



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1648 Urban Particulate Matter

This Standard Reference Material (SRM) is intended for use as a control material and in the evaluation of methods used in the analysis of atmospheric particulate matter and materials with a similar matrix. It consists of 2 grams of natural atmospheric particulate matter collected in an urban location. While not represented to be typical of the area in which it was collected, its use should typify the analytical problems of atmospheric samples obtained from industrialized urban areas.

The certified values for the constituent elements are shown in Table 1. Noncertified values are given for information only in Table 2. The analytical techniques used in the characterization of this SRM are shown in Table 3. The certified values are based on measurements of 6 to 30 samples by each of the analytical techniques indicated.

Notice and Warnings to Users: This material may contain a number of chemicals of unknown toxicities. Therefore, the utmost caution and care must be exercised in its use.

Expiration of Certification: This certification is valid for 5 years from date of purchase from NIST. Should any of the certified values change before the expiration of the certification, purchasers will be notified by NIST.

Stability: This material should be kept in its original bottle and stored at temperatures between 10-30 °C. It should not be exposed to intense sources of radiation, including ultraviolet lamps or sunlight. Ideally, the bottle should be kept in a desiccator in the recommended temperature range.

Use: A minimum of 100 mg of the dried material (See Drying Instructions) should be used for any analytical determination to be related to the certified values of this certificate.

Instructions for Drying: This material should be dried at 105 °C for 3 hours before use because the certified concentrations are reported on a "dry-weight" basis. Concentrations determined on undried samples must therefore be adjusted for the moisture content of the samples.

The original direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J.K. Taylor.

The technical and support aspects involved in the preparation, current and previous certification, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by T.E. Gills and W.P. Reed.

Gaithersburg, MD 20899
August 30, 1991
(Revision of Certificate dated 5-11-82)

William P. Reed, Chief
Standard Reference Materials Program

(over)

Homogeneity Assessment: Randomly selected bottles were used for the analytical measurements. Each analyst examined at least 6 bottles. No correlation was found between measured values and the bottling sequence. Also, the results of measurements of samples from different bottles were not significantly different than the measurements of replicate samples from single bottles. Accordingly, all bottles of this SRM have been assigned the same certified values of constituent elements.

Source and Preparation of Material: This SRM was prepared from urban particulate matter collected in the St. Louis, Missouri area in a baghouse specially designed for this purpose. The material was collected over a period in excess of 12 months and, therefore, is a time-integrated sample. The material was removed from the filter bags, combined in a single lot, screened through a fine-mesh sieve to remove extraneous materials and thoroughly blended in a V-blender. The material was then packaged into bottles sequentially numbered.

Table 1. Certified Values of Constituent Elements

<u>Major Constituents</u>		<u>Minor Constituents</u>	
<u>Element</u>	<u>Content^a</u> <u>Wt. Percent</u>	<u>Element</u>	<u>Content^a</u> <u>Wt. Percent</u>
Aluminum ^b	3.42 ± 0.11	Lead	0.655 ± 0.008
Iron	3.91 ± 0.10	Sodium ^b	0.425 ± 0.002
Potassium ^b	1.05 ± 0.01	Zinc	0.476 ± 0.014

<u>Trace Constituents</u>			
<u>Element</u>	<u>Content^a</u> <u>µg/g</u>	<u>Element</u>	<u>Content^a</u> <u>µg/g</u>
Arsenic	115 ± 10	Nickel	82 ± 3
Cadmium	75 ± 7	Selenium ^b	27 ± 1
Chromium	403 ± 12	Uranium	5.5 ± 0.1
Copper	609 ± 27	Vanadium ^b	140 ± 3

^aThe uncertainties of the certified values, except those noted, include errors associated with both measurement and material variability. They represent the 95 percent tolerance limits for individual subsamples, i.e., 95 percent of the subsamples from a single unit of this SRM would be expected to have a composition within the indicated range of values 95 percent of the time.

^bThese elements were recently certified as a part of the NIST update certification program. The value for each indicated constituent is the "best value" based on all measurement methods used and the associated uncertainty is expressed as the standard error considering variability within and between analytical methods.

Table 2. Noncertified Values for Constituent Elements

Note: The following values are not certified because they are not based on the results of either a reference method or two or more independent methods. These values are included for information only.

<u>Major Constituents</u>		<u>Minor Constituents</u>	
<u>Element</u>	<u>Content</u> <u>Wt. Percent</u>	<u>Element</u>	<u>Content</u> <u>Wt. Percent</u>
Sulfur	(5.0)	Chlorine	(0.45)
Magnesium	(0.8)	Titanium	(0.40)

<u>Trace Constituents</u>			
<u>Element</u>	<u>Content</u> <u>µg/g</u>	<u>Element</u>	<u>Content</u> <u>µg/g</u>
Antimony	(45)	Lanthanum	(42)
Barium	(737)	Rubidium	(52)
Bromine	(500)	Manganese	(860)
Cerium	(55)	Samarium	(4.4)
Cesium	(3)	Scandium	(7)
Cobalt	(18)	Silver	(6)
Europium	(0.8)	Thorium	(7.4)
Hafnium	(4.4)	Tungsten	(4.8)
Indium	(1.0)		
Iodine	(20)		

Supplemental Information

The values listed below are based on measurements made in a single laboratory and are given for information only. While there is no reason to suspect systematic bias in these numbers, no attempt was made to evaluate such bias attributable to either the method or the laboratory. The method used for each set of measurements is also listed. The uncertainties indicated are two times the standard deviation of the means.

<u>Constituent</u>	<u>Content Wt. Percent</u>
Nitrogen (NO ₃)	(1.07 ± 0.06)
Nitrogen (NH ₄)	(2.01 ± 0.08)
Sulfate	(15.42 ± 0.14)
SiO ₂	(26.8 ± 0.4)
Freon Soluble	(1.19 ± 0.47)

Methods Used:

Nitrate - Extraction with water and measurement by ASTM Method D992.

Ammonia - NaOH addition followed by steam distillation and titration.

Sulfate - Extraction with water and measurement by ASTM D516.

SiO₂ - Solution and measurement by ASTM Method E350.

Freon Soluble - Extraction with Freon 113, using the Method described in "Standard Methods in Examination of Water and Waste Water," 14th Edition, P. 518, American Public Health Association, Washington, D.C.

Table 3. Methods and Analysis

Method	Elements Determined
Atomic Absorption Spectrometry	Cd, Cu, Fe, K, Mn, Na, Ni, Pb, Se, V, Zn
Isotope Dilution Thermal Ionization Mass Spectrometry	Cd, Cr, Cu, Fe, Ni, Pb, U, Zn
Neutron Activation Analysis	Ag, Al, As, Ba, Br, Cd, Ce, Co, Cr, Cs, Eu, Fe, Hf, I, In, K, La, Mg, Mn, Na, Rb, Sb, Sc, Se, Sm, Th, Ti, V, W, Zn
Polarography	Cd, Ni, Pb, Zn
Spectrophotometry	As, Cu, Fe
Photon Activation Analysis	I
Ion Chromatography	Cl, S
DC Plasma Atomic Emission Spectrometry	Al
Flame Emission Spectrometry	Mn, Na, Se

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