National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2583

Trace Elements in Indoor Dust

(Nominal Mass Fraction of 90 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 dust. SRM 2583 is composed of dust collected from vacuum cleaner bags used in the routine cleaning of interior dwelling spaces. A unit of SRM 2583 consists of 8 g of particulate material, 99+ % of which passes a 100 µm (No. 145) sieve.

The certified values for five elements in SRM 2583 are listed in Table 1. The certified values are based on measurements using 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 2. 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 100 mg.

Element	Mass Fraction (mg/kg)
Arsenic (As)	7.0 ± 1.6
Cadmium (Cd)	7.3 ± 3.7
Chromium (Cr)	80 ± 22
Lead (Pb)	85.9 ± 7.2
Mercury (Hg)	1.56 ± 0.19

Certified Mass Fraction Values: 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, chromium, and lead are based on a 95 % prediction interval for the true value. This interval includes the combined effect of uncertainty components associated with material inhomogeneity, measurement uncertainty, and an allowance for differences between the analytical methods used [3]. The uncertainty for mercury, which exhibited no evidence of inhomogeneity, is based on a 95 % confidence interval for the true value, including the combined effect 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.

Expiration of Certification: The certification of **SRM 2583** lot 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 J.R. DeVoe, P.A. Pella, and R.L. Watters, Jr. of NIST.

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

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

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

Partial financial support for the development of this SRM was provided by the U.S. Environmental Protection Agency (EPA) 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).

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Stability: This material is considered to be stable.

Use: To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample mass of 100 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. 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 (cadmium, chromium, and lead) are to be determined, samples should be oven dried for 2 h at 110 ºC. Volatile elements (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 are then made to measurement values before comparing them to the certified values. At NIST, mass loss on drying at the time of certification was found to be 3.9 % with a standard deviation (n = 6) of 0.6 %.

COLLECTION, PREPARATION, AND ANALYSIS

Collection: The bulk of the material for this SRM was obtained 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 and the U.S. Environmental Protection Agency. The collection process was coordinated by E.D. Hardison and D.A. Binstock, of the Research Triangle Institute (RTI) (Research Triangle Park, NC), under the leadership of W.F. Gutknecht.

Preparation: The bags were labeled to provide source identification, 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, P. Seo, and D. Lillian of the NIST Chemistry Sciences Division. A sample of dust from each bag was passed through a 100 µm nylon sieve and measured by laboratory X-ray fluorescence. Only bags containing dust measuring 60 µg/g to 300 µg/g of lead were retained for preparing this SRM. The selected bags were processed by passing the contents of each bag through a coarse screen (2 mm hole size) to remove cotton and debris. Using a vibrating stainless steel sieve apparatus, the resultant material was screened in two successive steps, first through a 250 µm sieve and then a 100 µm sieve. All material passing a 100 µm sieve was combined, resieved five times through a 250 µm sieve to remove hairs, blended in cone blender and then bottled.

Analysis: Certification analyses were performed in the NIST Chemical Sciences Division. Analytical methods used at NIST are given in Table 2.

Table 2. Methods used for the Analysis of SRM 2583

Element	Methods[(a)]
Arsenic	**FIA-HGAAS**, **INAA**
Cadmium	**ID-ICPMS**, ICPMS
Chromium	**INAA**, **ICPMS**
Lead	**ID-ICPMS**, ID-ICPMS (CFM), XRF
Mercury	**FIA-CVAAS**, **INAA**

[(a)] Methods used for establishment of certified values are shown in bold-face type; methods used for information only values or to corroborate certified values are not in bold.

Methods:

FIA-CVAAS	Flow injection analysis cold vapor atomic absorption spectrometry
FIA-HGAAS	Flow injection hydride generation atomic absorption spectrometry
ICP-AES	Inductively coupled plasma atomic emission spectrometry
ICPMS	Inductively coupled plasma mass spectrometry
ID-ICPMS	Isotope dilution inductively coupled plasma mass spectrometry
ID-ICPMS (CFM)	Isotope dilution inductively coupled plasma mass spectrometry with continuous flow microwave digestion [4]
INAA	Instrumental neutron activation analysis
XRF	X-ray fluorescence spectrometry (wavelength-dispersive)

NIST Analysts: E.S. Beary, P.A. Pella, M.S. Epstein, M.S. Rearick, E.A. Mackey, A.F. Marlow , G.C. Turk, J.R. Moody, L.J. Wood, K.E. Murphy, and R. Saraswati (Guest scientist from the Defense Metallurgical Research Laboratory, India).

User Experience with SRM 2583

In order to demonstrate user experience with SRM 2583, a number of laboratories analyzed this material, each using its typical method. Among the participants, the range of digestion procedures used included the EPA-SW846-3051 and 3052 microwave digestion methods and the 3050A hotplate method. In addition, EPA-SW846-7470A and 7471 were used for Hg, and ultrasonic digestion methods were used for Pb. Instrumental methods included ICPMS, Graphite Furnace AAS, Flame AAS, and Hg cold vapor AAS. The results from this study were not used in calculating the certified values of SRM 2583. The results are given in Table 3. The summary statistics are based on 6 reported results for Pb, and 5 results for the other elements.

Table 3. Results of Round Robin Exercise

Element	Mean (mg/kg)	Minimum (mg/kg)	Maximum (mg/kg)	s[(a)] (mg/kg)
As	5.2	2.5	6.3	1.6
Cd	6.3	5.8	7.0	0.5
Cr	44	33	67	13
Hg	1.5	1.3	1.7	0.2
Pb	78	57	89	13

[(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 Jan 2016).

[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).

[4] Beary, E.S.; Paulsen, P.J.; Jassie, L.B.; Fassett, J.D.; *Determination of Environmental Lead Using Continuous-Flow Microwave Digestion Isotope Dilution Inductively Coupled Plasma Mass Spectrometry*, Analytical Chemistry, Vol. 69, No. 4, pp. 758–766 (1997).

Certificate Revision History: 29 January 2016 (Change of expiration date; editorial changes); 30 November 2010 (Change of expiration date; editorial changes); 22 June 1998 (Addition of round robin data and editorial revision of Table 2); 30 December 1996 (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.