National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1648

Urban Particulate Matter

This Standard Reference Material (SRM) is intended primarily 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 g 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 expressed in mass fraction, for the constituent elements are shown in Table 1. Noncertified values expressed in mass fraction, are given for information only in Table 2. The analytical methods 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 methods indicated.

NOTICE AND WARNING TO USERS

Expiration of Certification: The certification of SRM 1648 is valid, within the measurement uncertainty (ies) specified, until **31 December 2008**, provided the SRM is handled in accordance with instructions given in this certificate (see Instructions for Use). This certification is nullified if the SRM is damaged, contaminated, or 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. Return of the attached registration card will facilitate notification.

Instructions for Use: This material may contain a number of chemicals of unknown toxicities. Therefore, the utmost caution and care must be exercised in its use. A minimum of 100 mg of the dried material (See Instructions for Drying) should be used for any analytical determination to be related to the certified values of this certificate. When not in use, this material should be kept in its original bottle and stored at temperatures between 10 °C to 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.

Instructions for Drying: The certified concentrations are reported on a "dry-weight" basis. This material should be dried at 105 °C for 8 h before use because concentrations determined on undried samples must therefore be adjusted for the moisture content.

The technical and support aspects involved in the original preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills. Measurement activities for revision of this certificate were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Gaithersburg, MD 20899 — Thomas E. Gills, Chief

Certificate Issue Date: 28 April 1998 — Standard Reference Materials Program

11/16/78 (original certificate date); 5/11/82 (additional certification update); 8/30/91 (editorial)

*This revision reports a change in the certified value of vanadium, the addition of the manganese certified value, the subsequent removal of the manganese information value, and change in expiration date.

Statistical analysis of the revised certification data for manganese and vanadium was performed by K.R. Eberhardt of the NIST Statistical Engineering Division.

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 from 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, MO 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 sequentially numbered bottles.

Table 1. Certified Values of Constituent Elements

Major Constituents

Element	Content Mass Fraction, in %
Aluminum	3.42 ± 0.11
Iron	3.91 ± 0.10
Potassium	1.05 ± 0.01

Minor Constituents

Element	Content Mass Fraction, in %
Lead	0.655 ± 0.008
Sodium	0.425 ± 0.002
Zinc	0.476 ± 0.014

Trace Constituents

Element	Content mg/kg
Arsenic	115 ± 10
Cadmium	75 ± 7
Chromium	403 ± 12
Copper	609 ± 27
Manganese	786 ± 17

Element	Content mg/kg
Nickel	82 ± 3
Selenium	27 ± 1
Uranium	5.5 ± 0.1
Vanadium	127 ± 7

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

[b] The indicated constituent was certified as a part of the NIST update certification program, in August 1991. 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.

[c] The uncertainty in the certified value is calculated as $U = ku + B$ where u is the combined standard uncertainty calculated according to the ISO Guide [1] and k is a coverage factor. The additional quantity, B, is an allowance for the differences between methods of analysis and is taken to be equal to the difference between the most discrepant method value and the certified value. The expanded uncertainty (U) given is intended to approximate the 95 % level of confidence.

Table 2. Noncertified Values for Constituent Elements

Major Constituents		Minor Constituents	
Element	Content Mass Fraction, in %	Element	Content Mass Fraction, in %
Sulfur	5.0	Chlorine	0.45
Magnesium	0.8	Titanium	0.40

Trace Constituents

Element	Content mg/kg	Element	Content mg/kg
Antimony	45	Iodine	20
Barium	737	Lanthanum	42
Bromine	500	Rubidium	52
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		

Table 3. Methods of Analysis

Element	Methods	Element	Methods
Aluminum	DCPAES, NAA	Lead	AAS, IDMS, POL
Antimony	NAA	Magnesium	NAA
Arsenic	NAA, SPECTR	Manganese	AAS, NAA, DCPAES
Barium	NAA	Nickel	AAS, IDMS, POL
Bromine	NAA	Potassium	AAS
Cadmium	AAS, IDMS, NAA, POL	Rubidium	NAA
Cerium	NAA	Samarium	NAA
Cesium	NAA	Scandium	NAA
Chlorine	NAA	Selenium	AAS, NAA, FES
Chromium	IDMS, NAA	Sodium	AAS, NAA, FES
Cobalt	NAA	Silver	NAA
Copper	AAS, IDMS, SPECTR	Sulfur	IC
Europium	NAA	Thorium	NAA
Hanfium	NAA	Titanium	NAA
Indium	NAA	Tungsten	NAA
Iodine	NAA, PAA	Uranium	IDMS
Iron	AAS, IDMS, NAA, SPECTR	Vanadium	NAA
Lanthanum	NAA	Zinc	AAS, IDMS, NAA, POL

Methods

AAS	Atomic Absorption Spectrometry	FES	Flame Emission Spectrometry
DCPAES	DC Plasma Atomic Emission Spectrometry	NAA	Neutron Activation Analysis
IC	Ion Chromatography	PAA	Photon Activation Analysis
IDMS	Isotope Dilution Thermal Ionization Spectrometry	POL	Polarography
		SPECTR	Spectrophotometry

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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 values, no attempt was made to evaluate the bias attributable to either the method or the laboratory. The method used for each set of measurements and the standard deviation of the means are also listed.

Constituent	Mass Fraction, in %	One Standard Deviation
Nitrogen (NO_3)	1.07	0.03
Nitrogen (NH_3)	2.01	0.04
Sulfate	15.42	0.07
Silicon Dioxide (SiO_2)	26.8	0.2
Freon Soluble	1.19	0.24

Methods Used

Nitrate (NO_3) - Extraction with water and measurement by ASTM Method D992.
Ammonia (NH_3)- NaOH addition followed by steam distillation and titration.
Sulfate - Extraction with water and measurement by ASTM D516.
Silicon Dioxide (SiO_2)- 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 Ed., p. 518, American Public Health Association, Washington DC.

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and 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).

[2] Kucera, J. and Soukal, L., Low Uncertainty Determination of Manganese and Vanadium in Environmental and Biological Reference Materials by Instrumental Neutron Activation Analysis, Berm-7, (Apr. 97).