National Institute of Standards & Technology

Certificate

Standard Reference Material® 2930

Glass Filters for Spectrophotometry

This Standard Reference Material (SRM) is a primary transfer standard certified using a national reference spectrophotometer at NIST [1-3]. SRM 2930 is complementary to SRM 930e and SRM 1930, providing levels of transmittance/absorbance that extend the range covered by those SRMs. It is intended for use in the verification of the transmittance and absorbance scales of spectrophotometers in the visible spectral region. SRM 2930 is a set consisting of three individual neutral-density glass filters in separate metal holders and one empty filter holder. The exposed surface of the glass 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 glass filters when not in use. Each filter-containing holder bears an identification number for the set and individual filter numbers (.1, .3, or 90), which correspond to the nominal percent transmittance (100 × transmittance) of each filter to one significant figure.

Certified Values of Transmittance Density and Transmittance: Certified transmittance density values independently determined for each filter at 22 °C ± 1 °C and at five wavelengths in the visible portion 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 relative to 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 materials effects over the two-year period following certification. Spectral bandwidth values indicated in parentheses beside the wavelengths of certification in Tables 1a and 1b are the maximum values for which the certified values are valid.

Expiration of Certification: This certification is valid, within the measurement uncertainties specified, for two years from the date of certification given for this set in Tables 1a and 1b, provided the SRM is handled and stored in accordance with the instructions given in this certificate. 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 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.

Transmittance measurements were performed by M.V. Smith 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 issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by P. Fagan of the NIST Measurement Services Division.

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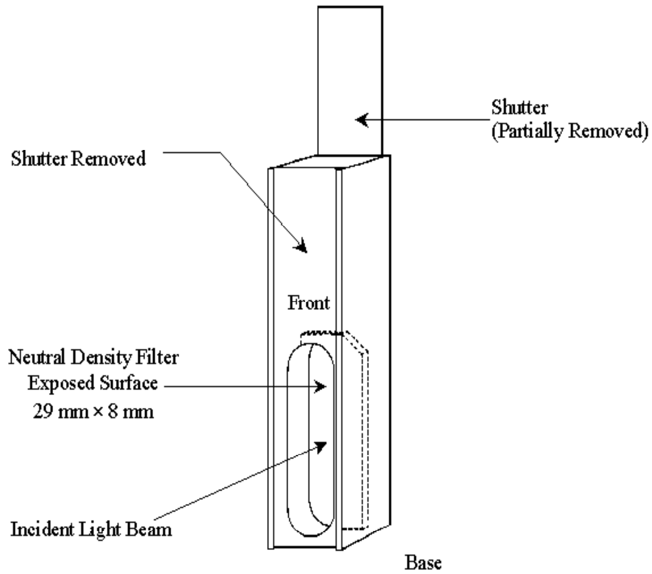

Figure 1. SRM 2930 Filter Holder

INSTRUCTIONS FOR USE

The transmittance of the filters depends upon the intrinsic properties of the material and the wavelength, spectral bandwidth, 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 glass, exposure to a harmful atmosphere, or careless handling as indicated (see "Storage and Handling") [1,4,5]. The measurement wavelengths indicated in Tables 1a and 1b should not be in error by more than 1 nm, and the maximum spectral bandwidth values should not be exceeded. The wavelength axis of the instrument may be calibrated using NIST SRM 2034 Holmium Oxide Solution Wavelength Standard from 240 nm to 650 nm.

Instrument verification should be performed at a sample temperature between 18 °C and 26 °C. 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 shutters provided with each filter must be removed at the time of measurement and be 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 defined by SRM 2930 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 (Table la or 1b). An acceptable level of agreement between a user's measurements and the certified value is assured 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].

Storage and Handling: Each SRM 2930 set is stored in a black anodized aluminum container to minimize contamination of the glass filter surfaces with particulate matter due to static charge. Each filter holder 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** [4]. It is necessary that the holder containing the filter be handled only by the metal sides and edges, and the use of soft, powder-free, plastic (polyethylene) gloves is recommended. When not in use, it should be stored in the container. Extended exposure to laboratory atmosphere and dusty surroundings should be avoided. However, dust may be removed by using a rubber-bulb air puffer without contacting the surface of the filter. If the surface of the glass filter becomes otherwise contaminated, no attempt should be made to clean it, and the SRM set should be returned to NIST for recertification.

Instrument Dependence Warning: Instruments for which wavelength dispersion occurs after the light has passed through the filter are particularly susceptible to minor deviations in the optical beam caused by the filter. If such effects are detected or suspected, the user should contact J.C. Travis, NIST Analytical Chemistry Division at (301) 975-4117, for assistance and instructions.

Source and Preparation of Material[1]: The neutral-density filters were produced by Starna Cells Inc., Atascadero, CA, from samples of Schott NG-9 (filters .1 and .3) and BK-7 (filter 90) glass, selected for best homogeneity and minimal inclusions and striae. The filters were ground and polished to the appropriate thicknesses to achieve the nominal transmittances of 0.001, 0.003, and 0.92 [1,4,5]. SRM 2930 has been polished to a flatness of one wavelength of the helium-neon laser 633 nm line over the central 5 mm × 20 mm filter area and to a parallelism of 0.1 mrad (20 ") or better. Prior to certification measurements, the glass filters were aged at NIST for at least six months, and each filter was examined for surface defects and thoroughly cleaned [4].

Determination of Transmittances: The transmittance measurements were made relative to air (an empty filter holder) at a sample temperature of 22 °C ± 1 °C using a high-accuracy spectrophotometer designed and built in the NIST Analytical Chemistry Division [3]. This instrument represents a primary transmittance standard; its transmittance accuracy is established over two decades in transmittance using the double-aperture method of linearity testing [3,4,7]. Accuracy beyond two decades is achieved by means of a two-stage "set-back" measurement described below. The effective spectral bandwidth used to determine the certified values was 0.8 nm. The transmittance measurements were made by projecting the vertical image of the slit (approximately 4 mm × 1 mm) onto the middle of the entrance face of the glass filter using 1:1 imaging and a convergent beam geometry with an aperture ratio of f/10. The filter was mounted in a multiple filter carriage in the spectrophotometer. The filters were 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 5 s followed by a signal integrating time of approximately 2 s. Each transmittance value recorded in a single experiment is the average of three transmittance measurements over the several minute time period required for three carriage cycles.

For the nominal 0.1 % and 0.3 % transmitting filters of this set, the transmittance is measured in the manner described, only relative to a nominal 10 % filter rather than relative to air. In this "set-back" method, the final reported transmittance is the product of the transmittance of the sample relative to the intermediate filter and the transmittance of the intermediate filter relative to air. For the two-decade double-aperture validation to apply to each of the two steps, the reference beam through the intermediate filter is adjusted to the same intensity as the reference beam through air, by means of the variable attenuator of the reference spectrophotometer.

Transmittance was determined several times during an aging period of at least six months. Only the final measurements were used as the basis of the certified values reported in Table 1b.

Uniformity: The transmittance uniformity of each SRM 2930 filter was tested at all five certification wavelengths to compare the transmittance density measured at the center of each filter with that measured 7 mm below the center. Filters were rejected if the relative difference of the two readings exceeded set tolerances of 0.0130, 0.0055, and 0.0013 absorbance units for filters .1, .3, and 90 (respectively) with a statistical confidence of 95 %. This limit is reflected in Table 4 and was established experimentally for early production filters of this type.

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 [8]. This uncertainty includes "Type A" uncertainties, which are evaluated by statistical methods, and "Type B" uncertainties, which are determined by other means. The standard uncertainties are combined by the root-sum-of-squares method. The expanded uncertainty defines an interval within which the unknown value of the transmittance density can be asserted to lie with a level of confidence of approximately 95 %.

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

The Type A standard uncertainty component for each level (Table 3) was determined from the results of a statistical analysis of six replicate measurements taken on different days at five wavelengths for eight filters at each of the three levels (.1, .3, and 90). The pooled standard deviation of replicates, s_p, was computed and reported as the standard uncertainty for each level. The degrees of freedom, ν for the pooled standard deviation were computed by multiplying the degrees of freedom for each set of replicates times the number of wavelengths for each level and filter times the number of filters at each level: $\nu = (6-1) \times 5 \times 8 = 200$.

The Type B uncertainty components of Table 4 were estimated from studies similar to those described in NIST Special Publication 260-116 [4] for the related glass filters. The Type B uncertainty components are derived from an estimate of the range ($\pm a_i$) with the assumption that the uncertainty is uniformly distributed. The resulting standard uncertainty component is then approximated as $a_i \div \sqrt{3}$ [8].

REFERENCES

[1] *Collected Papers from NBS Conference on Accuracy in Spectrophotometry and Luminescence Measurements*, J. Res. Natl. Bur. Stand., Vol. 76A, No. 5, pp. 375–510; US Government Printing Office: Washington, DC (1972).

[2] Burke, R.W.; Mavrodineanu, R.; *Accuracy in Analytical Spectrophotometry*; NBS Special Publication 260-81; US Government Printing Office: Washington, DC (1983).

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[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; U.S. Government Printing Office: Washington, DC (1992).

[7] Mielenz, K.D.; Eckerle, K.L.; *Spectrophotometer Linearity Testing Using the Double-Aperture Method*; Appl. Opt. 11, pp. 2294-2303 (1972).

[8] ISO; Gui*de to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); 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://physics.nist.gov/Pubs/.

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 at http://www.nist.gov/srm.

Table 1a. Certified Transmittance Density Values for SRM 2930, Set Number 123

Wavelength, nm (Maximum Spectral Bandwidth, nm)	**Transmittance Density ($-\log_{10}T$)**		
	Filter Number - Set Identification		
	.1 - 123	**.3 - 123**	**90 - 123**
440.0 (2.2)	3.0818 ± 0.0181	2.6502 ± 0.0090	0.0388 ± 0.0020
465.0 (2.7)	2.8823 ± 0.0181	2.4795 ± 0.0090	0.0384 ± 0.0020
546.1 (6.5)	2.9303 ± 0.0181	2.5212 ± 0.0090	0.0375 ± 0.0020
590.0 (5.4)	2.9724 ± 0.0181	2.5575 ± 0.0090	0.0373 ± 0.0020
635.0 (6.0)	2.7867 ± 0.0181	2.3980 ± 0.0090	0.0370 ± 0.0020

Table 1b. Certified Transmittance Values for SRM 2930, Set Number 123

Wavelength, nm (Maximum Spectral Bandwidth, nm)	**Transmittance (T)**		
	Filter Number - Set Identification		
	.1 - 123	**.3 - 123**	**90 - 123**
440.0 (2.2)	0.000828 ± 0.000034	0.002238 ± 0.000046	0.9144 ± 0.0042
465.0 (2.7)	0.001311 ± 0.000055	0.003315 ± 0.000069	0.9154 ± 0.0042
546.1 (6.5)	0.001174 ± 0.000049	0.003012 ± 0.000062	0.9172 ± 0.0042
590.0 (5.4)	0.001066 ± 0.000044	0.002770 ± 0.000057	0.9178 ± 0.0042
635.0 (6.0)	0.001634 ± 0.000068	0.003999 ± 0.000083	0.9184 ± 0.0042

Date of Recertification: 12/2/2003

Table 2. Transmittance Density Uncertainty for SRM 2930

Item	**Transmittance Density Uncertainty**		
	Filter Number		
	.1	**.3**	**90**
Combined Type A	0.0008	0.0005	0.0002
Combined Type B	0.0090	0.0045	0.0010
Combined Uncertainty ($\boldsymbol{u_c}$)	0.0090	0.0045	0.0010
Coverage Factor	2	2	2
Expanded Uncertainty ($\boldsymbol{U}$)	0.0181	0.0090	0.0020

Table 3. Type A Components of Transmittance Density Uncertainty for SRM 2930

Source	**Filter Number**			**DF**
	.1	**.3**	**90**	
Repeatability	0.0008	0.0005	0.0002	200

Table 4. Type B Components of Transmittance Uncertainty for SRM 2930

Source	**Filter Number**			**DF**
	.1	**.3**	**90**	
Uniformity	0.0075	0.0032	0.0008	∞
Transmittance Stability	0.0045	0.0025	0.0006	∞
Temperature	0.0018	0.0016	0.0001	∞
Linearity/Geometry	0.0010	0.0010	0.0002	∞
Combined Type B Uncertainty	0.0089	0.0043	0.0010	

Effective Degrees of Freedom > 30