National Institute of Standards & Technology

Certificate

Standard Reference Material® 2031a

Metal-on-Fused-Silica Filters for Spectrophotometry

This Standard Reference Material (SRM) is a primary transfer standard certified using the NIST National Reference Spectrophotometer [1,2]. It is intended for use in the verification of the transmittance and absorbance scales of spectrophotometers in the ultraviolet and visible spectral regions. SRM 2031a is a set that consists of three individual neutral density filters in separate metal holders and one empty filter holder stored in a black anodized aluminum container [3,4]. The exposed surface of each filter is approximately 29 mm × 8 mm, measuring from a point 1.5 mm above the base of the filter holder (see Figure 1). The filter holders are provided with shutters that protect the filters when not in use. Each filter-containing holder bears an identification number for the set and an individual filter number (10, 30, or 90) that corresponds to the nominal percent transmittance (100 × transmittance) of the filter. SRM 2031a differs from the prior series, SRM 2031, only with respect to the optical polishing tolerances.

Certified Values of Transmittance Density and Transmittance: Certified transmittance density values, independently determined for each filter at 22 °C ± 1 °C and at ten wavelengths in the ultraviolet and visible portions of the electromagnetic spectrum, are given in Table 1a. These values are calculated from measured transmittances (T) as $-\log_{10}(T)$, and should be indicated by the absorbance (A) scale of the spectrophotometer, if the filters are measured against air. The corresponding certified transmittance values are given in Table 1b. The expanded uncertainties for the certified transmittance density values of Table 1a are calculated from uncertainty components given in Tables 2, 3, and 4 (see Determination of Expanded Uncertainties). The expanded uncertainties for the transmittance values of Table 1b are calculated from the transmittance density uncertainties. The expanded uncertainties allow for possible changes due to slight surface contamination and fundamental material effects over the two year period following certification. The certified values are valid for instrumental spectral slit width values of 10 nm or less.

Expiration of Certification: This certification is valid within the measurement uncertainties specified for two years from the date of certification specified for this set in Tables 1a and 1b, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Instructions for Use). However, the certification will be nullified if the SRM is altered, contaminated, or damaged. The set may be returned to NIST for cleaning and recertification at two year intervals. Recertification can be arranged by contacting the NIST Optical Filters Program at (301) 975-4115.

The overall direction and coordination of technical measurements leading to certification were performed by J.C. Travis and G.W. Kramer of the NIST Analytical Chemistry Division.

The production and certification of this SRM were performed by M.V. Smith, J.C. Travis, and D.L. Duewer of the NIST Analytical Chemistry Division with the assistance of M.D. Maley of the NIST Analytical Chemistry Division.

Statistical support was provided by H.-k. Liu of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.W.L. Thomas.

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See Certificate Revision History on Page 4

INSTRUCTIONS FOR USE

The measured transmittance of the filters depends upon the intrinsic properties of the material and the wavelength, spectral slit width, and geometry of the optical beam. It can be affected by other factors such as stray light, temperature, and the positioning of the filter. Changes in the transmittance may be caused by changes in surface conditions, aging of the filter, exposure to a harmful atmosphere, or improper handling (see Storage and Handling) [3,4]. Because the transmittance of these filters exhibits appreciable optical neutrality, the dependence of transmittance on slit width is not critical, and spectral slit widths up to 10 nm may be used.

Instrument verification should be performed at an ambient temperature between 20 °C and 24 °C. The empty filter holder provided is to be used in the reference beam of the spectrophotometer so that approximately equivalent stray radiation conditions are maintained for both beams. The shutters provided with each filter must be removed at the time of measurement and replaced after the measurements have been completed. Measurements performed outside of these specified conditions or the optical geometry used for certification (see Determination of Transmittances) could produce transmittance values that differ from the certified values.

To demonstrate that a user's measurements are traceable, within acceptable limits, to the accuracy transferred by SRM 2031a, the user must first determine the required tolerances or acceptable uncertainty for the application in question. It is recommended that a number of replicate measurements be made for each filter and wavelength, with removal and replacement of the filter between replicate measurements. The user should then compare each mean value and the user-defined tolerance with the NIST certified value and expanded uncertainty (see Table 1a or 1b). An acceptable level of agreement between a user's measurements and the certified value is demonstrated if any part of the range defined by the NIST certified value and its expanded uncertainty overlaps any part of the user's tolerance band defined by the measured mean and the user-defined level of acceptable uncertainty [5].

NOTICE AND WARNINGS TO USERS

Storage and Handling: Each SRM 2031a set is stored in an aluminum container to minimize contamination of the filter surfaces with particulate matter due to static charge. Each filter is placed in a cylindrical cavity to prevent any contact between the filter face and the walls of the storage container. Each filter holder is provided with a flat leaf spring that is inserted into the cylindrical cavity of the unit for transportation. These springs should be removed during use. **Improper storage or handling of the filters may cause changes in the transmittance**. It is recommended that the filter in the holder be handled only by the edges with soft, powder-free, plastic (polyethylene) gloves and optical lens tissue. When not in use the filters should be stored in the container. Extended exposure to laboratory atmosphere and dusty surroundings should be avoided. If the surface of the filter becomes contaminated, the SRM set should be returned to NIST for recertification; do **NOT** attempt to clean it.

Instrument Dependence Warning: In some commercial instruments, the metal-on-fused-silica 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 such effects were studied and were found to be negligible, within the uncertainty specified, in the spectrophotometers tested [4]. However, for certain instruments, these effects could become significant. If such effects are detected or suspected, the user should contact J.C. Travis, NIST Analytical Chemistry Division, at (301) 975-4117 or via email at optical.filters@nist.gov for assistance and instructions.

Source and Preparation of Material: Ground and polished fused-silica components were produced by Starna Cells, Inc., Atascadero, CA. The evaporative chromium coating was applied by CVI Laser Corporation, Albuquerque, NM. Filters 10 and 30 were produced by evaporating different thicknesses of chromium metal onto 1.5 mm thick fused-silica plates that have been precision ground and polished. These metal films are protected by 1.9 mm thick fused-silica cover plates optically contacted to the base plates. Filter 90 is a 3 mm thick fused-silica plate having a nominal transmittance of 0.9. Each fused-silica piece used in SRM 2031a has been polished to a flatness of two fringes of the mercury green line (546.1 nm), and assembled filters were tested for a deviation from parallelism of less than 2×10^{-4} radians. Prior to certification measurements, the filters were aged at NIST for at least six months, and each filter was examined for surface defects and the condition of the optical contact.

Determination of Transmittances: The transmittance measurements are made against air (an empty filter holder) at an ambient temperature of 22 °C ± 1 °C using a high-accuracy spectrophotometer designed and built in the NIST Analytical Chemistry Division [1,2]. This instrument represents a primary transmittance standard; its transmittance accuracy is established using the double aperture method of linearity testing [1,2,6]. The effective spectral slit width used to determine the certified values is 1.0 nm. The transmittance measurements are made by projecting the vertical image of the slit (approximately 4 mm × 1 mm) onto the middle of the entrance face of the filter using 1:1 imaging and convergent beam geometry with an aperture ratio of f/10. The filter is mounted in a multiple filter carriage in the spectrophotometer. Each transmittance value reported in Table 1b is the average of three transmittance values over the several minute period required for three carriage cycles. The filters are measured in the spectrophotometer in a position perpendicular to the incident light beam, as shown in Figure 1. Each transmittance value is calculated from a measurement of the intensity transmitted through the filter and bracketing measurements of the intensity transmitted through an empty filter holder, with a settling time of approximately 3 seconds followed by a signal integration time of approximately 2 seconds. Transmittance is monitored several times over the filter aging period of at least six months. Only the final measurements are used as the basis for the certified values.

Uniformity: The transmittance density uniformity of each SRM 2031a filter is tested at all certified wavelengths by comparing the transmittance density measured at the center of each filter with that measured 7 mm below the center. Filters are rejected if the relative difference of the two readings exceeds the allowable limits that have been established. These limits are 0.0035 absorbance units for filter 10, 0.003 absorbance units for filter 30, and 0.001 absorbance units for filter 90. These limits are reflected in Table 4 and were established experimentally for prior filters of this type by sampling 1 mm × 4 mm areas over a region 5 mm wide × 20 mm long and located symmetrically about the center face of each filter [7].

Determination of Expanded Uncertainties: The expanded uncertainties, U, of the certified transmittance density values of Table 1a, reported in Table 2, are determined from combined standard uncertainties (i.e., estimated standard deviations) of component uncertainties reported in Tables 3 and 4, and a coverage factor $k = 2$ based on the Student's t-distribution for more than 30 degrees of freedom (DF) [8]. The expanded uncertainty defines an interval within which the unknown value of the transmittance can be asserted to lie with a level of confidence of approximately 95 %. This uncertainty includes "Type A" uncertainties evaluated by statistical methods and "Type B" uncertainties evaluated by other means. The standard uncertainties are combined by the root-sum-of-squares method.

The Type A standard uncertainty component for each level (Table 3) was determined from the results of a statistical analysis of three replicate measurements at each certification wavelength for 12 filters at each level (10, 30, and 90). The pooled standard deviation of replicates, s_p, was computed as the standard uncertainty for each level and wavelength. The standard uncertainty for the four wavelengths furnished by the UV lamp were indistinguishable from each other and slightly higher than that for the six wavelengths representing the visible lamp. The four UV wavelength standard uncertainties were pooled to represent a single conservative standard uncertainty for each of the ten wavelengths. The DF for the pooled standard uncertainty is given by multiplying the DF for each filter (2) by the number of filters (12) and the number of UV wavelengths (4).

Most of the Type B uncertainty components of Table 4 were estimated from studies described in NIST Special Publication 260-68 [4]. The Type B standard uncertainties are derived from an estimate of the ranges of the components [8].

REFERENCES

[1] Mavrodineanu, R., "An Accurate Spectrophotometer for Measuring the Transmittance of Solid and Liquid Materials," NBS Journal of Research **76A**, No. 5, pp. 405-425, (1972).

[2] Burke, R.W. and Mavrodineanu, R., "Accuracy in Analytical Spectrophotometry," NBS Special Publication 260-81, (1983).

[3] Mavrodineanu, R., "Considerations for the Use of Semi-Transparent Metallic Thin Films as Potential Transmittance Standards in Spectrophotometry," NBS Journal of Research **80A**, No. 4, pp. 637-641, (1976).

[4] Mavrodineanu, R. and Baldwin, J.R., "Metal-on-Quartz Filters as a Standard Reference Material for Spectrophotometry, SRM 2031," NBS Special Publication 260-68, (1979).

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[6] Mielenz, K.D. and Eckerle, K.L., "Spectrophotometer Linearity Testing Using the Double-Aperture Method," Appl. Optics **11**, pp. 2294-2303, (1972).

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[8] *Guide to the Expression of Uncertainty in Measurement*, ISBN 72-67-10188-9, lst Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC, (1994); available at http://physics.nist.gov/Pubs.

Certificate Revision History: 3 April 2002 (This revision reflects an editorial change to the certificate); 24 July 2001 (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.

TABLE 1a. Certified Transmittance Density Values for SRM 2031a, Set Number 654

Wavelength, nm	TRANSMITTANCE DENSITY ($-\log_{10}T$) SRM 2031a Set Identification - Filter Number		
	654-10	654-30	654-90
250.0	0.9889 ± 0.0042	0.4324 ± 0.0039	0.0367 ± 0.0019
280.0	1.0085 ± 0.0042	0.4292 ± 0.0039	0.0342 ± 0.0019
340.0	1.0340 ± 0.0042	0.4376 ± 0.0039	0.0318 ± 0.0019
360.0	1.0278 ± 0.0042	0.4378 ± 0.0039	0.0313 ± 0.0019
400.0	1.0286 ± 0.0042	0.4404 ± 0.0039	0.0308 ± 0.0019
465.0	1.0518 ± 0.0042	0.4417 ± 0.0039	0.0301 ± 0.0019
500.0	1.0423 ± 0.0042	0.4349 ± 0.0039	0.0300 ± 0.0019
546.1	1.0047 ± 0.0042	0.4195 ± 0.0039	0.0295 ± 0.0019
590.0	0.9613 ± 0.0042	0.4025 ± 0.0039	0.0295 ± 0.0019
635.0	0.9231 ± 0.0042	0.3854 ± 0.0039	0.0294 ± 0.0019

SAMPLE

Date of Certification: October 30, 2001

TABLE 1b. Certified Transmittance Values for SRM 2031a, Set Number **654**

Wavelength, nm	TRANSMITTANCE (T)		
	SRM 2031a Set Identification - Filter Number		
	654-10	**654-30**	**654-90**
250.0	0.1026 ± 0.0010	0.3695 ± 0.0033	0.9189 ± 0.0038
280.0	0.0981 ± 0.0010	0.3722 ± 0.0033	0.9243 ± 0.0038
340.0	0.0925 ± 0.0009	0.3651 ± 0.0032	0.9294 ± 0.0038
360.0	0.0938 ± 0.0009	0.3649 ± 0.0032	0.9305 ± 0.0038
400.0	0.0936 ± 0.0009	0.3627 ± 0.0032	0.9315 ± 0.0038
465.0	0.0887 ± 0.0009	0.3617 ± 0.0032	0.9330 ± 0.0038
500.0	0.0907 ± 0.0009	0.3674 ± 0.0032	0.9334 ± 0.0038
546.1	0.0989 ± 0.0010	0.3806 ± 0.0033	0.9343 ± 0.0038
590.0	0.1093 ± 0.0011	0.3958 ± 0.0035	0.9344 ± 0.0038
635.0	0.1194 ± 0.0012	0.4118 ± 0.0036	0.9346 ± 0.0038

SAMPLE

Date of Certification: October 30, 2001

Table 2. Transmittance Density Uncertainty for SRM 2031a

SRM 2031a	Transmittance Density Uncertainty		
	Filter Number		
	10	30	90
Type A	0.0003	0.0002	0.0001
Combined Type B	0.0021	0.0019	0.0009
Combined Uncertainty (u_c)	0.0021	0.0020	0.0010
Effective Degrees of Freedom	>30	>30	>30
Coverage Factor	2	2	2
Expanded Uncertainty (U)	0.0042	0.0039	0.0019

Table 3. Type A Components of Transmittance Density Uncertainty for SRM 2031a

Source	Standard Uncertainty			DF
	Filter Number			
	10	30	90	
Pooled Replicate Measurements	0.0003	0.0002	0.0001	96

Table 4. Type B Components of Transmittance Density Uncertainty for SRM 2031a

Source	Standard Uncertainty			DF
	Filter Number			
	10	30	90	
Homogeneity	0.0014	0.0012	0.0004	∞
Transmittance Stability	0.0015	0.0015	0.0008	∞
Temperature	0.0001	0.0001	0.0001	∞
Linearity/Geometry	0.0003	0.0001	0.0003	∞
Combined Type B Uncertainty	0.0021	0.0019	0.0009	∞

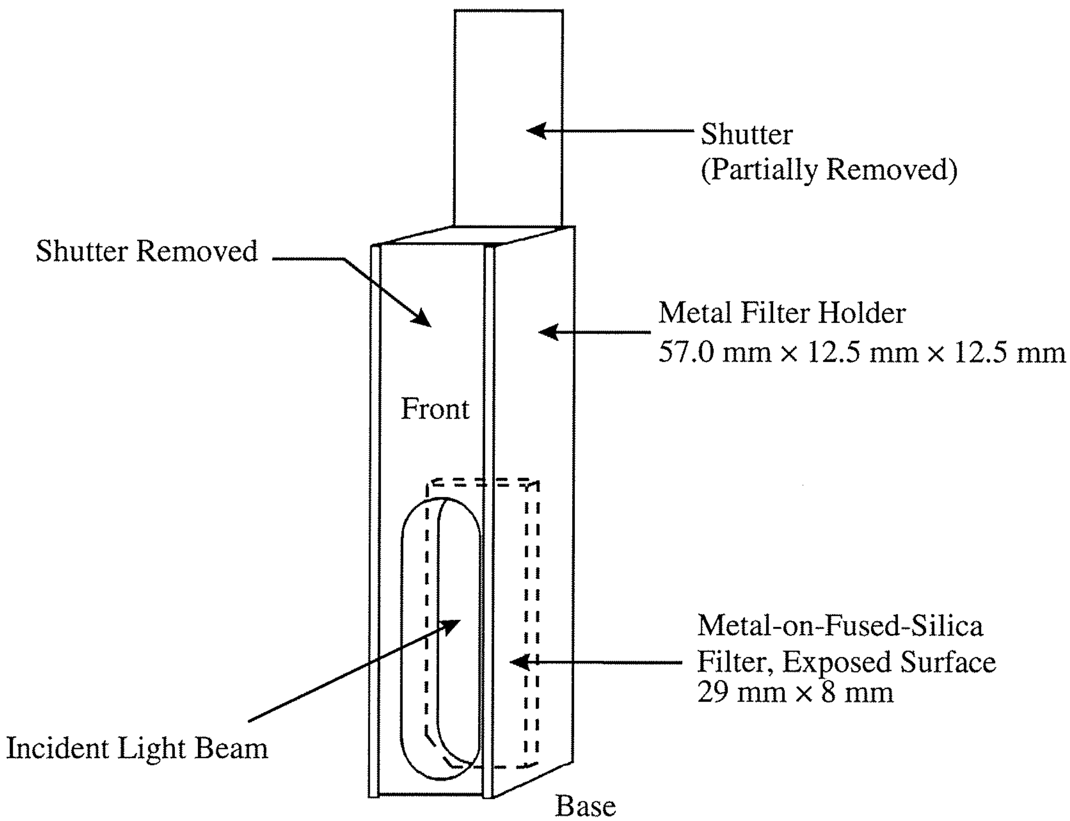

Figure 1. Metal Holder for Metal-on-Fused-Silica Filters