National Institute of Standards and Technology
U.S. Department of Commerce

Date of Issue:
03 July 2023

Standard Reference Material® 2586

Trace Elements in Soil Containing Lead from Paint

(Nominal Mass Fraction 500 mg/kg Lead)

CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for evaluating analytical methods for the determination of 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.

Description: A unit of SRM 2586 consists of approximately 55 g of material with a particle size of <75 µm (200 mesh).

Certified Values: Certified values and uncertainties for four environmentally important elements in SRM 2586 are provided in Table 1. A 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 [1]. The certified mass fractions of the elements in Table 1 are metrologically traceable to the International System of Units (SI) unit of mass fraction expressed as milligrams per kilogram.

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

Element	Mass Fraction [(a)] (mg/kg)
Arsenic (As)	8.7 ± 1.5
Cadmium (Cd)	2.71 ± 0.54
Chromium (Cr)	301 ± 45
Lead (Pb)	432 ± 17

[(a)] 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 [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].

Non-Certified Values: Non-certified values for additional elements and perfluorooctane sulfonate (PFOS) in soil are provided in Appendix A.

Additional Information: Values of potential interest to users, methods used for the analysis of SRM 2586, and additional information are provided in Appendix B.

Period of Validity: The certified values delivered by **SRM 2586** are valid within the measurement uncertainty specified until **30 September 2033**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

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Safety: SRM 2586 is intended for research use. Please consult the Safety Data Sheet for this product.

Storage: SRM 2586 is packaged as a dry material in glass bottles. The SRM must be stored in its original bottle at temperature room temperature (20 °C ± 10 °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" below. 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.

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REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Jul 2023).

[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 (JCGM) (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Jul 2023); 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Jul 2023).

[3] Schiller, S.B.; Eberhardt, K.R.; *Combining Data from Independent Chemical Analysis Methods,* Spectrochimica Acta, Vol. 46B, pp. 1607–1613 (1991).

[4] 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*; Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Jul 2023)

[5] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall, London, UK (1993).

[6] Searle, S.; Casella, G.; McCulloch, C.; *Variance Components*, John Wiley, Hoboken, NJ (1992).

[7] Federal Register; *Hazardous Waste Test Methods SW-846*, *Update 2* (1995).

Certificate Revision History: 03 July 2023 (Change of period of validity; updated format; editorial changes); **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).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis 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.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the SI or other higher-order reference systems. Non-certified mass fraction values and uncertainties for elements in SRM 2586 are given below in Table A1.

Table A1. Non-Certified 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 values derived from a single method at NIST are the means of the measurements for the individual method (Table B2). The expanded uncertainty is calculated as, $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO/JCGM Guide [2], and k is the coverage factor corresponding to approximately 95 % confidence.

The element values derived from two or more methods (Table B2) 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 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 non-certified value and method means [3].

The non-certified mass fraction value and uncertainty for PFOS in SRM 2586 is provided in Table A2

Table A2. Non-Certified Mass Fraction (Dry-Mass Basis) for Perfluorooctane Sulfonate (PFOS) in SRM 2586

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

The non-certified value for PFOS is the equally weighted mean from the combination of results provided by NIST and an interlaboratory comparison (method information in Table B2). 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 [2], 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 [2,4–6].

Period of Validity: The non-certified values are valid within the measurement uncertainty specified until **30 September 2033.** The value assignments are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Appendix and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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APPENDIX B

Values of potential interest to users: Values for elements in SRM 2586 are provided in Table B1 below. The values were measured using the methods described below (Table B2). Uncertainties for the values in Table B1 were not assigned because measurements were not made to sufficiently account for all sources of uncertainty or their magnitude.

Table B1. Mass Fractions (Dry-Mass Basis) for Elements with Values of Potential Interest to Users

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 B2. 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 [7], and others. The results are summarized in Table B3. As these methods may not affect 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 B3. Results of Round Robin Exercise in SRM 2586 with Values of Potential Interest to Users

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.

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 the Maryland Department of Public Health.

Preparation: The preparation of SRM 2586 was performed at USGS laboratory (Denver, CO. 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 ^{60}Co irradiation. After sterilization the material was re-combined, blended for 3 h, and bottled.

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