



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2583

Trace Elements in Indoor Dust Nominal 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 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.

Table 1. Certified Mass Fractions

| Element | Mass Fraction, in mg/kg | | |
|----------|-------------------------|-------|------|
| Arsenic | 7.0 | \pm | 1.6 |
| Cadmium | 7.3 | \pm | 3.7 |
| Chromium | 80 | \pm | 22 |
| Lead | 85.9 | \pm | 7.2 |
| Mercury | 1.56 | \pm | 0.19 |

Certified Values and Uncertainties: 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.

Expiration of Certification: The certification of this SRM lot is valid within the measurement uncertainties specified until December 31, 2010, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Use section). However, the certification will be nullified if the SRM is contaminated or modified.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Gaithersburg, MD 20899
Certificate Issue Date: December 30, 1996

Thomas E. Gills, Chief
Standard Reference Materials Program

The 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 the NIST Analytical Chemistry 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 (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.

NOTICE AND WARNING TO USERS

Stability: This material is considered to be stable. NIST will monitor this material and will report any substantive changes in certification to the purchaser. Return of the attached registration card will facilitate notification.

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 °C. 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, 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 Analytical Chemistry Division (ACD). 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 $\mu\text{g/g}$ to 300 $\mu\text{g/g}$ 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 about 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 Analytical Chemistry Division. Analytical methods used at NIST are given in Table 2. NIST analysts are listed after Table 2.

Table 2. Methods used for the Analysis of SRM 2583^a

| | |
|----------|-------------------------------|
| Arsenic | FIA-HGAAS, INAA |
| Cadmium | ID-ICPMS, ICPMS |
| Chromium | INAA, ICPMS |
| Lead | ID-ICPMS, ICP-AES, XRF |
| Mercury | FIA-CVAAS, INAA |

^aMethods 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 |
| 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 | R. Saraswati* |
| A.F. Marlow | G.C. Turk |
| J.R. Moody | L.J. Wood |
| K.E. Murphy | |

*R. Saraswati, a Guest Scientist from the Defense Metallurgical Research Laboratory, India.

REFERENCES

- [1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).
- [2] Schiller, S.B. and Eberhardt, K.R., Combining Data from Independent Chemical Analysis Methods, *Spectrochimical Acta*, **46B** (12), pp. 1607-1613, (1991).
- [3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993).