



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2586

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

This Standard Reference Material (SRM) is intended for use in evaluating analytical methods for the determination of lead, trace elements, and perfluorooctane sulfonate (PFOS) 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 of SRM 2586 consists of approximately 55 g of material with a particle size of <75 μm (200 mesh).

Certified Mass Fraction Values: The certified mass fraction 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 [2]. The measurands are the certified mass fractions of the elements in Table 1 and are metrologically traceable to the SI unit of mass fraction expressed as milligrams per kilogram.

Reference Mass Fraction Values: Reference mass fraction values for additional elements are provided in Table 2. The reference mass fraction value for PFOS is provided in Table 3. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet NIST criteria for certification [2] and are provided with associated uncertainties that may reflect only measurement reproducibility and may not include all sources of uncertainty or may reflect a lack of sufficient statistical agreement determined by the methods indicated. The measurands are the mass fractions listed in Tables 2 and 3, as determined by the methods indicated, and are metrologically traceable to the SI unit of mass fraction expressed as milligrams per kilogram.

Information Mass Fraction Values: Information mass fraction values are listed in Table 4 for additional elements. An information value may be of interest and use to the SRM user; however, insufficient information is available to adequately assess the uncertainty associated with these values [2]. Information values cannot be used to establish metrological traceability.

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 or register online) will facilitate notification.

Overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by R.L. Watters, Jr. of the NIST Office of Reference Materials and G.C. Turk and C.E. Bryan of the NIST Chemical Sciences Division.

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).

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Statistical consultation was provided by L.M. Gill and J.H. Yen of the NIST Statistical Engineering Division.

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

INSTRUCTIONS FOR STORAGE, HANDLING, AND USE

Storage: SRM 2586 is packaged as a dry material in glass bottles. The SRM must be stored in its original bottle at temperatures less than 30 °C away from fumes and direct sunlight.

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.

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.

COLLECTION, PREPARATION, AND ANALYSIS⁽¹⁾

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 U.S. Geological Survey (USGS) laboratory (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 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 ⁶⁰Co 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, at USGS, and during an interlaboratory comparison. The analytical methods used and the analysts are listed in Table 5. All values are reported as mass fractions [2], on a dry-mass basis (see “Instructions for Drying”), and are based on measurements using a sample mass of at least 200 mg.

Certified Mass Fraction Values and Uncertainties: Certified values and uncertainties for elements in SRM 2586 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 USGS (Table 5).

Table 1. Certified Mass Fractions (Dry-Mass Basis) for Elements in SRM 2586

Element	Mass Fraction mg/kg
Arsenic (As)	8.7 ± 1.5
Cadmium (Cd)	2.71 ± 0.54
Chromium (Cr)	301 ± 45
Lead (Pb)	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/JCGM and NIST Guides [3] 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

⁽¹⁾ Certain commercial equipment, instrumentation, or materials are identified 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.

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 [4].

Reference Mass Fraction Values for the Elements: Reference values and uncertainties for elements in SRM 2586 are given in Table 2. The reference values for Ce, Dy, Eu, Gd, Hg, La, Nd, Pr, Sm, Y, and Yb are 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 5).

Table 2. Reference Mass Fractions (Dry-Mass Basis) for Elements in SRM 2586

Element	Mass Fraction mg/kg			Element	Mass Fraction mg/kg		
Aluminum (Al)	66 520	±	760	Neodymium (Nd)	27.2	±	0.8
Barium (Ba)	413	±	18	Phosphorus (P)	1 001	±	77
Calcium (Ca)	22 180	±	540	Potassium (K)	9 760	±	180
Cerium (Ce)	57.1	±	1.9	Praseodymium (Pr)	6.78	±	0.23
Dysprosium (Dy)	4.69	±	0.14	Samarium (Sm)	6.02	±	0.14
Europium (Eu)	1.33	±	0.03	Silicon (Si)	291 500	±	2 100
Gadolinium (Gd)	6.04	±	0.27	Sodium (Na)	4 680	±	730
Iron (Fe)	51 610	±	890	Strontium (Sr)	84.1	±	8.0
Lanthanum (La)	27.0	±	1.0	Terbium (Tb)	0.79	±	0.02
Magnesium (Mg)	17 070	±	840	Titanium (Ti)	6 050	±	660
Manganese (Mn)	1 000	±	18	Ytterbium (Yb)	2.06	±	0.05
Mercury (Hg)	0.367	±	0.038	Yttrium (Y)	20.5	±	0.8
				Zinc (Zn)	352	±	16

The element reference values derived from a single method at NIST are the means of the measurements for the individual method. The expanded uncertainty is calculated as, $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO/JCGM Guide [3], and k is the coverage factor corresponding to approximately 95 % confidence.

The element reference values derived from two or more methods 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 [3] 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 [4].

Reference Mass Fraction Value for Perfluorooctane Sulfonate (PFOS): The reference value and uncertainty for PFOS in SRM 2586 is provided in Table 3. The mass fraction for PFOS is based on three sets of data (two sets from NIST and one set from an interlaboratory study) (Table 5).

Table 3. Reference Mass Fraction (Dry-Mass Basis) for Perfluorooctane Sulfonate (PFOS) in SRM 2586

	Mass Fraction µg/kg
Perfluorooctane Sulfonate (PFOS)	3.41 ± 1.33

The reference value is the equally weighted mean from the combination of results provided by NIST and the interlaboratory study. The uncertainty of each method mean is the standard deviation of that mean. The expanded uncertainty of the combined mean is calculated as, $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO/JCGM Guide [3], and k is the coverage factor corresponding to approximately 95 % confidence. The uncertainty of the combined mean is calculated using a bootstrap procedure based on a Gaussian random effects model for the between-method effects [3,5–7].

Information Mass Fraction Values: Information values for elements in SRM 2586 are provided in Table 4. The values are either from a single method or are the mean of two methods (Table 5).

Table 4. Information Mass Fractions (Dry-Mass Basis) for Elements

Element	Mass Fraction mg/kg	Element	Mass Fraction mg/kg
Beryllium (Be)	1.4	Nickel (Ni)	75
Cobalt (Co)	35	Niobium (Nb)	6
Copper (Cu)	81	Scandium (Sc)	24
Erbium (Er)	3.3	Selenium (Se)	0.6
Gallium (Ga)	14	Thorium (Th)	7
Holmium (Ho)	1.1	Thulium (Tm)	0.5
Lithium (Li)	25	Vanadium (V)	160

Table 5. Methods Used for the Analysis of SRM 2586

Method	Lab	Analysts	Elements ^(a)
Electrothermal Atomic Absorption Spectrometry (AAS)	NIST	B. Buehler, M.S. Epstein	As, Cr
Flow Injection – Cold Vapor Atomic Absorption Spectrometry (CVAAS)	NIST	M.S. Epstein	Hg
Inductively Coupled Plasma Mass Spectrometry (ICP-MS)	NIST	L.L. Yu	As, Cd
Inductively Coupled Plasma Mass Spectrometry (ICP-MS)	NIST	C.E. Bryan, S.E. Long	Ce, Dy, Eu, Gd, La, Nd, Pr, Sm, Tb, Y, Yb,
Instrumental Neutron Activation Analysis (INAA)	NIST	R. Zeisler	As, Cr
Isotope Dilution – Inductively Coupled Plasma Mass Spectrometry (ID-ICP-MS)	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 (AAS)	USGS	P. Hageman	As, Se
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	USGS	P.H. Briggs	Al, Ba, Be, Ca, Co, Cr , Cu, Fe, Ga, K, Li, Mg, Mn, Na, Nb, Ni, P, Pb , Sc, Sr, Th, Ti, V, Zn
Inductively Coupled Plasma Mass Spectrometry (ICP-MS)	USGS	A. Meier	Er, Ho, Tm
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
Liquid Chromatography with Triple Quad Mass Spectrometry (LC-MS/MS)	NIST	J.L. Reiner	PFOS
Liquid Chromatography with Triple Quad Mass Spectrometry (LC-MS/MS)	Interlaboratory Comparison		PFOS

^(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 [8], 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 6. Results of Round Robin Exercise in SRM 2586

Element	Mean (mg/kg)	Minimum (mg/kg)	Maximum (mg/kg)	Standard Deviation (mg/kg)	n
Arsenic (As)	6.7	4.7	10.4	1.6	20
Cadmium (Cd) ^(a)	2.3	1.2	3.3	0.5	15
Chromium (Cr)	114	57	156	25	23
Mercury (Hg)	0.30	0.12	0.40	0.07	20
Lead (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

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Certificate Revision History: 16 August 2019 (Correction of PFOS units in Table 3; editorial changes); 12 April 2017 (Change information values to reference values for Dy, Eu, Gd, Pr, Sm, Tb, and Y and update reference values for Ce, La, Nb, and Yb; addition of PFOS reference value; editorial changes); 11 March 2013 (Change of expiration date; editorial changes); 24 June 2008 (Change of expiration date; 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 <https://www.nist.gov/srm>.