National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2584

Trace Elements in Indoor Dust

(Nominal Mass Fraction of 1 % 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 dust. SRM 2584 is composed of dust collected from vacuum cleaner bags used in the cleaning of interior dwelling spaces. A unit of SRM 2584 consists of 8 g of particulate material, 99+ % of which passes a 100 μm (No. 145) sieve.

Certified Mass Fraction Values: The certified values for five elements in SRM 2584 are listed 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 in account. The certified values are based on measurements from two or more independent analytical methods or a single NIST primary method. Analytical methods used for the characterization of this SRM are given in Table 4. 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 100 mg.

Reference Mass Fraction Values: Reference values for mass fractions of 10 elements are given in Table 2. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty. The reference values and uncertainties are based on measurements from two or more analytical methods performed at NIST and/or the U.S. Geological Survey (USGS).

Information Mass Fraction Values: Information values are provided in Table 3 for the mass fractions of 22 additional elements. Information values are considered to be values that will be of interest and use to the SRM user, but for which insufficient information is available to assess the uncertainties associated with the values. The information values are based on measurements from a single analytical method. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 2584** is valid, within the measurement uncertainty specified, until **31 December 2023**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, 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.

Overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by P.A. Pella and G.C. Turk of the NIST Chemical Sciences Division.

Statistical consultation was provided by S.D. Leigh and K.R. Eberhardt of the NIST Statistical Engineering Division.

Partial financial support for the development of this SRM was provided by the U.S. Environmental Protection Agency (USEPA) under the direction of project managers S.L. Harper and M.E. Beard of the EPA Office of Research and Development, National Exposure Research Laboratory, Research Triangle Park, NC.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 29 January 2016
Certificate Revision History on Last Page

Steven J. Choquette, Acting Director
Office of Reference Materials

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Stability: This material is considered to be stable.

Use: To relate analytical determinations to the certified values in this Certificate of Analysis, a minimum sample mass of 100 mg should be used and the sample should be handled 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. This SRM must be stored in an air conditioned or similar cool and dry environment away from sunlight and fumes.

Instructions for Drying: When nonvolatile elements such as cadmium, chromium, and lead are to be determined, samples should be oven dried for 2 h at 110 °C. Volatile elements, such as arsenic and mercury, should be determined on samples as-received; separate samples should be dried according to these instructions to obtain a correction factor for moisture. Moisture corrections should then be made to measurement values before comparing them to the certified values.

Certified Mass Fractions: The certified values for lead and cadmium were determined by isotope dilution mass spectrometry (IDMS). The certified values for the remaining elements were determined by combining data from two or more independent analytical methods in the manner described by Schiller and Eberhardt [2]. Because of evidence of inhomogeneity, the uncertainties for arsenic, cadmium, and lead are each based on a 95 % prediction interval for the true value. This interval includes the combined effects of uncertainty components associated with material inhomogeneity, measurement uncertainty, and an allowance for differences between the analytical methods used [3]. The uncertainties for chromium and mercury, which exhibited no evidence of inhomogeneity, are each based on a 95 % confidence interval for the true value, including the combined effects of uncertainty components associated with measurement uncertainty and an allowance for differences between the analytical methods used. The measurands are the total mass fractions of the elements listed in Table 1 and are metrologically traceable to the SI unit for mass, expressed as milligrams per kilogram.

Table 1. Certified Mass Fractions

Element	Mass Fraction (mg/kg)		
Arsenic (As)	17.4	±	4.2
Cadmium (Cd)	10.0	±	1.1
Chromium (Cr)	135.0	±	9.1
Lead (Pb)	9761	±	67
Mercury (Hg)	5.20	±	0.24

Reference Mass Fraction Values: The uncertainties are based on a 95 % confidence interval for the true value, including the combined effect of the measurement uncertainty for each method and an allowance for differences between the analytical methods used [2]. The measurands are the mass fractions of the elements listed in Table 1 as determined by the methods listed in Table 4. The values are metrologically traceable to the SI unit for mass, expressed as milligrams per kilogram.

Table 2. Reference Mass Fractions

Element	Mass Fraction (mg/kg)		
Aluminum (Al)	23 200	±	600
Calcium (Ca)	63 300	±	3 000
Iron (Fe)	16 400	±	1 200
Potassium (K)	9 500	±	1 400
Lanthanum (La)	19	±	2
Magnesium (Mg)	15 900	±	300
Sodium (Na)	27 700	±	1 200
Phosphorus (P)	2 000	±	120
Titanium (Ti)	4 200	±	300
Zinc (Zn)	2 580	±	150

Table 3. Information Mass Fractions

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Antimony (Sb)	14	Nickel (Ni)	90
Barium (Ba)	1300	Niobium (Nb)	10
Beryllium (Be)	0.7	Rubidium (Rb)	33
Bismuth (Bi)	9	Scandium (Sc)	4
Cerium (Ce)	35	Selenium (Se)	2
Cesium (Cs)	1.4	Silicon (Si)	106000
Cobalt (Co)	10	Strontium (Sr)	160
Copper (Cu)	320	Thorium (Th)	4
Gallium (Ga)	6.4	Uranium (U)	1.6
Lithium (Li)	17	Vanadium (V)	34
Manganese (Mn)	370	Yttrium (Y)	10
Molybdenum (Mo)	5.5		

COLLECTION, PREPARATION, AND ANALYSIS[(1)]

Collection: Approximately 65 % of the material used for SRM 2584 was obtained from households in Montana, New Jersey, Ohio, and Wisconsin involved in lead poisoning intervention programs in which HEPA® vacuum cleaners were used to remove dust and other surface debris from homes where cases of lead poisoning had occurred. This material was mixed with low level material taken from the sources used for the preparation of SRM 2583, namely routine vacuum cleaner bags from households, cleaning services, motels, and hotels from North Carolina, Maryland, Ohio, and New Jersey. The vacuum cleaner bags were collected under the direction of the Research Triangle Institute (RTI) and the EPA. The collection process was coordinated by E.D. Hardison and D.A. Binstock of RTI (Research Triangle Park, NC) under the leadership of W.F. Gutknecht.

Preparation: From RTI the bags were labeled, boxed and sent to Neutron Products (Dickerson, MD) for radiation sterilization, and then shipped to NIST for processing. The initial screening and preparation to select suitable material were directed by P.A. Pella and performed by A.F. Marlow, C. Desai, and P. Seo of NIST. Final processing and blending was performed by the NIST Office of Reference Materials. The raw material from each bag was mixed and tumbled in a modified food processor using chopping blades and a compressed air jet. While still tumbling, the

[(1)] Certain commercial equipment, instruments, or materials are identified in this certificate in order 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.

dust was separated from unwanted debris by vacuuming through a series of screens into a clean HEPA vacuum cleaner. The dust collected in this manner was then screened through a 90 μm stainless steel sieve using vibration and a vacuum. Processed sub-lots of approximately 5 kg each were set aside and analyzed for lead by X-ray fluorescence in order to develop a blending protocol for the target lead concentration. Selected high and low level sub-lots were blended in a cone blender and then bottled.

Analysis: Certification analyses were performed in the NIST Chemical Sciences Division. Reference and information value analyses were performed by the USGS(Denver, CO) using inductively coupled plasma mass spectrometry (ICPMS) and wavelength dispersive X-ray fluorescence spectrometry (WDXRF) and by the NIST Chemical Sciences Division using instrumental neutron activation analysis (INAA). Analytical methods used for this SRM are given in Table 4.

Table 4. Methods Used for the Analysis of SRM 2584

Method[a]	Element[b]
CVAAS	**Hg**
HR-ICPMS	**As**, **Cr**
ICPMS (NIST)	Cd
ICPMS (USGS)	Al, Ba, Be, Bi, Ca, Ce, Co, Ca, Cu, Fe, Ga, K, La, Li, Mg, Mn, Mo, Na, Nb, Ni, P, Rb, Sb, Sc, Se, Sr, Th, Ti, U, V, Y, Zn
ID-ICPMS	Cd, Pb
ID-TIMS	**Cd**, **Pb**
INAA	**As**, **Cr**, Fe, **Hg**, La, Zn
WDXRF (USGS)	Al, Ca, Fe, K, Mg, Na, P, Si, Ti

[a] Methods:

CVAAS	Cold vapor atomic absorption spectrometry
ICPMS	Inductively coupled plasma mass spectrometry
ID-ICPMS	Isotope dilution quadrupole inductively coupled plasma mass spectrometry
ID-TIMS	Isotope dilution thermal ionization mass spectrometry
INAA	Instrumental neutron activation analysis
HR-ICPMS	High resolution inductively coupled plasma mass spectrometry
WDXRF	Wavelength dispersive X-ray fluorescence spectrometry

[b] Methods used for establishment of certified values are indicated by bold-face type.

Analysts NIST Chemical Sciences Division: C.M. Beck, J.R. Sieber, S.J. Christopher, R.D. Vocke, A.F. Marlow, L.L. Yu, K.E. Murphy, R.L. Zeisler, M.S. Rearick.

User Experience with SRM 2584

In order to demonstrate user experience with SRM 2584, a number of laboratories analyzed this material, each using its typical method. For lead, this was done through the Environmental Lead Proficiency Analytical Testing Program (ELPAT), where SRM 2584 was loaded onto dust wipes and included as an unknown for ELPAT Round Number 022. The ELPAT results have been converted from μg/wipe to mg/kg. Data for arsenic, cadmium, chromium, and mercury were supplied by volunteer laboratories in a round robin exercise organized by NIST. For these elements the SRM was analyzed directly. Among the participants, the range of digestion procedures used included various standard and in house hotplate, microwave, hot block, and water bath methods. Instrumental methods included ICPMS, ICP Atomic Emission Spectrometry, Graphite Furnace AAS, Flame AAS, and CVAAS. The results from this study were not used in calculating the certified values of SRM 2584. The results are given in Table 5 below. The summary statistics are based on 118 reported results for lead and 13 to 16 results for the other elements.

Table 5. Results of Round Robin Exercise

Element	Mean (mg/kg)	Minimum (mg/kg)	Maximum (mg/kg)	s[a] (mg/kg)
As	21.0	17.5	40.2	7.2
Cd	9.8	8.2	13.9	1.6
Cr	70.3	47.4	108	18.2
Hg	4.1	1.0	5.5	1.3
Pb	8 953	7 633	9 845	602

[a] s is one standard deviation.

REFERENCES

[1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at http://www.nist.gov/pml/pubs/sp811/index.cfm (accessed Sep 2015).

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

[3] 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 Jan 2016); 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 Jan 2016).

Certificate Revision History: 29 January 2016 (Change of expiration date; editorial changes); 07 November 2010 (Change of expiration date; editorial changes); 15 December 1999 (Original certification 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.