National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 2708

Zinc Sulfide Thin Film on Polycarbonate for X-ray Fluorescence Spectrometry

This Standard Reference Material (SRM) is intended primarily for use in the standardization of x-ray fluorescence spectrometers for sulfur. 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 2708 consists of a single layer about 0.02 µm thick of zinc sulfide deposited onto a polycarbonate filter. The film-covered filter is mounted on an aluminum ring to maintain a uniform and reproducible geometry.

The certified value for sulfur is given in Table 1 and is based on measurements by isotope-dilution thermal ionization mass spectrometry (ID-TIMS). A noncertified value, for information only, is provided for zinc.

Notice and Warnings to Users: Zinc sulfide is deposited on the nonshiny or recessed side of the filter mounted on an aluminum retaining ring. Proper use of this SRM requires that the recessed side face the excitation source. The certification of this SRM is valid two years from the date of purchase.

Storage: This SRM should be stored in the container provided at a temperature of 20-25 °C. NIST will continue to monitor this SRM and if the storage requirements change and/or certification becomes invalid, the purchasers who return the enclosed registration card will be promptly notified.

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. To increase the SRM film lifetime, its use should be limited to the calibration of secondary thin-film standard samples. These secondary standards should then be used for routine calibrations. 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 incident x-ray beam should be suitably collimated to prevent irradiation of the epoxy. Assuming an elliptical beam, a beam size of 20 mm (long axis) by 15 mm (short axis) incident on the SRM surface is recommended.

The overall direction and coordination of the technical measurements leading to the certification were under the direction of P.A. Pella of the NIST Inorganic Analytical Research Division.

Statistical analysis of the experimental data was performed by S.B. Schiller of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills.

Gaithersburg, MD 20899
December 30, 1991

William P. Reed, Chief
Standard Reference Materials Program

(over)

Table 1

SRM No. 2708

Serial No. _______

Element	Certified Value, kg [µg]	Estimated [1] Uncertainty, kg [µg]
Sulfur	_____ $\times 10^{-9}$	± _____ $\times 10^{-9}$

Non-Certified, kg [µg]

Zinc ($\times 10^{-9}$)[2]

Note: Customary units are presented in brackets for user convenience and familiarity.

[1]The total estimated uncertainty is based on a 95% confidence, 95% coverage tolerance interval. This means that the true amount of sulfur will be covered by the certified value minus and plus the estimated uncertainty.

[2]The value in parenthesis for zinc is not certified, but is for information only.

NOTE: To convert the values to areal concentration in units of micrograms per square centimeter, divide the values in Table 1 by 10.06 cm^2.

Supplemental Information

Preparation: The films were fabricated at NIST by focused ion beam coating from a ZnS target onto polycarbonate filters (47 mm diameter, 0.1 µm pore size). Because of the sputtering process, the ZnS film composition does not correspond to ideal stoichiometry, but is sulfur enriched. The film composition, however, is sufficiently stable for its intended purpose.

The microhomogeneity of the sulfur and zinc in representative films was characterized by E. Steel of the Surface and Microanalysis Division using electron probe microanalysis, and was found suitable for this application. However, the uniformity of the sulfur deposit is not sufficient to recommend this SRM for standardization of x-ray microfluorescence instruments. (See recommended area).

X-ray absorption correction for sulfur: Because of the low mass thickness of these films, the x-ray attenuation of sulfur $K\alpha$ X rays should be negligible. However, if the user wishes to apply a mass attenuation correction for sulfur the magnitude of the correction will depend on the type of excitation source and source-sample-detector geometry of the spectrometer employed. For best results the correction should be determined for the particular instrument used.

Other elements not certified: Because of the nature of the sputtering process, some argon is entrapped in the film. Also, the major contaminant in the film is aluminum, which should not exceed ten per cent of the sulfur mass. Other contaminants from the vacuum chamber are chromium, iron, and nickel, at low levels.

Analytical Methods

1. Isotope dilution thermal ionization mass spectrometry
2. X-ray fluorescence spectrometry
3. Electron probe microanalysis

Chemical Science and Technology Laboratory

Analysts

1. K.E. Murphy
2. W.R. Kelly
3. P.A. Pella
4. E.B. Steel

Cooperating Analysts

-R.H. Sarver, J. Cooper, Keystone/NEA, Tigard, Oregon.
-R.D. Giauque, Lawrence Berkeley Laboratory, University of California, Berkeley, California.
-T. Cahill, R. Eldred, Physics-Air Quality Group, University of California, Davis, California.