National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1486

Bone Meal

This Standard Reference Material (SRM) is intended primarily for use in evaluating analytical methods used for the determination of selected major, minor, and trace elements in bone and in material of a similar matrix. It consists of steamed bone meal that was sieved and blended to a high degree of homogeneity. A unit of SRM 1486 consists of approximately 50 g of bone meal.

Certified Mass Fraction Values: Certified mass fraction values for constituent elements on a dry-mass basis, are provided 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 [1]. The measurands are the total concentrations of the elements reported in Table 1. Metrological traceability is to the SI unit of mass expressed as derived unit of mass fraction.

Reference Mass Fraction Value: A reference mass fraction value for mercury is provided in Table 2. Reference values are non-certified values that are the best estimate of the true value; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties 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 [1]. The measurand is the concentration of mercury, as determined by the methods indicated in the text. Metrological traceability is to the SI unit of mass expressed as derived unit of mass fraction.

Information Values: Information values for additional constituents are provided in Table 3. An information value is considered to be a value that will be of interest and use to the SRM user, but for which insufficient information is available to assess adequately the uncertainty associated with the value, or only a limited number of analyses were performed [1]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1486** is valid, within the measurement uncertainty specified, until **01 October 2025**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for 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 or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification of SRM 1486 were provided by S.E. Long of the NIST Chemical Sciences Division and W.F. Koch formerly of the NIST Inorganic Analytical Research Division.

Analytical measurements at NIST were made by C.E. Bryan, B.L. Catron, S.E. Long, T.W. Vetter, R.D. Vocke, and L.J. Wood of the NIST Chemical Sciences Division, and by D.S. Braverman, R. Demilrap, J.D. Fassett, K.M. Garrity, R.R. Greenberg, J.R. Moody, P.J. Paulsen, P.A. Pella, T.A. Rush, J.M. Smeller, and S.F. Stone formerly of NIST.

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Certificate Issue Date: 17 February 2017
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Statistical consultation was provided by S.B. Schiller, L.M. Oakley, and J.H. Yen of the NIST Statistical Engineering Division.

Additional collaborating laboratory measurements were made by A.R. Byrne (Josef Stefan Institute, Ljubljana, Slovenia); N. Miller-Ihli (Nutrient Composition Laboratory, U.S. Department of Agriculture, Beltsville, MD); J.B. Bodkin (College of Earth and Mineral Sciences, Mineral Characterization Laboratory, The Pennsylvania State University, University Park, PA); and B. Dainowski (University of Alaska Fairbanks, Fairbanks, AK).

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

INSTRUCTIONS FOR STORAGE AND USE

Storage: SRM 1486 must be stored in its original bottle, tightly capped and away from sunlight or ultraviolet radiation.

Use: Prior to use, the contents of the bottle should be thoroughly mixed by gently rotating the bottle by hand and inverting several times. The mass fractions of constituents in SRM 1486 are reported on a dry-mass basis. A separate sub-sample should be removed from the bottle at the time of analysis and dried (see "Instructions for Drying") to determine a moisture correction factor. Correction for moisture is to be made to the data before comparison with the certified values.

Instructions for Drying: Samples should be dried under vacuum for 24 h, or for 2 h at 105 °C in a conventional drying oven to obtain a correction factor for moisture. The mass loss determined at NIST using this method was approximately 2.4 %. The mass loss determined by the user may be different, depending on ambient conditions when the bottle is sampled.

SOURCE, PREPARATION AND ANALYSIS[(1)]

Source and Preparation of Material: The material for SRM 1486 was obtained from the Espoma Company (Millville, NJ). The entire material lot was sieved through a nominal 355 µm sieve (45 mesh), blended in a cone blender, and radiation sterilized.

Homogeneity: Samples from randomly selected bottles of SRM 1486 were tested for homogeneity using X-ray fluorescence spectrometry. No evidence of material heterogeneity was observed in any of the elements measured, which included strontium, zinc, copper, iron, phosphorus, calcium and potassium.

Certification Analyses: The certified mass fraction values are the weighted means of method results from a primary analytical method, or the weighted means of results from at least two independent analytical methods or laboratories. The uncertainty of the certified values is a two-sided 95 % confidence interval for the mean (coverage factor $k = 2$).

The reference mass fraction value for mercury is the mean of results from a single primary method [2,3]. The uncertainty provided is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence, consistent with the ISO/JCGM Guide [4]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is the combined uncertainty and k is the coverage factor ($k = 2.36$) corresponding to approximately 95 % confidence.

Elements other than those certified are present in this material. Those that were determined, but not certified, are provided as additional information values on the composition.

[(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.

Table 1. Certified Mass Fraction Values (Dry-Mass Basis) for SRM 1486

Constituent Element	Mass Fraction (%)			Constituent Element	Mass Fraction (mg/kg)		
Calcium (Ca)[a,b,c]	26.58	±	0.24	Iron (Fe)[d,e]	99	±	8
Magnesium (Mg)[b,f]	0.466	±	0.017	Lead (Pb)[e]	1.335	±	0.014
Phosphorus (P)[a,d]	12.30	±	0.19	Potassium (K)[e,g]	412	±	4
				Strontium (Sr)[e,g]	264	±	7
				Zinc (Zn)[d,e]	147	±	16

[a] Gravimetry
[b] Instrumental neutron activation analysis (INAA)
[c] Titrimetry
[d] Inductively coupled plasma optical emission spectrometry (ICP-OES)
[e] Isotope dilution thermal ionization mass spectrometry (ID TIMS)
[f] Isotope dilution inductively coupled plasma mass spectrometry (ID ICPMS)
[g] Flame atomic emission spectrometry (FAES)

Table 2. Reference Mass Fraction Value (Dry-Mass Basis) for SRM 1486

Constituent Element	Mass Fraction (mg/kg)		
Mercury (Hg)[a]	0.0023	±	0.0014

[a] Isotope dilution cold vapor inductively coupled plasma mass spectrometry (ID CVICPMS) [2,3]

Table 3. Information Values (Dry-Mass Basis) for SRM 1486

Constituent Element	Mass Fraction (%)	Constituent Element	Mass Fraction (mg/kg)
Carbon, Total (C)	18.6	Aluminum (Al)	<1
Silicon (Si)	<0.02	Arsenic (As)	0.006
Sodium (Na)	0.5	Cadmium (Cd)	0.003
		Copper (Cu)	0.8
Loss on Ignition at	31.5	Fluorine (F)	800
1000 °C		Manganese (Mn)	1
		Selenium (Se)	0.13

REFERENCES

[1] 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 (2000); available at http://www.nist.gov/srm/upload/SP260-136.pdf (accessed Feb 2017).

[2] Christopher, S.J.; Long, S.E.; Rearick, M.S.; Fassett, J.D.; *Development of Isotope Dilution Cold Vapor Inductively Coupled Plasma Mass Spectrometry and Its Application to the Certification of Mercury in NIST Standard Reference Materials*; Anal. Chem., Vol. 73, pp. 2190–2199 (2001).

[3] Long, S.E.; Kelly, W.R.; *Determination of Mercury in Coal by Isotope Dilution Cold-Vapor Generation Inductively Coupled Plasma Mass Spectrometry*; Anal. Chem., Vol. 74, pp. 1477–1483 (2002).

[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 (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Feb 2017); 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/pml/pubs/index.cfm (accessed Feb 2017).

Certificate Revision History: 17 February 2017 (Change of expiration date, addition of a reference value for mercury, editorial changes); 18 December 1992 (Original certificate issue 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 http://www.nist.gov/srm.