



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 1085b

#### Wear-Metals in Lubricating Oil

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of methods and in the calibration of apparatus used in the analysis of engine lubricating oils and other materials of similar matrix for metal content. A unit of SRM 1085b consists of 10 ampoules: five 5 mL amber borosilicate ampoules, each containing approximately 1.2 g of a blend of 21 constituent elements in a base oil at a nominal concentration of 300 mg/kg; and five ampoules, each containing approximately 1.2 g of a matching base oil intended for use as an analytical blank and for matrix matching.

**Certified Values:** The certified values, expressed as mass fractions [1], for 10 elements 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 taken into account [2]. The certified values are based on measurements using two or more independent analytical methods (see Methods). All values are based on measurements using a sample mass of at least 250 mg.

**Reference Values:** The reference values, expressed as mass fractions, for seven elements 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 [2]. The reference values for arsenic and chlorine are included even though they were not included in the 21 elements blended to the nominal 300 mg/kg concentration.

**Information Values:** The information values, expressed as mass fractions, are provided in Table 3 for the concentrations of six elements. Information values for the mass fractions of elements in the blank base oil are given in 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. Each information value was determined by a single method.

**Expiration of Certification:** The certification of this SRM is valid until **01 August 2010**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for 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) will facilitate notification.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by G.C. Turk of the NIST Analytical Chemistry Division.

Analytical measurements were performed by R. Demiralp, W.R. Kelly, A.F. Marlow, J.R. Sieber, G.C. Turk, and L.L. Yu of the NIST Analytical Chemistry Division. Statistical consultation was provided by S.D. Leigh and M.S. Levenson of the NIST Statistical Engineering Division.

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Gaithersburg, MD 20899  
Certificate Issue Date: 13 January 2009  
*See Certificate Revision History on Last Page*

Support aspects involved in the preparation, certification, and issuance of this SRM 1085b were coordinated through the NIST Measurement Services Division.

**Preparation:** The oil blend of 21 elements at a nominal concentration of 300 mg/kg and matching base oil were prepared by Conostan Division, Conoco Specialty Products, Inc. (Ponca City, OK)<sup>1</sup>. The oil blend was prepared by gravimetrically blending 21 assayed, single-element concentrates into white mineral base oil. The single-element concentrates were assayed by classical wet chemical methods of analysis. Ampoules of SRM 1085b were filled and sealed under argon gas at NIST.

**Instructions for Use:** To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample mass of 250 mg should be used. The blank base oil ampoules should be used for the preparation of analytical blanks. The mass fraction of elements in the blank base oil (see Table 4) is insignificant relative to that of the 300 mg/kg blend. The SRM should be stored at room temperature and protected from temperature extremes. Any unused portion of an opened ampoule cannot be stored for future use and should be disposed of properly.

**Caution:** Small glass chips may separate from the ampoule when opened. Care must be taken to avoid contamination of the SRM.

Table 1. Certified Values for Elements in SRM 1085b

Element	Mass Fraction <sup>a</sup> (mg/kg)
Cadmium	302.9 ± 5.1
Chromium	302.9 ± 3.9
Copper	295.6 ± 8.5
Lead	297.7 ± 6.8
Magnesium	297.3 ± 4.1
Nickel	295.9 ± 7.4
Silver	304.6 ± 8.9
Sodium	305.2 ± 7.0
Vanadium	297.8 ± 4.6
Zinc	296.8 ± 6.8

<sup>a</sup> Each certified value is expressed as a mass fraction ± an expanded uncertainty. The expanded uncertainty is equal to  $U = ku_c$ , where  $u_c$  is the combined standard uncertainty and  $k = 2$  is the coverage factor for an approximate confidence level of 95 %. The uncertainty in the certified value was calculated according to the NIST and ISO guidelines [3] and includes a component of uncertainty for each method and a component that accounts for possible biases among the methods [4].

Table 2. Reference Values for Elements in SRM 1085b

Element	Mass Fraction <sup>b</sup> (mg/kg)
Aluminum	300.4 ± 9.3
Arsenic	51.3 ± 6.7
Chlorine	57.6 ± 9.5
Iron	301.2 ± 5.0
Phosphorus	299.9 ± 7.2
Silicon	300.2 ± 5.0
Titanium	301.1 ± 2.9

<sup>b</sup> Each reference value is expressed as a mass fraction ± an expanded uncertainty. The expanded uncertainty is equal to  $U = ku_c$ , where  $u_c$  is the combined standard uncertainty and  $k = 2$  is the coverage factor for an approximate confidence level of 95 %. The uncertainty in the certified value was calculated according to the NIST and ISO guidelines [3] and includes a component of uncertainty for each method and a component that accounts for possible biases among the methods [4].

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<sup>1</sup>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 are necessarily the best available for the purpose.

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Table 3. Information Values for Elements in SRM 1085b

Element	Mass Fraction (mg/kg)
Barium	314
Boron	300
Calcium	298
Manganese	289
Molybdenum	296
Tin	294

Table 4. Information Mass Fractions for Blank Base Oil in SRM 1085b

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Aluminum	< 7	Molybdenum	< 0.02
Arsenic	< 0.02	Sodium	≤ 0.3
Barium	≤ 0.2	Nickel	< 1
Cadmium	< 0.002	Lead	< 0.03
Calcium	< 5	Phosphorus	< 5
Chlorine	≤ 0.7	Silicon	< 5
Chromium	< 0.07	Silver	< 0.2
Copper	< 0.09	Tin	< 8
Iron	< 2	Titanium	< 1
Magnesium	≤ 0.3	Vanadium	< 0.005
Manganese	< 0.04	Zinc	< 0.15

### Methods

Aluminum	XRF, GRAV	Lead	ICPMS, GRAV
Arsenic	INAA	Molybdenum	INAA, XRF, GRAV
Barium	ICPMS, XRF, GRAV	Nickel	ICPMS, XRF, GRAV
Boron	GRAV	Phosphorus	XRF, GRAV
Cadmium	INAA, GRAV	Silicon	XRF, GRAV
Calcium	XRF, GRAV	Silver	INAA, XRF, GRAV
Chlorine	INAA	Sodium	INAA, GRAV
Chromium	INAA, ICPMS, XRF, GRAV	Tin	INAA, XRF, GRAV
Copper	ICPMS, XRF, GRAV	Titanium	XRF, GRAV
Iron	XRF, GRAV	Vanadium	ICPMS, XRF, GRAV
Magnesium	ICPMS, XRF, GRAV	Zinc	INAA, ICPMS, XRF, GRAV
Manganese	ICPMS, XRF, GRAV		

ICPMS Inductively coupled plasma mass spectrometry at NIST

INAA Instrumental neutron activation analysis at NIST

XRF X-ray fluorescence spectrometry at NIST

GRAV Gravimetric preparation of the SRM from assayed concentrates. Performed by Conostan Division, Conoco Specialty Products, Inc.

## REFERENCES

- [1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at: [http://ts.nist.gov/WeightsAndMeasures/Metric/mpo\\_pubs.cfm](http://ts.nist.gov/WeightsAndMeasures/Metric/mpo_pubs.cfm).
- [2] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, U.S. Government Printing Office: Washington, DC (2000); Available at [http://www.cstl.nist.gov/nist839/NIST\\_special\\_publications.htm](http://www.cstl.nist.gov/nist839/NIST_special_publications.htm).
- [3] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); 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://physics.nist.gov/Pubs/>.
- [4] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results From Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105; p. 571-579 (2000).

**Certificate Revision History:** 13 January 2009 (Extension of period of validity and technical revisions to remove certified values for four elements [Mn, Mo, Sn, and Ba] due to lack of stability and to change the four elements to information values); 09 August 2000 (Original certificate date).

*Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*