

# Standard Reference Material<sup>®</sup> 2587

## Trace Elements in Soil Containing Lead from Paint

(Nominal 3000 mg/kg Lead)

### CERTIFICATE OF ANALYSIS

**Purpose:** The certified values delivered by 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 2587 is composed of soil samples collected from a suburban garden known to have been contaminated by lead based house paint.

**Description:** A unit of SRM 2587 consists of approximately 55 g of material with a particle size of <75  $\mu\text{m}$  (200 mesh).

**Certified Values:** Certified values and uncertainties for four environmentally important elements in SRM 2587 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 2587

Element	Mass Fraction <sup>(a)</sup> (mg/kg)
Arsenic (As)	13.7 $\pm$ 2.3
Cadmium (Cd)	1.92 $\pm$ 0.23
Chromium (Cr)	92 $\pm$ 11
Lead (Pb)	3 242 $\pm$ 57

<sup>(a)</sup> The certified values are equally weighted means from the combination of results provided by NIST and USGS. The uncertainty is calculated as,  $U = k u_c + B$ . 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 in soil are provided in Appendix A.

**Additional Information:** Values of potential interest to users, methods used for the analysis of SRM 2587, and additional information are provided in Appendix B.

**Period of Validity:** The certified values delivered by **SRM 2587** 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>).

**Safety:** SRM 2587 is intended for research use. Please consult the Safety Data Sheet for this product.

**Storage:** SRM 2587 is packaged as a dry material in glass bottles. The SRM must be stored in its original bottle at 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.

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

<b>Certificate Revision History:</b> <b>03 July 2023</b> (Change of period of validity; updated format; editorial changes); <b>11 March 2013</b> (Extension of the certification period; editorial changes); <b>24 June 2008</b> (Update of expiration date and editorial changes); <b>03 March 1999</b> (Original certificate date).
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*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](mailto:srminfo@nist.gov); or the Internet at <https://www.nist.gov/srm>.*

\* \* \* \* \* **End of Certificate of Analysis** \* \* \* \* \*

# APPENDIX A

**Non-Certified Values:** Non-certified values for 14 additional elements in SRM 2587 are provided in Table A1. The value for Hg is derived from a single method performed at NIST. The values for the remaining elements are derived from two or more methods performed at NIST and/or USGS (Table B2). 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.

Table A1. Non-Certified Mass Fractions

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Aluminum (Al)	58 600 ± 1 700	Phosphorus (P)	970 ± 100
Barium (Ba)	568 ± 12	Potassium (K)	15 830 ± 550
Calcium (Ca)	9 270 ± 200	Silicon (Si)	331 300 ± 2 600
Iron (Fe)	28 130 ± 250	Sodium (Na)	11 270 ± 330
Magnesium (Mg)	6 690 ± 250	Strontium (Sr)	126 ± 19
Manganese (Mn)	651 ± 23	Titanium (Ti)	3 920 ± 650
Mercury (Hg)	0.290 ± 0.009	Zinc (Zn)	335.8 ± 7.6

The non-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 non-certified value and method means [3].

**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>).

\*\*\*\*\* End of Appendix A \*\*\*\*\*

# APPENDIX B

**Values of potential interest to users:** Values for elements in SRM 2587 are listed in Table B1. 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 for Values of Potential Interest to Users

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Beryllium (Be)	9.2	Nickel (Ni)	36
Cerium (Ce)	57	Niobium (Nb)	14
Cobalt (Co)	14	Scandium (Sc)	11
Copper (Cu)	160	Thorium (Th)	7.5
Gallium (Ga)	13	Vanadium (V)	78
Lanthanum (La)	29	Ytterbium (Yb)	1.6
Lithium (Li)	32	Yttrium (Y)	15
Neodymium (Nd)	25		

Table B2. Methods Used for the Analysis of SRM 2587

Method	Lab	Analysts	Elements <sup>(a)</sup>
Electrothermal Atomic Absorption Spectrometry	NIST	B. Buehler, M.S. Epstein	<b>As, Cr</b>
Flow Injection – Cold Vapor Atomic Absorption Spectrometry	NIST	M.S. Epstein	Hg
Inductively Coupled Plasma Mass Spectrometry	NIST	L.L. Yu	<b>As, Cd</b>
Instrumental Neutron Activation Analysis	NIST	R. Zeisler	<b>As, Cr</b>
Isotope Dilution – Inductively Coupled Plasma Mass Spectrometry	NIST	E.S. Beary, K.E. Murphy	<b>Cd, Pb</b>
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, <b>Cr</b> , Fe, K, Mg, Mn, P, <b>Pb</b> , Si, Sr, Ti, Zn
Inductively Coupled Plasma Atomic Emission Spectrometry	USGS	P.H. Briggs	Al, <b>As</b> , Ba, Be, Ca, Ce, Co, <b>Cr</b> , Cu, Fe, Ga, K, La, Li, Mg, Mn, Na, Nb, Nd, Ni, P, <b>Pb</b> , Sc, Sr, Th, Ti, V, Y, Yb, Zn
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

<sup>(a)</sup>Certified Elements shown in **Bold**.

**User Experience with SRM 2587:** In order to demonstrate user experience with SRM 2587, 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 2587 was included as an unknown for Round Robin number 12. Data for As, Cd, Cr, and Hg, were supplied by volunteer laboratories in a round robin exercise organized by NIST. The sample preparation methods include EPA-SW846-3050A, EPA-SW846-3051 [4] and others. As these methods may not affect complete sample dissolution, the results obtained using these methods tend to be lower than the certified values. The results are summarized in Table B3. These results were not used in calculating the certified values of SRM 2587.

Table B3. Results of Round Robin Exercise for Values of Potential Interest to Users

Element	Mean (mg/kg)	Minimum (mg/kg)	Maximum (mg/kg)	Std. Deviation (mg/kg)	N
As	12.2	7.1	16.6	2.2	20
Cd <sup>(a)</sup>	1.8	1.0	2.5	0.4	17
Cr	34	21	53	8	22
Hg	0.25	0.07	0.33	0.06	19
Pb	3 091	2 590	3 479	228	40

<sup>(a)</sup> The results reported for Cd from four laboratories were erroneously high and are not included in the summary statistics given here.

**Collection:** Soil material used in the preparation of SRM 2587 was collected from a suburban residence in the Hartford, CT area known to be contaminated by lead-based paint. The soil was collected within the top 25 cm after surficial organic material had been removed. Visual inspection of the collected soil revealed moderate quantities of partially decomposed organic matter as well as minor amounts of masonry debris. Uncontaminated soil from the Boston, MA area was collected for use as a diluent.

**Preparation:** The preparation of SRM 2587 was performed at the USGS laboratory (Denver, CO). The collected soils were dried at room temperature for 5 days, disaggregated, and sieved. The material with particle size under 2 mm was collected in 20 L containers and chemically analyzed. The information from this preliminary analysis was then used to combine and blend the sub-sets into a single set with a target lead concentration of 3000 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 hours 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.

\*\*\*\*\* End of Appendix B \*\*\*\*\*