National Institute of Standards & Technology

# Certificate

## Standard Reference Material® 2031b

### Metal-on-Fused-Silica Neutral Density Filters (250 nm to 635 nm)

Set Identification: <<Set Serial No>>

This Standard Reference Material (SRM) is intended for use in the verification of the transmittance and absorbance scales of spectrophotometers in the ultraviolet and visible spectral regions. SRM 2031b is a transfer standard certified using the NIST Materials Measurement Laboratory Transfer Spectrophotometer (MMLTS) [1] with traceability through the NIST second generation High Accuracy Spectrophotometer (HAS II), modeled after the original instrument [2]. A unit consists of three individual neutral-density filters in separate metal holders and one empty filter holder, all 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.

**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 1. 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 with the empty filter holder in the reference beam. The corresponding certified transmittance values are given in Table 2. 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 5 nm or less (see “Instrument Dependence Warning”).

**Expiration of Certification:** This certification of **SRM 2031b** is valid, within the measurement uncertainty specified, for two years from the date of certification listed in Tables 1 and 2, provided the SRM is handled and stored in accordance with instructions given in this certificate (see “Instructions for Handling, Storage, and Use”). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

**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. Registration (see attached sheet) will facilitate notification. The set may be returned to NIST for cleaning and recertification as required by expiration or contamination. Recertification can be arranged by contacting the NIST Optical Filters Program at (301) 975–8533 or at optical.filters@nist.gov. Instructions for recertification are located on the NIST web site [5].

Overall direction and coordination of technical measurements leading to certification were performed by S.J. Choquette of the NIST Biosystems and Biomaterials Division.

The production and certification of this SRM were performed by M.V. Smith and J.C. Travis of the NIST Biosystems and Biomaterials Division, and D.L. Duewer of the NIST Chemical Sciences Division.

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

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Certificate Issue Date: 03 May 2013

Robert L. Watters, Jr., Director
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## INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

**Handling and Storage:** The SRM 2031b 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. It is recommended that the filter in the holder be handled only by the edges with soft, powder-free, polyethylene gloves, and optical lens tissue. When not in use, the filters should be stored in the supplied storage 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. **Note:** Improper storage or handling of the filters may cause changes in the transmittance.

**Use:** The measured transmittance of the filters depends upon the intrinsic properties of the material and the wavelength 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 [3,4]. Because the transmittance of these filters exhibits appreciable optical neutrality, the dependence of transmittance on slit width is not anticipated, and spectral slit widths up to 5 nm may be used (See "Instrument Dependence Warning").

Instrument verification should be performed at a sample 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 2031b, 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 in Table 1 or Table 2. 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 [6].

*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. In addition, unexpected spectral bandwidth effects have also been reported in some commercial instruments. If such effects are detected or suspected, the user should contact NIST Biosystems and Biomaterials Division, at (301) 975-4117 or via email at optical.filters@nist.gov for assistance and instructions as described in reference 5.

## PREPARATION AND VALUE ASSIGNMENT(1)

**Preparation**: The neutral density inserts to filters 10 and 30 were produced by evaporating different thicknesses of inconel metal alloy onto 3.0 mm thick fused-silica plates that have been precision ground and polished by Starna Cells, Inc., (Atascadero, CA). The evaporative inconel coating was applied by CVI Laser Corporation (Albuquerque, NM). The metal films are protected by a 10 nm thick evaporated layer of silicon dioxide applied by Precision Photonics Corporation (Boulder, CO). Filter 90 is produced using an uncoated fused-silica plate identical to the substrates used for the other two filters. Each fused-silica piece used in SRM 2031b 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 $2 \times 10^{-4}$ radians. Prior to certification measurements, the filters were aged at NIST for a minimum of six months, and each filter was examined for surface defects. SRM 2031b differs from the prior series by using an evaporated silicon dioxide protective overcoating rather than a protective cover plate in the construction.

(1) Certain commercial equipment, instrumentation, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

**Determination of Transmittances:** The transmittance measurements are made with the empty filter holder used as the reference at an ambient temperature of 22 °C ± 1 °C using an Agilent Cary 6000i spectrophotometer qualified as a transfer spectrophotometer [1]. Transmittance traceability is maintained by comparison measurements to the second generation NIST HAS II [2] with the results of the comparison reflected in the certification uncertainties [1]. The effective spectral slit width used to determine the certified values is 0.8 nm. The transmittance measurements are made by projecting the vertical image of the slit onto the middle of the entrance face of the filter, with the optical centerline 15 mm above the base of the filter. The filter is mounted in a multiple filter carriage in the spectrophotometer. Each transmittance value reported in Table 2 is the average of three transmittance values measured with a one second averaging time, with the instrument cycling through the ten wavelengths, three times for each filter in the carriage. 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 a measurement of the blank (empty filter holder) at the beginning of the measurement of each set of three filters. Transmittance is monitored for temporal drift several times over the filter aging period of six months; the aging period is extended for sets with excessive drift. Only the final measurements are used as the basis for the certified values.

**Uniformity:** The transmittance density uniformity of each SRM 2031b filter is tested at five certified wavelengths by comparing the transmittance density measured at the center of each filter with that measured 5 mm above and 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 reflected in the uncertainty determination.

**Traceability:** Traceability in regular spectral transmittance is achieved by comparative measurements of metal-on-fused-silica neutral-density filters on the MMLTS and the HAS II. The master set of reference filters is calibrated annually by the HAS II and measured at least quarterly on the MMLTS to assess the comparability of the scales. The comparison measurements are used to compute the bias of the MMLTS with respect to the HAS II for each nominal transmittance density level and wavelength [1].

**Determination of Expanded Uncertainties:** The expanded uncertainties, $U$, of the certified transmittance density values of Table 1 are determined from the measured bias and from standard uncertainties (i.e., estimated standard deviation equivalents) of component uncertainty sources discussed below, and a coverage factor $k = 2$ based on the Student's $t$-distribution [1,7]. The expanded uncertainty defines an interval within which the unknown value of the transmittance density would be asserted to lie with a level of confidence of approximately 95 % for more than 30 degrees of freedom (DF). This uncertainty includes "Type A" uncertainties evaluated by statistical methods and "Type B" uncertainties evaluated by other means [7].

Bias determination for traceability was by means of three independent measurements made on each of two control sets on the MMLTS and on the HAS II. The average and sample standard deviation of the bias were computed for each filter level (10, 30, or 90) and wavelength. The maximum value of the bias and of the standard uncertainty of the bias over all ten wavelengths were assigned to conservatively represent all wavelengths at that level. The resulting bias and standard uncertainty of the bias were combined with the uncertainty components representing filter properties and direct measurement properties as described in reference 1 to yield the final expanded uncertainty values reflected in Tables 1 and 2.

Most of the Type B uncertainty components were estimated from studies described in NIST Special Publication 260-68 [4]. These components include uniformity (see "Uniformity"), temporal stability (drift), temperature dependence, and the linearity and geometry of the measurement. The Type B standard uncertainties are derived from an estimate of the ranges of the components [7].

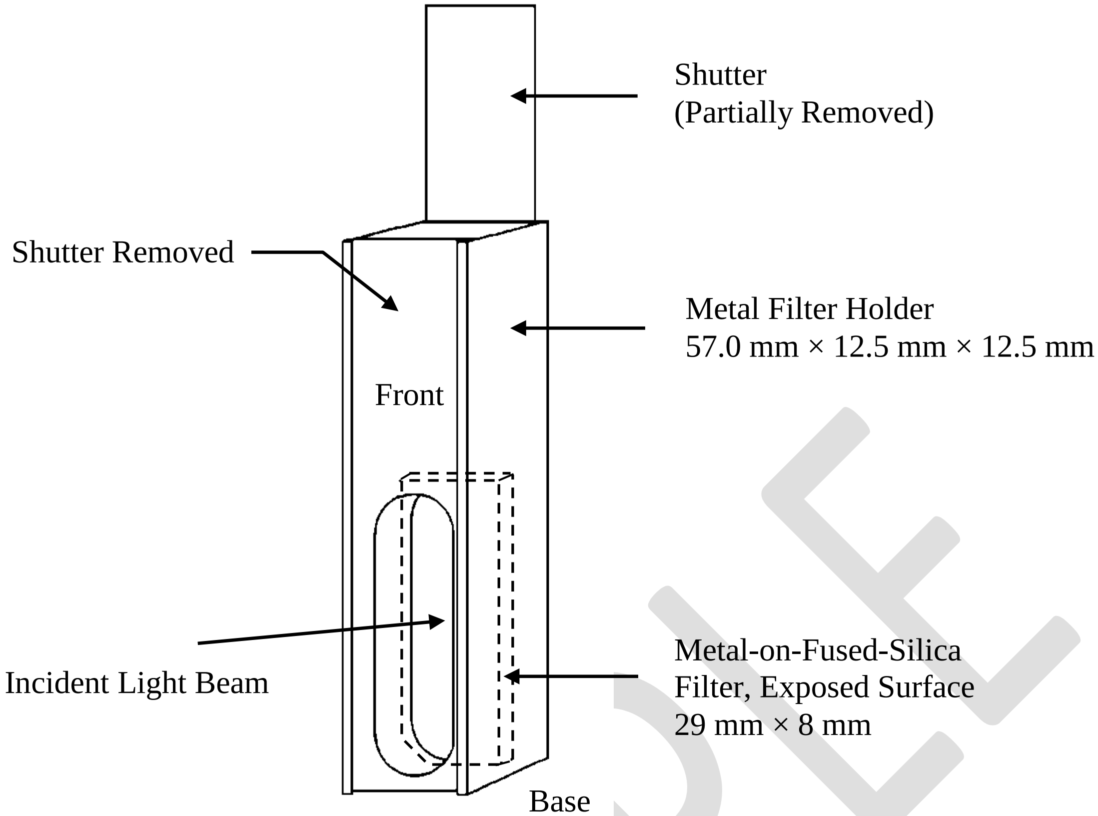


Figure 1. Metal holder for Metal-on-Fused-Silica Filters

Table 1. Certified Transmittance Density Values for SRM 2031b

| Wavelength (nm) | Transmittance Density ($-\log_{10}T$) | | |
|---|---|---|---|
| | Set Identification – Filter Number | | |
| | Set Serial No – 10 | Set Serial No – 30 | Set Serial No – 90 |
| 250.0 | Sample | Sample | Sample |
| 280.0 | Sample | Sample | Sample |
| 340.0 | Sample | Sample | Sample |
| 360.0 | Sample | Sample | Sample |
| 400.0 | Sample | Sample | Sample |
| 465.0 | Sample | Sample | Sample |
| 500.0 | Sample | Sample | Sample |
| 546.1 | Sample | Sample | Sample |
| 590.0 | Sample | Sample | Sample |
| 635.0 | Sample | Sample | Sample |

Date of Certification: <<date>>.

Table 2. Certified Transmittance Values for SRM 2031b

| Wavelength (nm) | Transmittance (T) | | |
|---|---|---|---|
| | Set Identification – Filter Number | | |
| | Set Serial No – 10 | Set Serial No – 30 | Set Serial No – 90 |
| 250.0 | Sample | Sample | Sample |
| 280.0 | Sample | Sample | Sample |
| 340.0 | Sample | Sample | Sample |
| 360.0 | Sample | Sample | Sample |
| 400.0 | Sample | Sample | Sample |
| 465.0 | Sample | Sample | Sample |
| 500.0 | Sample | Sample | Sample |
| 546.1 | Sample | Sample | Sample |
| 590.0 | Sample | Sample | Sample |
| 635.0 | Sample | Sample | Sample |

Date of Certification: <<date>>.

## REFERENCES


[1] Travis, J.C.: Smith, M.V.; Choquette, S.J.; Liu, H-k.; *Certified Transmittance Density Uncertainties for Standard Reference Materials using a Transfer Spectrophotometer*; NIST Technical Note 1715 (2011); available at http://www.nist.gov/publication-portal.cfm (accessed May 2013). It is also available on the SRM details page as a data file available at https://www-s.nist.gov/srmors/view_detail.cfm?srm=2031B.

[2] Mavrodineanu, R.; *An Accurate Spectrophotometer for Measuring the Transmittance of Solid and Liquid Materials*, J. Res. NBS, Vol. 76A, p. 405 (1972).

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

[4] Mavrodineanu, R.; Baldwin, J.R.; *Metal-on-Quartz Filters as a Standard Reference Material for Spectrophotometry, SRM 2031*; NIST Special Publication 260-68 (1979); available at http://www.nist.gov/srm/upload/SP260-68.PDF (accessed May 2013).

[5] *NIST Recertification of Neutral Density Standard Reference Materials for Regular Spectral Transmittance*; available at http://www.nist.gov/mml/bbd/bioassay/optical_filter_recertification.cfm (accessed May 2013).

[6] Becker, D.; Christensen, R.; Currie, L.; Diamondstone, B.; Eberhardt, K.; Gills, T.; Hertz, H.; Klouda, G.; Moody, J.; Parris, R.; Schaffer, R.; Steel, E.; Taylor, J.; Watters, R.; Zeisler, R.; *Use of NIST Standard Reference Materials for Decisions on Performance of Analytical Chemical Methods and Laboratories*; NIST Special Publication 829 (1992).

[7] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed May 2013); see also Taylor, B.N.; 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://www.nist.gov/pml/pubs/tn1297/index.cfm (accessed May 2013).


*Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.*