U.S. Department of Commerce
Malcolm Baldrige
Secretary
National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1832

Thin Glass Film on Polycarbonate for

X-Ray Fluorescence Spectrometry

This Standard Reference Material (SRM) is intended for use in the standardization of x-ray fluorescence spectrometers. It may be useful in particular applications such as 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 which contains known concentrations 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 are given in Table 1 and are based on measurements made using various analytical techniques (see section under analysts and analytical techniques).

Use: The glass film is deposited on the nonshiny or recessed side of the filter mounted in an aluminum retaining ring. Proper use of this SRM requires that the recessed or nonshiny side face the x-ray excitation source.

The certification of this SRM is valid two years from date of purchase.

Storage

This SRM should be stored in the container provided at a temperature of 20-25 °C. NBS will continue to monitor this SRM and if storage requirements change and/or certification becomes invalid the purchasers will be promptly notified.

Notice and Caution to User

Exposure of these films to x-radiation from high-powered x-ray tubes (e.g., 2000-3000 watts) 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 employing 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 film should be masked to shield the epoxy from radiation. Radiation damage to the epoxy may allow the filter to separate from the retaining ring.

The overall direction and coordination of the technical measurements leading to certification were under the direction of P.A. Pella of the NBS Gas and Particulate Science Division.

The technical and support aspects involved in the certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W.P. Reed.

Gaithersburg, MD 20899
May 14, 1984

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(over)

Supplemental Information

Preparation

The films were prepared by focused 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 NBS Glass and Optical Materials Group. The films were fabricated at Commonwealth Scientific Corporation, Alexandria, Va., under the supervision of P.A. Pella of the NBS Gas & Particulate Science Division.

The gravimetric measurements of the film weights and the mounting of the films on aluminum rings were performed by A. Marlow and K. Garlow; the microhomogeneity determinations of the elemental composition of these films were made by D. Newbury (using electron probe microanalysis (EPMA)), of the Gas and Particulate Science Division, and T. Cahill and coworkers (using proton-induced x-ray fluorescence spectrometry) at the University of California at Davis.

X-Ray Line Interferences

Because of the nature of the sputter deposition process, some argon, as well as some iron from the sputtering chamber, are entrapped in these films. The cobalt K_α x-ray line should be corrected for interference of the iron K_β line in SRM 1832.

X-Ray Absorption Corrections

Because of the finite thickness of the glass films, x-ray absorption corrections should be made, especially for the low atomic number elements Al, Si, K, Ca, and Ti. Tabulated approximate absorption corrections are given in Table 2 to serve as an example of their magnitude. The user should be aware, however, that various source-sample-detector geometries may require different absorption corrections, and for best results should be determined on the particular instrument to be used.

Analytical Methods Used and Analysts

Analytical Methods:

A. Atomic absorption spectrometry
B. Direct current plasma emission spectrometry
C. Inductively coupled plasma emission spectrometry
D. Isotope dilution thermal ionization mass spectrometry
E. Neutron activation analysis

Analysts

Center for Analytical Chemistry

1. D.E. Newbury
2. M.S. Epstein
3. J.R. Moody
4. P.J. Paulsen
5. T.C. Rains
6. T.A. Rush
7. S.F. Stone
8. R.L. Watters, Jr.
9. R.L. Zeisler
10. Y.K. Zhang

Cooperating Analysts

11. J. Rhodes, Columbia Scientific Industries Corporation, Austin, Texas.
12. R.D. Giauque, Lawrence Berkeley Laboratory, University of California, Berkeley, California.
13. J. Cooper, C.A. Frazier, NEA Inc., Beaverton, Oregon.
14. R.B. Kellogg, Northrop Services, Inc., Research Triangle Park, North Carolina.
15. T. Cahill, R. Eldred, Physics-Air Quality Group, University of California, Davis, California.

Table 1

SRM No.

1832

Serial No: 86 Film Weight: 1.517 mg

Element	Certified[1] Value, (% by wt.)	Estimated Uncertainty,[2] (% by wt.)
Sodium	(6.8 %)[3]	
13,73 Aluminum	9.11	± 0.6
33,01 Silicon	21.89	± 0.7
18,5 Calcium	12.28	± 0.8
Vanadium 4,19	2.78	± 0.3
4,22 Manganese	2.80	± 0.3
0,93 Cobalt	0.62	± 0.04
2,29 Copper	1.49	± 0.1
Argon	(1.0 %)[3]	
Iron	(0.4 %)[3]	

[1]The certified value listed for an element is based on the results of NBS-CAC and cooperative laboratory analyses. The values from cooperating laboratories were averaged for each element. The results were then averaged with the respective mean values from the NBS-CAC laboratories to obtain the certified values.

[2]The estimated uncertainty listed for an element is based on an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability.

[3]Values in parentheses are not certified, but are given for information purposes only.

NOTE: To convert the certified values from % by weight to micrograms per square centimeter, use the following expression: (% by wt) x 10^{-2} x film wt. (μg)/10.06 cm^2. The uncertainties in the film weight and area are small compared to the estimated uncertainty in the certified values and are, therefore, not included in this expression.

Table 2

Approximate X-Ray Absorption Corrections

Element and X-Ray Line	Correction Factor
Al K_α	1.17
Si K_α	1.14
K K_α	1.07
Ca K_α	1.04
Ti K_α	1.04
V K_α	1.02
Mn K_α	1.02
Fe K_α	1.02
Co K_α	1.01
Cu K_α	1.01
Zn K_α	1.01
Pb L_α	1.0

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SRM 1833 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 which contains known concentrations 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 are given in Table 1 and are based on measurements made using various analytical techniques (see section under analysts and analytical techniques).

Use: The glass film is deposited on the nonshiny or recessed side of the filter mounted in an aluminum retaining ring. Proper use of this SRM requires that the recessed or nonshiny side face the x-ray excitation source.

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X-Ray Line Interferences

Because of the nature of the sputter deposition process, some argon, as well as some iron from the sputtering chamber, are entrapped in these films. The potassium K_α x-ray line should be corrected for interference of the argon K_β line in SRM 1833.

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Table 1

SRM No.

1833

Serial No: 1113 Film Weight: 1.552 mg.

Element	Certified[1] Value, (% by wt.)		Estimated Uncertainty[2] (% by wt.)
Silicon	21.55	±	1.4
Potassium	11.08	±	1.1
Titanium	8.25	±	1.2
Iron	9.17	±	0.3
Zinc	2.52	±	0.2
Lead	10.64	±	0.5
Argon	(1.0 %)[3]		

[1]The certified value listed for an element is based on the results of NBS-CAC and cooperative laboratory analyses. The values from cooperating laboratories were averaged for each element. The results were then averaged with the respective mean values from the NBS-CAC laboratories to obtain the certified values.

[2]The estimated uncertainty listed for an element is based on an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability.

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Mn K_α	1.02
Fe K_α	1.02
Co K_α	1.01
Cu K_α	1.01
Zn K_α	1.01
Pb L_α	1.0