



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2586

Trace Elements in Soil Containing Lead from Paint (Nominal 500 mg/kg Lead)

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and for the calibration of apparatus used to determine lead and other trace elements in soil. SRM 2586 is a blended mixture of soil samples collected from urban areas where the principal source of lead is believed to be from old house lead-based paint. All values are reported as mass fractions [1], on a dry-mass basis and are based on measurements using a sample mass of at least 200 mg. A unit consists of approximately 55 g of material with a particle size of <75 μm (200 mesh).

Certified Mass Fraction Values: The certified values for four environmentally important elements 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 accounted for by NIST.

Reference Mass Fraction Values: Reference values for 18 additional elements are provided in Table 2. Reference values are noncertified 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 and may not include all sources of uncertainty.

Information Concentration Values: Information values are listed in Table 3. These values may be of interest to the SRM user; however, insufficient information is available to adequately assess the uncertainty associated with these values.

Expiration of Certification: The certification of **SRM 2586** is valid, within the uncertainty specified, until **30 September 2023**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage, Handling, 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.

Partial financial support for the development of this SRM was provided by the U.S. Environmental Protection Agency (EPA) under the direction of project manager B. Schumacher of the EPA National Exposure Research Laboratory, Las Vegas, NV.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by R.L. Watters, Jr. and G.C. Turk of the NIST Chemical Sciences Division.

Statistical consultation was provided by L.M. Gill of the NIST Statistical Engineering Division.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 11 March 2013
Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
Office of Reference Materials

INSTRUCTIONS FOR STORAGE, HANDLING, AND USE

Use: To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample mass of 200 mg should be used and the sample should be dried according to the “Instructions for Drying”. Sample preparation procedures should also be designed to effect complete dissolution in order to relate the determined value to the certified value. The SRM must be stored in its original bottle at temperatures less than 30 °C away from fumes and direct sunlight.

Instructions for Drying: Samples should be oven dried for 2 h at 105 °C. For the determination of volatile elements (arsenic and mercury), samples should be analyzed as received; separate samples should be dried according to these instructions to obtain a correction factor for moisture. Moisture corrections are then made to the measurement results before comparing them to the certified values. This alternative drying method may also be used for nonvolatile elements.

Certified Mass Fraction Values and Uncertainties: Certified values and uncertainties are provided in Table 1. The certified values are based on results from two or more NIST independent analytical methods and additional results from methods used at the U.S. Geological Survey (USGS) (Table 4). All values are reported as mass fractions [1], on a dry basis (see “Instructions for Drying”), and are based on measurements using a sample mass of at least 200 mg.

Table 1. Certified Mass Fractions

Element	Mass Fraction mg/kg
Arsenic	8.7 ± 1.5
Cadmium	2.71 ± 0.54
Chromium	301 ± 45
Lead	432 ± 17

The certified values are equally weighted means from the combination of results provided by NIST and USGS. The uncertainty is calculated as, $U = ku_c + B$. The quantity u_c is the combined standard uncertainty calculated according to the ISO and NIST Guides [2] and is intended to represent, at the level of one standard deviation, the combined effect of within-method variation and material inhomogeneity. The coverage factor, k , is determined from the Student- t distribution corresponding to the calculated effective degrees of freedom and 95 % level of confidence for each element. B is a bias adjustment for the difference between methods, which is the maximum difference between the certified value and method means [3].

Reference Mass Fraction Values and Uncertainties: Reference values for concentrations of 18 elements are given in Table 2. The reference value for Hg is derived from a single method performed at NIST. The reference values for the remaining elements are derived from two or more methods performed at NIST and/or USGS.

Table 2. Reference Mass Fractions

Element	Mass Fraction mg/kg	Element	Mass Fraction mg/kg
Aluminum	66 520 ± 760	Neodymium	26.4 ± 2.9
Barium	413 ± 18	Phosphorus	1 001 ± 77
Calcium	22 180 ± 540	Potassium	9 760 ± 180
Cerium	58 ± 8	Silicon	291 500 ± 2 100
Iron	51 610 ± 890	Sodium	4 680 ± 730
Lanthanum	29.7 ± 4.8	Strontium	84.1 ± 8.0
Magnesium	17 070 ± 840	Titanium	6 050 ± 660
Manganese	1 000 ± 18	Ytterbium	2.64 ± 0.51
Mercury	0.367 ± 0.038	Zinc	352 ± 16

The reference values are equally weighted means from the combination of results provided by NIST and USGS. The uncertainty is calculated as, $U = ku_c + B$. The quantity u_c is the combined standard uncertainty calculated according

to the ISO Guide [2] and is intended to represent, at the level of one standard deviation, the combined effect of within-method variation and material inhomogeneity. The coverage factor, k , is determined from the Student- t distribution corresponding to the calculated effective degrees of freedom and 95 % level of confidence for each element. B is a bias adjustment for the difference between methods, which is the maximum difference between the certified value and method means [3].

Information Concentration Values: Information values are provided in Table 3. The values are either from a single method or are the mean of two methods.

Table 3. Information Mass Fractions

Element	Mass Fraction mg/kg	Element	Mass Fraction mg/kg
Beryllium	1.4	Nickel	75
Cobalt	35	Niobium	6
Copper	81	Praseodymium	7.3
Dysprosium	5.4	Samarium	6.1
Erbium	3.3	Scandium	24
Europium	1.5	Selenium	0.6
Gadolinium	5.8	Terbium	0.9
Gallium	14	Thorium	7
Holmium	1.1	Thulium	0.5
Lithium	25	Vanadium	160
		Yttrium	21

COLLECTION, PREPARATION, AND ANALYSIS

Collection: Soil material used in the preparation of SRM 2586 was derived from approximately 20 000 samples collected in the Baltimore, MD area as part of an EPA study conducted in 1990. Selection and classification of the soil samples used for this SRM were coordinated through R. Fahy of the Maryland Department of Public Health.

Preparation: The preparation of SRM 2586 was performed at the USGS laboratory in Denver, CO, under the direction of S.A. Wilson. The 20 000 soil samples were initially combined in 10 separate 40 L containers, the contents of which were blended and chemically analyzed. This information was then used to combine and blend the sub-sets into a single set with a target lead concentration of 500 mg/kg. The blended mixture was ground to <75 μm (200 mesh) using a Hardinger⁽¹⁾ ball mill equipped with an air separator system and mixed for 20 h using a cross-flow V-blender. The material was then split into 8 kg aliquots and sterilized using Co-60 irradiation. After sterilization the material was re-combined, blended for 3 h, and bottled.

Analysis: Certification analyses were performed in the NIST Chemical Sciences Division and at USGS. The analytical methods used and the analysts are listed in Table 4.

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

Table 4. Methods Used for the Analysis of SRM 2586

Method	Lab	Analysts	Elements ^(a)
Electrothermal Atomic Absorption Spectrometry	NIST	B. Buehler, M.S. Epstein	As, Cr
Flow Injection – Cold Vapor Atomic Absorption Spectrometry	NIST	M.S. Epstein	Hg
Inductively Coupled Plasma Mass Spectrometry	NIST	L.L. Yu	As, Cd
Instrumental Neutron Activation Analysis	NIST	R. Zeisler	As, Cr
Isotope Dilution – Inductively Coupled Plasma Mass Spectrometry	NIST	E.S. Beary, K.E. Murphy	Cd, Pb
X-Ray Fluorescence Spectrometry with Fusion Sample Preparation, Calibrated with Fusions of Mixed Pure Element Compounds	NIST	P.A. Pella, A.F. Marlow, E. Ramirez (Guest Scientist from Centro Nacional de Metrologia, Mexico)	Al, Ba, Ca, Cr , Fe, K, Mg, Mn, P, Pb , Si, Sr, Ti, Zn
Hydride Generation Atomic Absorption Spectrometry	USGS	P. Hageman	As, Se
Inductively Coupled Plasma Atomic Emission Spectrometry	USGS	P.H. Briggs	Al, Ba, Be, Ca, Ce, Co, Cr , Cu, Fe, Ga, K, La, Li, Mg, Mn, Na, Nb, Nd, Ni, P, Pb , Sc, Sr, Th, Ti, V, Y, Yb, Zn
Inductively Coupled Plasma Mass Spectrometry	USGS	A. Meier	Ce, Dy, Er, Eu, Gd, Ho, La, Nd, Pr, Sm, Tb, Tm, Yb
X-Ray Fluorescence Spectrometry with Fusion Sample Preparation, Calibrated with Fusions of Geological Reference Materials	USGS	S.A. Wilson, J.S. Mee, D.F. Siems	Al, Ca, Fe, K, Mg, Na, P, Si, Ti

^(a)Certified Elements shown in **Bold**.

User Experience with SRM 2586: In order to demonstrate user experience with SRM 2586, a number of laboratories analyzed this material, using a variety of dissolution and instrumental methods. For lead, this was done through the Environmental Lead Proficiency Analytical Testing Program (ELPAT), where SRM 2586 was included as an unknown for Round Robin number 13. Data for As, Cd, Cr, and Hg were supplied by volunteer laboratories in a round robin exercise organized by NIST. The sample preparation methods included EPA-SW846-3050A, EPA-SW846-3051 [4] and others. As these methods may not effect complete sample dissolution, the results obtained using these methods tend to be lower than the certified values. These results were not used in calculating the certified values of SRM 2586.

Table 5. Results of Round Robin Exercise

Element	Mean mg/kg	Minimum mg/kg	Maximum mg/kg	Standard Deviation mg/kg	N
As	6.7	4.7	10.4	1.6	20
Cd ^(a)	2.3	1.2	3.3	0.5	15
Cr	114	57	156	25	23
Hg	0.30	0.12	0.40	0.07	20
Pb	401.2	336.5	472	37.8	67

^(a)The results reported for Cd from five laboratories were erroneously high and are not included in the summary statistics given here.

REFERENCES

- [1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*, NIST Special Publication 811, 1995 ed. (1995); available at <http://www.nist.gov/pml/pubs/index.cfm> (accessed Mar 2013).
- [2] 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 http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Mar 2013); 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 Mar 2013).
- [3] Schiller, S.B.; Eberhardt, K.R.; *Combining Data from Independent Chemical Analysis Methods*, *Spectrochimica Acta*, Vol. 46B, pp. 1607–1613 (1991).
- [4] Federal Register 1-13-95 SW-846 update #2, Final.

Certificate Revision History: 11 March 2013 (Extension of the certification period; editorial changes); 24 June 2008 (Update of expiration date and editorial changes); 03 March 1999 (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 <http://www.nist.gov/srm>.