National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1085c

Wear Metals in Lubricating Oil

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis of engine lubricating oils and other materials of similar matrix for elemental content. It can be used to validate value assignment of in-house reference materials. A unit of SRM 1085c consists of 10 amber borosilicate ampoules, each containing approximately 1.2 g of oil: five ampoules of a blend of constituent elements in a base oil at a nominal mass fraction of 300 mg/kg; and five ampoules of the matching base oil intended for use as an analytical blank and for matrix matching.

Certified Mass Fraction Values: The certified mass fraction values for constituents in SRM 1085c are listed in Table 1 [1]. Value assignment categories are based on the definitions of terms and modes used at NIST for certification of chemical reference materials [2]. 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. A certified value is the present best estimate of the true value.

Reference Mass Fraction Value: A reference mass fraction value for SRM 1085c is given in Table 2. A reference value is a non-certified value that is the present best estimate of the true value; however, the value does not meet the NIST criteria for certification [2] 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 agreement among multiple analytical methods.

Information Mass Fraction Values: An information mass fraction value for SRM 1085c is listed in Table 3. Information values for the mass fractions of elements in the matching base oil are given in Table 5. 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. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1085c** is valid, within the measurement uncertainty specified, until **01 October 2024**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). Accordingly, periodic recalibration or recertification of this SRM is not required. 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, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of technical measurements for the certification of this SRM was performed by J.R. Sieber of the NIST Chemical Sciences Division.

Measurements for value assignment of SRM 1085c were performed by A.F. Marlow, R. Oflaz, R.L. Paul, S.A. Rabb, D. Sahin, and J.R. Sieber of the NIST Chemical Sciences Division. Additional measurements were performed by SCP Science, Baie D'Urfé (Quebec, Canada).

Statistical consultation for this SRM was provided by A.L. Pintar of the NIST Statistical Engineering Division.

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Certificate Issue Date: 26 January 2016

Steven J. Choquette, Acting Director
Office of Reference Materials

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

INSTRUCTIONS FOR USE

To relate analytical determinations to the values in this Certificate of Analysis, a minimum sample quantity of 200 mg is recommended. The blank base oil ampoules should be used for the preparation of analytical blanks. To prevent contamination, do not return oil to an ampoule after removal. Any unused portion of an opened ampoule cannot be stored for future use and should be disposed of properly. Store the material in its original packaging at room temperature, away from light, and protected from temperature extremes. This material is not intended for frequent use as a routine quality assurance material.

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

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

PREPARATION AND ANALYSIS[(1)]

The material for this SRM and the matching base oil were provided by SCP Science, Baie D'Urfé (Quebec, Canada). The oil blend was prepared by gravimetrically blending assayed, single-element concentrates into mineral base oil. Multiple containers of the formulated oil were blended and ampoules were filled and sealed under argon at NIST. Homogeneity testing was performed at NIST using prompt gamma-ray activation analysis for boron and X-ray fluorescence spectrometry for the remaining elements, except chlorine. The homogeneity was found to be satisfactory at sample masses of 200 mg and greater. Aluminum, calcium and potassium showed some variance among ampoules, which was captured in the estimates of uncertainty.

Certified Mass Fraction Values: The certified mass fraction values and 95 % coverage intervals for SRM 1085c are given in Table 1. Each measurand is the mass fraction of the total amount of each element in lubricant base oil. Each certified value is an estimate of the average of the measurand over all ampoules. The estimate comes from fitting a statistical model to the results of measurements using the test methods listed in Table 4. The Bayesian paradigm was used for statistical inference [3]. The expanded uncertainty interval is calculated in a manner consistent with the ISO/JCGM Guides [4,5], and it expresses contributions from all recognized sources of uncertainty, including differences between analytical methods, dispersion of values from sample preparation and replicated measurement, instrument calibration, and balance calibration. The certified mass fraction values are metrologically traceable to the SI derived unit for mass fraction expressed as milligrams per kilogram.

[(1)] Certain commercial equipment, instrumentation, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institutes of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 1. Certified Mass Fraction Values for SRM 1085c Wear Metals in Lubricating Oil

Constituent	Mass Fraction (mg/kg)	95 % Coverage Interval (mg/kg)
Aluminum (Al)	292	264 to 321
Barium (Ba)	306	296 to 317
Boron (B)	304	301 to 308
Cadmium (Cd)	301	291 to 310
Calcium (Ca)	299	269 to 330
Chromium (Cr)	302	289 to 314
Copper (Cu)	298	287 to 310
Iron (Fe)	301	289 to 313
Lead (Pb)	303	294 to 312
Magnesium (Mg)	300	294 to 305
Manganese (Mn)	299	289 to 311
Molybdenum (Mo)	305	295 to 316
Nickel (Ni)	306	295 to 316
Phosphorus (P)	304	302 to 307
Potassium (K)	295	282 to 309
Silicon (Si)	293	288 to 297
Silver (Ag)	298	290 to 309
Sodium (Na)	300	289 to 310
Tin (Sn)	298	291 to 306
Titanium (Ti)	300	288 to 311
Zinc (Zn)	285	273 to 296

Reference Mass Fraction Value: A reference mass fraction value and 95 % coverage interval for vanadium (V) in SRM 1085c are given in Table 2. The measurand is the mass fraction of the vanadium in lubricant base oil. The reference value is interpreted as an estimated mean, where the mean is taken over all ampoules. The estimate comes from fitting a statistical model to the results of measurements using the test methods listed in Table 4. The Bayesian paradigm was used for statistical inference [3]. The expanded uncertainty interval is calculated in a manner consistent with the ISO/JCGM Guides [4,5], and it expresses contributions from all recognized sources of uncertainty, including differences between analytical methods, dispersion of values from sample preparation and replicated measurement, instrument calibration, and balance calibration. The reference value is metrologically traceable to the SI derived unit for mass fraction expressed as milligrams per kilogram.

Table 2. Reference Mass Fraction Value for SRM 1085c Wear Metals in Lubricating Oil

Constituent	Mass Fraction (mg/kg)	95 % Coverage Interval (mg/kg)
Vanadium (V)	285	278 to 291

Information Mass Fraction Values: An information mass fraction value for chlorine (Cl) in SRM 1085c is given in Table 3 based on an estimate obtained by instrumental neutron activation analysis at NIST. No uncertainty is provided because there is insufficient information available for its assessment. Information values for the blank base oil are given in Table 5. The blank base oil was characterized as part of the development of SRM 1085b [6].

Table 3. Information Mass Fraction Value for SRM 1085c Wear Metals in Lubricating Oil

Constituent	Mass Fraction (mg/kg)
Chlorine (Cl)	120

Table 4. Test Methods Used in Analyses of SRM 1085c Wear Metals in Lubricating Oil

Gravimetric Preparation from Assayed Concentrates: All elements, except Cl
Inductively Coupled Plasma Optical Emission Spectrometry: P, Si
Instrumental Neutron Activation Analysis: Al, Cl, Cu, Mn, Na, Ti, V, Zn
Prompt Gamma-Ray Activation Analysis: B
X-Ray Fluorescence Spectrometry: Ag, Ba, Ca, Cd, Cr, Fe, K, Mg, Mn, Mo, Ni, Pb, Sn

Table 5. Information Mass Fractions for the Matching Base Oil Packaged with SRM 1085c

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Aluminum (Al)	< 7	Manganese (Mn)	< 0.04
Arsenic (As)	< 0.02	Molybdenum (Mo)	< 0.02
Barium (Ba)	≤ 0.2	Sodium (Na)	≤ 0.3
Cadmium (Cd)	< 0.002	Nickel (Ni)	< 1
Calcium (Ca)	< 5	Phosphorus (P)	< 5
Chlorine (Cl)	≤ 0.7	Silicon (Si)	< 5
Chromium (Cr)	< 0.07	Silver (Ag)	< 0.2
Copper (Cu)	< 0.09	Tin (Sn)	< 8
Iron (Fe)	< 2	Titanium (Ti)	< 1
Lead (Pb)	< 0.03	Vanadium (V)	< 0.005
Magnesium (Mg)	≤ 0.3	Zinc (Zn)	< 0.15

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 www.nist.gov/pml/pubs/index.cfm/ (accessed Jan 2016).

[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 www.nist.gov/srm/publications.cfm (accessed Jan 2016).

[3] Gelman, A.; Carlin, J.B.; Stern, H.S.; Dunson, D.B.; Vehtari, A.; Rubin, D.B.; *Bayesian Data Analysis*; 3rd ed., CRC Press (2014).

[4] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jan 2016); 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 www.nist.gov/pml/pubs/tn1297/index.cfm (accessed Jan 2016).

[5] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to the Expression of Uncertainty in Measurement – Propagation of Distributions Using a Monte Carlo Method*; JCGM (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Jan 2016).

[6] SRM 1085b; *Wear Metals in Lubricating Oil*, National Institute of Standards of Technology; U.S. Department of Commerce, NIST: Gaithersburg, MD (13 January 2009); available at https://www-s.nist.gov/srmors/view_detail.cfm?srm=1085B (accessed Jan 2016).

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 http://www.nist.gov/srm.