Standards*, Vol. 05.01 (1999).

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[16] ASTM D 4927-96, *Annual Book of ASTM Standards*, Vol. 05.02 (1999).

[17] DIN 51577, Part 2, English, Deutsches Institut Fur Normung, Beuth Verlag GmbH, Berlin (1993).

[18] ASTM D 5291-96, *Annual Book of ASTM Standards*, Vol. 05.03 (1999).

[19] ASTM D 5762-98, *Annual Book of ASTM Standards*, Vol. 05.03 (1999).

[20] ASTM D 4047-91, *Annual Book of ASTM Standards*, Vol. 05.02 (1999).

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Certificate Revision History: 01 October 2010 (Editoral changes to Table 4); **29 April 2010** (This revision reflects an extension of the certification period and 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) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.