National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1832

Thin Glass Film on Polycarbonate for X-ray Fluorescence Spectrometry

Serial No.

This Standard Reference Material (SRM) is intended primarily for use in the standardization of X-ray fluorescence spectrometers, such as in the elemental analysis of particulate matter collected on filter media and in applications where X-ray spectrometer calibration functions are determined using thin film standards.

SRM 1832 consists of a silica-based glass film that has been deposited onto a polycarbonate filter. The glass film is a continuous layer approximately 0.55 μm thick that contains known mass fractions of the oxides of selected elements. The film-covered filter is mounted on an aluminum ring to maintain a uniform and reproducible geometry.

The certified values given in Table 1 are based on measurements made using various analytical techniques (see NIST Methods). Specimens of SRM 1832 are individually certified. This particular specimen was taken from retained stock that was set aside from the original production run prior to the original issuance of SRM 1832. The certified values for this specimen were assigned in the same manner using the same standard specimens as for the specimens of the original issuance, except that new measurements were made using currently available equipment.

Notice and Caution: Exposure of the films to X-radiation from high-powered X-ray sources causes severe film embrittlement and eventual destruction, even after exposures as short as one-half hour. To increase film lifetime, use should be limited to calibration of secondary thin-film standard samples for routine use. Measurements should be performed using the lowest practicable X-ray tube power for excitation.

Because the epoxy material used in mounting the filter to the retaining ring is also susceptible to radiation damage, the X-ray source radiation should be collimated or the films should be masked to shield the epoxy from exposure. Radiation damage to the epoxy may cause the filter to separate from the retaining ring.

Expiration of Certification: The certification of this SRM is valid within the specified measurement uncertainties until **01 May 2008**, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification will be nullified if this SRM is damaged or modified (see Notice and Caution).

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.

The overall direction and coordination of the preparation of this SRM and of the technical measurements leading to certification of the original issue of SRM 1832 were performed by P.A. Pella formerly of the NIST Analytical Chemistry Division. Additional measurements were performed under the direction of J.R. Sieber of the NIST Analytical Chemistry Division.

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

Willie E. May, Chief
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Certificate Issue Date: 01 August 2000
See Certificate Revision History on Last Page

Nancy M. Trahey, Chief
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NIST analytical measurements were performed by M.S. Epstein, J.R. Moody, D.E. Newbury, P.J. Paulsen, T.C. Rains, T.A. Rush, J.R. Sieber (analysis for re-issue), S.F. Stone, R.L. Watters, Jr., R.L. Zeisler, and Y.K. Zhang of the NIST Analytical Chemistry Division.

Collaborating Laboratories

The following laboratories contributed to the certification of this SRM:

Columbia Scientific Industries Corporation, Austin, TX; J. Rhodes
Lawrence Berkeley Laboratory, University of California, Berkeley, CA; R.D. Giauque
NEA Inc., Beaverton, OR; J. Cooper and C.A. Frazier
Northrup Services, Inc., Research Triangle Park, NC; R.B. Kellogg
Physics-Air Quality Group, University of California, Davis, CA; T. Cahill and R. Elrod

Use: The glass film is deposited on the non-shiny, recessed side of the filter mounted in the aluminum retaining ring. Proper use of this SRM requires that the recessed, non-shiny side face the X-ray excitation source. The polycarbonate filter material is subject to stress due to embrittlement, which may result in small cracks. As long as the filter remains relatively flat and no hole is within the area viewed by the X-ray spectrometer, the specimen remains useable.

Storage: This SRM should be stored in the container provided at a temperature of 20 °C to 25 °C.

Preparation: The films were prepared using focussed ion beam sputtering from a glass target onto polycarbonate filters (47 mm diameter, 0.1 μm pore size).

The glass targets for producing the thin films were fabricated by D. Blackburn and D. Kauffman of the National Bureau of Standards Glass and Optical Materials Group. The films were fabricated at Commonwealth Scientific Corporation, Alexandria, VA, under the supervision of P.A. Pella.

Gravimetric measurements of film weights and the mounting of films on aluminum rings were performed by A.F. Marlow and K. Garlow of the NIST Analytical Chemistry Division. Microhomogeneity determinations of the elemental composition of the films were made at NIST using electron probe microanalysis and at the University of California at Davis using proton-induced X-ray fluorescence spectrometry.

X-ray Line Interference: Due to the nature of the sputter deposition process, some argon from the ion beam and some iron from the sputtering chamber are entrapped in these films. The cobalt K-$L_{2,3}$ X-ray line should be corrected for interference of the iron K-$M_{2,3}$ line in SRM 1832.

NIST Methods

AAS – Atomic absorption spectrometry
ICP – Inductively coupled plasma emission spectrometry
NAA – Neutron activation analysis
DCP – Direct current plasma emission spectrometry
IDTIMS – Isotope dilution thermal ionization mass spectrometry

Table 1. SRM 1832

Serial No. _____ Film Mass: _____ mg

Element	Certified Value (in % m/m)	Estimated Uncertainty (in % m/m)
Sodium	( )	---
Aluminum		
Silicon		
Argon	( )	---
Calcium		
Vanadium		
Manganese		
Cobalt		
Copper		

Certified Values and Uncertainties: The certified value listed for an element is based on the results of NIST and cooperative laboratory analyses. The values from cooperating laboratories were averaged for each element. The results were then averaged with the respective NIST mean values to obtain the certified values. The estimated uncertainty listed for an element is expressed at the 99 % confidence level and based on an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability. Values in parentheses are not certified and are given for information purposes only.

NOTE: To convert the certified values from mass fraction (in %) to micrograms per square centimeter ($\mu g/cm^2$), use the following expression: (% m/m) x 10 x Film Mass (mg) ÷ 10.06 cm^2. The uncertainties in the film mass and area values are small compared to the estimated uncertainty in the certified values. Therefore, they are not included in this expression.

X-ray Absorption Corrections: Due to the finite thickness of the glass films, X-ray absorption corrections should be made, especially for the low atomic number elements aluminum, silicon, potassium, calcium, and titanium. Tabulated approximate absorption correction factors are given in Table 2 to serve as an example of their magnitudes. The user should be aware, however, that various source-sample-detector geometrical arrangements may require different absorption correction factors. For the best results, the correction factors should be determined on the particular instrument to be used.

Table 2. X-ray Absorption Correction Factors (approximate)

Element	X-ray Line	Correction Factor
Aluminum	$K\text{-}L_{2,3}$	1.17
Silicon	$K\text{-}L_{2,3}$	1.14
Potassium	$K\text{-}L_{2,3}$	1.07
Calcium	$K\text{-}L_{2,3}$	1.04
Titanium	$K\text{-}L_{2,3}$	1.04
Vanadium	$K\text{-}L_{2,3}$	1.02
Manganese	$K\text{-}L_{2,3}$	1.02
Iron	$K\text{-}L_{2,3}$	1.02
Cobalt	$K\text{-}L_{2,3}$	1.01
Copper	$K\text{-}L_{2,3}$	1.01
Zinc	$K\text{-}L_{2,3}$	1.01
Lead	$L_3\text{-}M_{4,5}$	1.00

Certificate Revision History: 01 August 2000 (New certification measurements performed, expiration date added, and information value for iron removed); 14 May 1984 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.