U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Calibration

Standard Reference Material 2031

Metal-on-Quartz Filters for Spectrophotometry

This Standard Reference Material (SRM) is intended for use in the verification of the transmittance and absorbance scales of conventional spectrophotometers in the ultraviolet and visible regions of the electromagnetic spectrum. SRM 2031 consists of three individual filters in their metal holders and one empty filter holder. Two filters, having nominal transmittances of 10 and 30 percent, were produced by evaporating different thicknesses of chromium metal on 1.5-mm thick fused silica plates that had been precision ground and polished. These metal films are protected by 1.5-mm clear fused silica cover plates optically contacted to the base plates. The third filter is a single fused silica plate 3-mm thick, having a nominal transmittance of 90 percent. The metal holders for these filters are provided with shutters to protect the filters when not in use. The shutters must be removed at the time of measurement and be replaced after the measurements have been completed. Each filter holder bears a filter number (10, 30, or 90) and a set identification number.

Set and Filter Ident. Number	TRANSMITTANCE (T)									
	Wavelength, nm									
	250.0	280.0	340.0	360.0	400.0	465.0	500.0	546.1	590.0	635.0

Set and Filter Indent. Number	TRANSMITTANCE DENSITY ($-\log_{10}T$)									
	Wavelength, nm									
	250.0	280.0	340.0	360.0	400.0	465.0	500.0	546.1	590.0	635.0

Date of Certification:

Gaithersburg, MD 20899
October 22, 1984
(Revision of Certificates
dated 6-1-79 and 9-20-82)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(over)

The transmittance values (T) can be converted to percent transmittance (%T) by multiplying by 100. The transmittance densities were calculated from the measured transmittance (T). These transmittance densities should be indicated by the absorbance scale of the spectrophotometer when the filters are measured against air. All of the certified transmittance values were obtained by measuring against air at an ambient temperature of 23.5 °C.

The uncertainty of the certified transmittance values of the two metal film filters is ± 1.0 percent. This uncertainty includes 0.5 percent for random and systematic errors of the calibration procedure, as well as 0.5 percent for possible changes in the transmittance with time. The total uncertainty of the certified transmittance values for the clear quartz plate is 0.5 percent (see note below). The long-term stability of the filters with the chromium film has not been rigorously established. Measurements to date, however, suggest that the transmittance of the chromium-coated filters could change by as much as 0.5 percent in the first year after calibration. Therefore, this SRM should be returned to the National Bureau of Standards for free verification of the transmittance values on the first anniversary of the certification date. Information regarding subsequent verifications or recalibrations, including costs for such service, will be included with this first verification report. Before returning this SRM to NBS, information regarding such shipment should be obtained from the Service Analysis Coordinator, Center for Analytical Chemistry, Room B222, Chemistry Building, National Bureau of Standards, Gaithersburg, MD 20899. Telephone: (301) 921-2141.

When not in use, the filters should be stored in their holders with the shutters in place and in the metal container provided for this purpose. Extended exposure to laboratory atmosphere and dirty surroundings should be avoided.

The transmittance measurements were made using the high-accuracy spectrophotometer designed and built in the NBS Center for Analytical Chemistry [1]. This instrument is a primary transmittance standard; its transmittance accuracy was established using the double-aperture method of linearity testing [1,3,5,6].

Transmittance measurements for SRM 2031 were made by producing the vertical image of the slit (about 8 mm by 1.5 mm), using a convergent beam geometry with an aperture ratio f:10, in the middle of the entrance face of the filter. The filters were measured in the spectrophotometer in a position perpendicular to the incident light beam. A spectral bandpass of 1.6 nm was used for measurements at all wavelengths. Because the transmittances of these filters exhibit an appreciable optical neutrality, the dependence of transmittance on bandpass is not critical and wider bandpasses may be used in routine measurements. For a quantitative discussion of this subject, the user should consult reference 5, pp. 32 and 33.

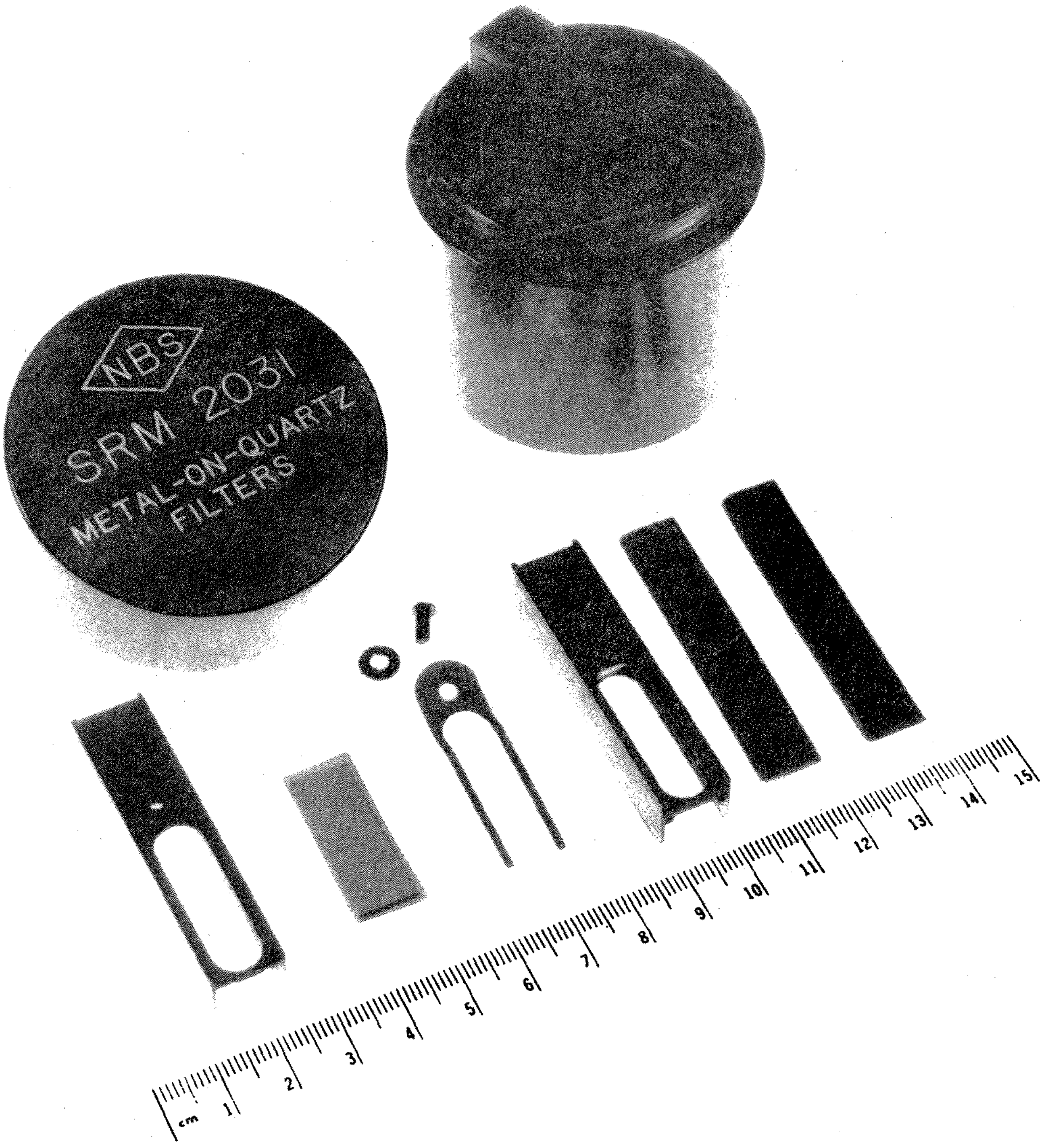
The transmittance, T, was measured against air in the reference beam; hence it includes the reflection losses that occur at the air-filter interface. Under these circumstances the measured transmittance, T, has a corresponding transmittance density, defined as $-\log_{10} T$. The internal transmittance, T_i, of a material is defined as the transmittance of the material corrected for reflection losses. This is obtained experimentally when the measurements are made against a blank sample in the reference beam. The absorbance, A, of a material is related to this internal transmittance, T_i, by the expression, $A = -\log_{10} T_i$.

The exposed surface of each filter is approximately 29 x 8 mm, measuring from a point 1.5 mm above the base of the filter holder (see figure). The empty filter holder provided is to be used in the reference beam of the spectrophotometer so that approximately equivalent conditions of stray radiation are achieved for both beams. The transmittance of the filters depends upon the intrinsic properties of the material, wavelength, spectral bandpass, geometry of the optical beam, temperature, and positioning of the filter. While changes in ambient temperature of 1 or 2 °C from 23.5 °C have not significantly affected the calibration, the effect of temperature variations exceeding 2 °C have not been investigated. Changes in the transmittance may be caused by surface conditions, aging of the material, exposure to a harmful atmosphere, or careless handling [2,3,4,5,7].

SRM 2031 is stored in a black-anodized aluminum container provided with a threaded cap made of the same metal. Each filter is placed in a cylindrical cavity to prevent any contact between the filter face and the walls of the storage container. Contamination of the filter surface with particulate matter due to static charges is minimized through the metallic nature of the container. A flat leaf spring is inserted into the cylindrical cavity with each filter holder to minimize damage during transportation. These springs can be removed during normal use in the laboratory.

NOTE: In some commercial instruments, the metal-on-quartz filters can generate reflection effects in the sample compartment that can degrade the accuracy of the measured transmittances. During the development of SRM 2031, the presence and magnitude of reflection effects were studied and were found negligible, within the uncertainty specified, in all spectrophotometers tested (see Ref. 5 page 4, pp. 16-30 for additional details of this study). However for certain instruments, these effects could become significant. If such effects are detected or suspected, the user should contact R.W. Burke, NBS Inorganic Analytical Research Division, for assistance and instructions.

The filter is shown in the assembled unit with its front surface facing up. The filter, in its filter holder, should be placed in the cuvette compartment of the spectrophotometer with its front surface facing the incident light beam and the rear surface facing the photodetector.



Top: Cylindrical container with its screw cap, both made of black-anodized aluminum alloy. Four filter holders can be stored in the cylindrical container. Bottom (from left to right): Aluminum alloy filter holder, 12.5 mm square and 58 mm high; Metal-on-quartz filter; Retaining spring of beryllium-copper with nylon screw and washer; Assembled unit; and Two Delrin shutters. All metal and plastic parts are flat black.

Prior to certification measurements, each filter was examined for surface defects and the condition of the optical contact [5]. Should the surface of the filter become contaminated, no attempt should be made to clean it unless the user has the facilities to demonstrate that the cleaning treatment will not alter the surface or degrade the accuracy of the certified values. As SRM 2031 is a transfer standard, the only means available to verify its integrity is to remeasure its transmittance with a primary standard instrument similar to that used in this certification [1,5]. In most cases, where verification or recertification of the transmittance values is desirable, it will be most expeditious to return the filters to the National Bureau of Standards for measurement.

Further information concerning the selection, preparation, and properties of SRM 2031 will be found in reference 5.

The research, development, and initial production of this SRM were conducted by R. Mavrodineanu and J.R. Baldwin, NBS Inorganic Analytical Research Division.

The transmittance measurements were performed by R.W. Burke and M.V. Smith, NBS Inorganic Analytical Research Division. Technical leadership for the preparation and measurements leading to certification was provided by R.W. Burke.

The overall direction and coordination of technical measurements leading to certification were performed under the chairmanship of J.R. DeVoe, NBS Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by L.J. Powell.

We wish to acknowledge the cooperation of George N. Bowers, Jr., M.D., of Hartford Hospital, Hartford, Connecticut; Royden N. Rand, Ph.D., of the Eastman Kodak Co. Research Laboratories, Rochester, New York; and Donald S. Young, M.D., Ph.D., of the Mayo Clinic, Rochester, Minnesota.

The contributions of H.E. Bennett and J.M. Bennett of the Michelson Laboratory, Physical Optics Branch, Naval Weapons Center, China Lake, California, who helped in the initial production of SRM 2031 are also gratefully acknowledged.

References

1. R. Mavrodineanu, An Accurate Spectrophotometer for Measuring the Transmittance of Solid and Liquid Materials, NBS Journal of Research 76a, No. 5, 405-425 (1972).
2. R. Mavrodineanu, Solid Materials to Check the Photometric Scale of Spectrophotometers, NBS Tech. Note 544, O. Menis, and J.I. Shultz, Eds., pp. 6-17, U.S. Government Printing Office, Washington, D.C. 20402 (Sept. 1970); ibid, NBS Tech. Note 584, pp. 2-21 (December 1971).
3. K.S. Gibson, Spectrophotometry, NBS Circ. 484 (Sept. 1949).
4. R. Mavrodineanu, Considerations for the Use of Semi-Transparent Metallic Thin Films as Potential Transmittance Standards in Spectrophotometry. NBS Journal of Research 80A, No. 4, 637-641 (1976).
5. R. Mavrodineanu and J.R. Baldwin, Metal-on-Quartz Filters as a Standard Reference Material for Spectrophotometry, SRM 2031. NBS Special Publication 260-68, U.S. Government Printing Office, Washington, D.C. 20402 (1979).
6. K.D. Mielenz and K.L. Eckerle, Spectrophotometer Linearity Testing Using the Double-Aperture Method, Appl. Optics 11, 2294-2303 (1972).
7. R.W. Burke and R. Mavrodineanu, Accuracy in Analytical Spectrophotometry NBS Special Publication 260-81, U.S. Government Printing Office, Washington, DC 20402 (1983).