



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 2783

#### Air Particulate on Filter Media

This Standard Reference Material (SRM) is an air particulate sample reduced in particle size to simulate PM<sub>2.5</sub> air particulate matter (particles with an aerodynamic equivalent diameter of 2.5  $\mu\text{m}$ ) and deposited on a polycarbonate filter membrane. It is primarily intended for use in the evaluation and calibration of methods of analysis for common and toxic elements contained in various fractions of airborne particulate matter collected on filter media. A unit of SRM 2783 includes two loaded filters and two blank filters.

**Certified Values:** The certified values for elemental content of the SRM, expressed as mass of element deposited on the filter membrane, are provided 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 into account [1]. The certified values for all elements are based on results of NIST methods and results from collaborating laboratories using independent and complementary analytical methods.

**Reference Values:** The reference values for elemental content, expressed as mass of element deposited on the filter membrane, are provided in Table 2. Reference values for selected elements in blank filters are provided in Table 4. 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 reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1].

**Information Values:** Supplementary information values for average deposit area and mass loading per loaded filter are provided in Table 3. Information values for blank filters are provided in Table 5. An information value is considered to be a value that will be of use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value.

**Expiration of Certification:** The certification of **SRM 2783** is valid, within the measurement uncertainty specified, until **01 September 2021**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). This 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) will facilitate notification.

This SRM has been developed in cooperation with the International Atomic Energy Agency (IAEA) Laboratories, Seibersdorf, Austria and collaborating laboratories in several countries.

The material was prepared by R. Oflaz and R.L. Zeisler of the NIST Analytical Chemistry Division. The coordination of the technical measurements leading to certification was performed by R.L. Zeisler.

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Certificate Issue Date: 13 December 2011  
*Certificate Revision History on Last Page*

Analytical measurements at NIST were performed by R. Oflaz, M.E. Howard, A.P. Lindstrom, K.E. Murphy, J.R. Sieber, L.J. Wood, L.L. Yu, R.L. Zeisler of the NIST Analytical Chemistry Division.

Results were also provided by analysts at other collaborating laboratories: M. Bogovac, A. Mendoza, A. Markowicz at IAEA Laboratories Seibersdorf, Austria; I. Orlic at ANSTO, Australia; W. Maenhaut at Ghent University, Belgium; P. Artaxo at University of São Paulo, Brazil; M. Jakšić at Rudjer Bošković Institute; Croatia; J. Kučera at Nuclear Physics Institute, Czech Republic; M. Gallorini; E. Rizzio at University of Pavia; Italy; J. Weinstein at U.S. EPA, Research Triangle Park, NC, USA.

Statistical analyses leading to certified and reference values were performed by S.D. Leigh of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

## INSTRUCTIONS FOR HANDLING, STORAGE, AND USE<sup>(1)</sup>

**Handling:** Please note that no fixation or surface coating was used on the filters, therefore they should be handled with care to avoid material loss or contamination. The filter membranes are mounted in Millipore PetriSlide containers (Figure 1). These containers are holding the filter membranes in place by means of a plastic ring inside the lid. To remove the filter from the container, grasp the lid with the thumb and finger of one hand at the grip, while holding the slide, i.e., bottom portion of the casing, with the other hand. The lid is then opened with a slight twisting upward motion. **Users are cautioned to use care when opening the container because electrostatic charges may cause the filter membrane to move.** The filter membrane can be picked up at the notched side of the casing with round-tipped (blunt) forceps.

**Storage:** The material should be kept in its original container and stored at temperatures less than 30 °C and away from sources of light.

**Use:** The filters should be used as received; the surface with the deposit shall **NOT** be touched or brought into contact with implements. Filters can be used for multiple non-destructive assays; the filter membranes should be stored in the Millipore PetriSlide containers after use; the deposit (shiny) side of the filter must face upward. If a filter is partitioned for analysis or not analyzed in whole due to the analytical technique used, a minimum sample area of 1 cm<sup>2</sup> should be used for analytical determinations to be related to elemental mass loading values provided. The value of the total deposit area should be determined for the filter being measured because the deposited area is smaller than the filter area. The deposited area can be determined directly, or the information value for average deposited area in Table 3 may be used.

**Blanks:** Blank filters are supplied to augment the assays of the aerosol loaded filters with the determination of analytical blanks associated with the filter matrix. A heterogeneous distribution of bromine in the filter lots prevented a value assignment for this element. A significant amount of chromium and nickel is found by techniques that assay the whole filter, i.e., matrix and aerosol loading together, such as instrumental neutron activation analysis (INAA). Reference and information values established for several elements are listed in Table 4 and Table 5.

## PREPARATION, HOMOGENEITY, AND ANALYSIS

**Source and Preparation of Material:** The aerosol material deposited on the filters is intended to represent a material of PM<sub>2.5</sub> particle size distribution with elemental concentrations typical of those of an urban industrial area. A special collection of PM<sub>2.5</sub> material for this project from an EPA national monitoring site in Baltimore, MD, had yielded sufficient contemporary material [2], however controlled deposit on filter membranes was not achievable because of variable retention of ultra-fine particles. Therefore an earlier prepared and successfully tested batch of urban particulate matter [3] was used. This urban dust was collected by the IAEA Laboratories from the air intake system of the Vienna General Hospital (AKH), in Vienna, Austria. The hospital is located in a mixed industrial urban area, with several nearby major highways. The dust was collected with vacuum cleaners from the electrostatic filters of the hospital air intake ducts. Approximately 9 kg of material was collected in three batches over the period September 1994 to December 1995. The batches of material were combined and sieved to particle sizes below 70 µm to eliminate coarse materials. The sieved sample was homogenized with a V-shaped blender for

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<sup>(1)</sup> Certain commercial equipment, instruments or materials are identified in this certificate 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.

4 h. A portion of this material was then successively air-jet milled three times to obtain a more homogeneous distribution of smaller particles that simulates the respirable fractions of an urban aerosol. The material has a median particle size of 3.2  $\mu\text{m}$  and a size range (full width at half maximum) of 2.5  $\mu\text{m}$ . The air-jet milled material was designated IAEA-396a/m. Small sample analysis studies have demonstrated a high degree of homogeneity for this material [4].

All preparation for the deposit on the filter membranes and handling of the filters was carried out in the NIST Specimen Bank clean room. Batches of 700 mL to 900 mL each of air particulate matter suspensions were prepared by weighing appropriate portions of bulk air particulate matter into 1 L Erlenmeyer flasks, adding the appropriate mass of pure water containing a mass fraction of 0.1 % Triton X-100, and placing the flask in an ultra-sonicating bath. A 140  $\mu\text{g/mL}$  starting ratio yielded a 100  $\mu\text{g/mL}$  particulate suspension for deposit after 1 h of ultra-sonication, followed by 15 h equilibration and an additional 1 h of ultra-sonication. During the subsequent dispensation, the particulate was kept in suspension by vigorous stirring. Each of these batches was prepared for one day of filter preparation (about 150 filters each); a total of 15 batches were prepared yielding 2200 individual filters.

Two Millipore filtration units were used for the preparation. The filters were Nuclepore polycarbonate membrane filters, 0.4  $\mu\text{m}$  pore size (Corning, Lots 8265024 and 8265025). After mounting the filters into the filtration assembly, the filters were covered with about 40 mL of pure water, then 5 mL aliquots of the suspension were pipetted on top with a 5 mL adjustable pipette. Because two staff members were preparing the SRM filters, the pipettes were calibrated to deliver the same volume by mass. This calibration was controlled before and during the preparation of each SRM batch. After dispensation into the filtration apparatus, vacuum was applied and the aerosol matter was deposited on the filters. After filtering to dryness, the filters were removed from the filtration apparatus and allowed to air dry under Class 10 clean air flow.

**Homogeneity:** The average mass loadings and homogeneity of the deposition procedure for the lot was determined by gravimetry using ten preweighed filters per batch and weighing the prepared filters after drying and conditioning. The pooled relative standard deviation of this procedure was 5.5 %. Considering the difficulties of these mass determinations, the loaded filters were accepted as a homogeneous lot. The homogeneity of elemental mass loadings was determined by INAA on 30 filters, 2 from each batch selected at random. Twenty elements ranging from several nanograms to tens of microgram amounts exhibited a relative standard deviation attributable to heterogeneity of less than 2.5 %. The within-filter homogeneity was determined from micro-beam energy dispersive x-ray fluorescence (EDXRF) and micro-beam proton-induced x-ray emission (PIXE) data. Variances in elemental mass loading not exceeding 4 % relative standard deviation were observed for sampling areas ranging from 1  $\text{mm}^2$  to 30  $\text{mm}^2$ . A sampling area of 1  $\text{cm}^2$  is deemed necessary for any certified value in Table 1 to be valid within the stated uncertainty.

## VALUE ASSIGNMENT

The analytical techniques used for measurement of each element are listed in Table 6.

**Certified Values:** Certified values are weighted means of results from two or more analytical methods [5]. The uncertainty listed with each value is an expanded uncertainty, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance, and a component of variance for material heterogeneity following the ISO Guide [6].

Table 1. Certified Mass Loading Values for Selected Elements

Elements	$\text{ng}^{(a)}$			Elements	$\text{ng}^{(a)}$		
Al	23210	$\pm$	530	Mg	8620	$\pm$	520
As	11.8	$\pm$	1.2	Mn	320	$\pm$	12
Ba	335	$\pm$	50	Na	1860	$\pm$	100
Ca	13200	$\pm$	1700	Ni	68	$\pm$	12
Co	7.7	$\pm$	1.2	Pb	317	$\pm$	54
Cr	135	$\pm$	25	Sb <sup>(b)</sup>	71.8	$\pm$	2.6
Cu	404	$\pm$	42	Ti	1490	$\pm$	240
Fe	26500	$\pm$	1600	V	48.5	$\pm$	6.0
K	5280	$\pm$	520	Zn	1790	$\pm$	130

<sup>(a)</sup> Conversion to  $\text{ng/cm}^2$  must use area of deposit (not total filter membrane).

<sup>(b)</sup> Expanded uncertainty calculated according to BOB, “Type B On Bias” [7]. Although this method has been discontinued since the original certification, the numerical value of the uncertainty assessment is valid.

**Reference Values:** Reference values are based on weighted means of results from one method determined in several collaborating laboratories or from two or more analytical methods performed by selected collaborating laboratories. The uncertainty listed with each value is an expanded uncertainty, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method or inter-procedure bias with a pooled within-source variance, and a component of variance for material heterogeneity following the ISO Guide [6]. These results do not fulfill the criteria for certification since a full estimate of method bias or results from NIST methods are not available.

Table 2. Reference Mass Loading Values for Selected Elements

Elements	ng <sup>(a)</sup>			Elements	ng <sup>(a)</sup>		
Ce	23.4	±	3.5	Si	58600	±	1600
Rb	24.0	±	5.5	Sm	2.04	±	0.15
S	1050	±	260	Th	3.23	±	0.59
Sc	3.54	±	0.28	U <sup>(b)</sup>	1.234	±	0.024
				W	5.0	±	1.8

<sup>(a)</sup> Conversion to ng/cm<sup>2</sup> must use area of deposit (not total filter membrane).

<sup>(b)</sup> Data from a single source only; uncertainty does not include an estimate for between-source variance.

Table 3. Supplementary Information Values

Average deposit area per filter: 9.96 cm<sup>2</sup> (1 s = 0.04 cm<sup>2</sup>)  
Average mass loading per filter: 485 µg (1 s = 10 µg)

**Reference Values for Blank Filters:** Reference values are based on weighted means of results from one method determined in several collaborating laboratories or from two or more analytical methods performed by selected collaborating laboratories. The uncertainty listed with each value is an expanded uncertainty, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method or inter-procedure bias with a pooled within-source variance, and a component of variance for material heterogeneity following the ISO Guide [6]. These results do not fulfill the criteria for certification since a full estimate of method bias or results from NIST methods are not available. The uncertainty for the nickel reference value does not include an estimate for between-source variance because nickel data are from a single source only.

Table 4. Reference Values for Selected Elements in Blank Filters<sup>(b)</sup>

Elements	ng <sup>(a)</sup>		
Ni	8	±	2
Cr	70	±	4

<sup>(a)</sup> Conversion to ng/cm<sup>2</sup> must use area of deposit (not total filter membrane).

<sup>(b)</sup> Only found by techniques that include complete filter matrix in analysis.

**Information Values for Blank Filters:** Information values are given for SRM 2783 blank filters to assist users in the determination of the components of their procedural blank that are from the blank filter alone. Information values are based on results that did not allow complete assessment of Type A and Type B components of uncertainty, hence, only an estimated mean without an uncertainty is given for each element. For all blank filters analyzed, only elements for which blank values were observed above the detection limit of the method used are reported. These limits of detection are generally lower than 1 % relative of the certified or reference value in the loaded filter.

Table 5. Information Values for Selected Elements in Blank Filters

Elements	ng <sup>(a)</sup>
Al	30
Na	15
Ba	0.4
Co	0.04
Pb	0.4
S	100
Zn	50

<sup>(a)</sup> Conversion to ng/cm<sup>2</sup> must use area of deposit (not total filter membrane).

Table 6. Analytical Techniques Used for Value Assignment

Element	Methods	Element	Methods
Al	INAA, EDXRF, ICP-MS, PIXE	Pb	EDXRF, ID-ICP-MS, PIXE
As	INAA, PIXE	Rb	INAA, EDXRF
Ba	INAA, EDXRF, ID-ICP-MS, PIXE	S	EDXRF, PIXE
Ca	INAA, EDXRF, PIXE	Sb	INAA, EDXRF, ID-ICP-MS
Ce	INAA	Sc	INAA
Co	INAA, ICP-MS	Si	INAA, EDXRF, PIXE
Cr	INAA, EDXRF, PIXE	Sm	INAA
Cu	INAA, EDXRF, ID-ICP-MS, PIXE	Th	INAA
Fe	INAA, EDXRF, PIXE	Ti	INAA, EDXRF, PIXE
K	INAA, EDXRF, PIXE	U	ID-ICP-MS
Mg	INAA, PIXE	V	INAA, ICP-MS, PIXE
MN	INAA, EDXR, PIXE	W	INAA
Na	INAA, ICP-MS, PIXE	Zn	INAA, EDXRF, PIXE
Ni	EDXRF, ID-ICP-MS, PIXE		

## Key to Methods:

INAA	Instrumental neutron activation analysis
EDXRF	Energy-dispersive X-ray fluorescence, including micro-EDXRF
ID-ICP-MS	Isotope dilution inductively coupled plasma mass spectrometry
ICP-MS	Inductively coupled plasma mass spectrometry
PIXE	Proton-induced X-ray emission analysis, including micro-PIXE

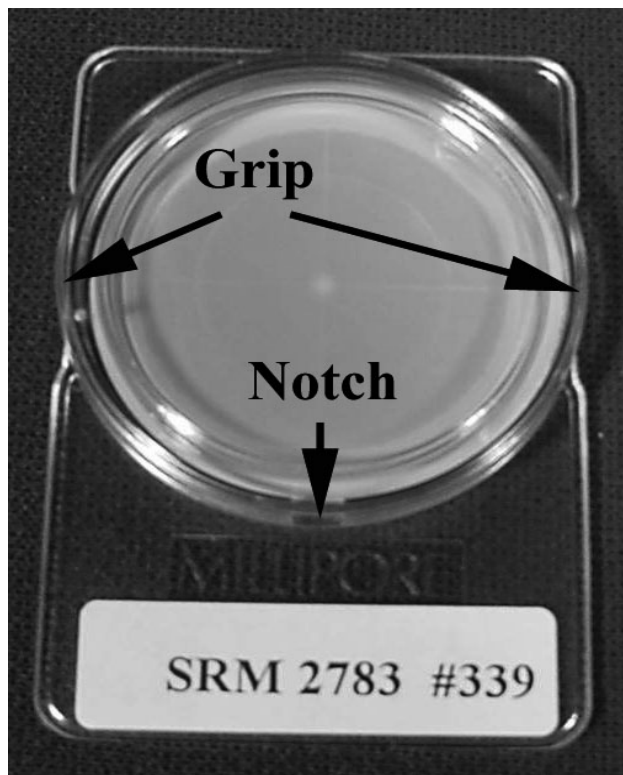


Figure 1. Millipore PetriSlide container

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**Certificate Revision History:** 13 December 2011 (Editorial changes); 19 July 2011 (Extension of the certification period; editorial changes); 02 January 2002 (This revision reflects an editorial correction to Table 1); 19 November 2001 (Original certificate 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) 926-4751; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*