National Institute of Standards & Technology

Certificate

Standard Reference Material® 1930

Glass Filters for Spectrophotometry

This Standard Reference Material (SRM) is a primary transfer standard certified using a national reference spectrophotometer at NIST [1-3]. It is complementary to SRM 930e, providing levels of transmittance/absorbance that are outside the range covered by that SRM. It is intended for use in the verification of the transmittance and absorbance scales of spectrophotometers in the visible spectral region. SRM 1930 is a set consisting of three individual glass neutral density filters in separate metal holders and one empty filter holder. The exposed surface of the glass is approximately 29 mm x 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 50), which correspond to the nominal percent transmittance (100 x transmittance) of each filter.

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 bandpass 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 K.R. Eberhardt and 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 Standard Reference Materials Program by J.W.L. Thomas

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See Certificate Revision History on Page 4

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Instructions for Use: The transmittance of the filters depends upon the intrinsic properties of the material and the wavelength, spectral bandpass, 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) [1,4-6]. The measurement wavelengths indicated in Tables 1a and 1b should not be in error by more than 1 nm, and the maximum spectral bandpass values should not be exceeded. The wavelength axis of the instrument may be calibrated using NIST SRM 2034 Holmium Oxide Solution Wavelength Standard.

Instrument verification should be performed at an ambient temperature between 20 °C and 24 °C [4]. 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 1930 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 [7].

Storage and Handling: Each SRM 1930 set is stored in a blue anodized aluminum container to minimize contamination of the glass 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** [4]. 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 it should be stored in the container. Extended exposure to laboratory atmosphere and dusty surroundings should be avoided. If the surface of the glass filter becomes contaminated, no attempt should be made to clean it. However, dust may be removed by using a rubber-bulb air puffer without contacting the surface of the filter. 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 by the SRM unit. 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: The neutral filters were produced by either Starna Cells Inc., Atascadero, CA, or Schott Glass Technologies, Inc. of Duryea, PA, from samples of Schott NG-3 and NG-11 glass, selected for best homogeneity and minimal inclusions and striae[1]. The filters were ground and polished to the appropriate thicknesses to achieve the nominal transmittances of 0.01, 0.03, and 0.5 [1,4,5]. SRM 1930 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 an ambient 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

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[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.

using the double-aperture method of linearity testing [3,4,6,8]. The effective spectral bandpass used to determine the certified values was 0.8 nm. The transmittance measurements were made by projecting the vertical image of the slit (approximately 6 mm x 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 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 time period required for three carriage cycles. 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. Transmittance was determined in this way several times during an aging period of at least six months. Only the final measurements were used as the basis of the certified values.

Uniformity: The transmittance uniformity of each SRM 1930 filter was tested at 546 nm (with a 30 nm effective spectral bandpass) using a commercial spectrophotometer 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 the allowable limit of 0.0013 absorbance units with a statistical confidence of 95 %. This limit is reflected in Table 4 and was established experimentally for prior filters of this type by sampling 1 mm x 4 mm areas over a region 5 mm wide by 20 mm long and located symmetrically about the center face of each filter [9].

Determination of Overall 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 t-distribution for more than 30 degrees of freedom [10]. 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 %.

The Type A standard uncertainty component for each level (Table 3) was determined from the results of a statistical analysis of two replicate measurements taken on different days at five (5) wavelengths for twelve (12) filters at each of the three levels (1, 3, and 50). The pooled standard deviation of replicates, s_p, was computed and reported as the standard uncertainty for each level. The degrees of freedom, *DF*, for the pooled standard deviation was computed by multiplying the degrees of freedom for each set of replicates times the number of filters at each level by the number of wavelengths for each level and filter: *DF = 1´ 12 ´ 5 = 60*.

The Type B uncertainty components of Table 4 were estimated from studies described in NIST Special Publication 260-116 [4]. 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 $a_i \div \sqrt{3}$ [10].

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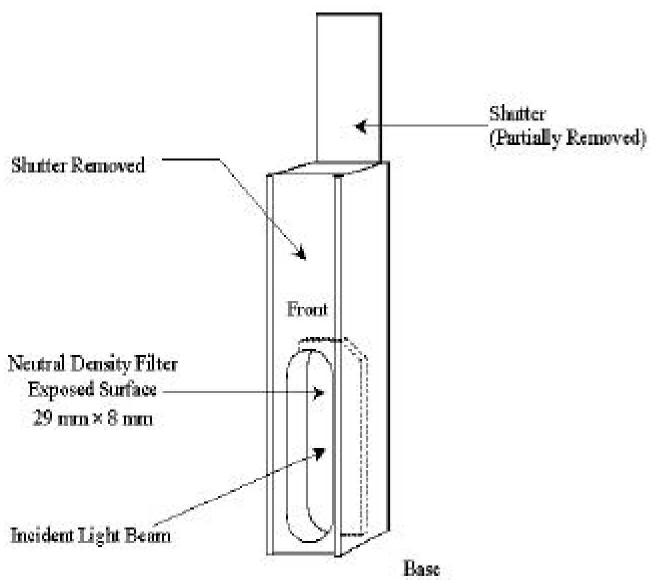

Figure 1. SRM 1930 Filter Holder

Certificate Revision History: 26 May 2000 (Technical changes due to new instrumentation); 17 August 1994 (To conform to NIST new uncertainty policy) ; 27 September 1993 (NBS to NIST revision); 15 March 1987 (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 , Set Number

Wavelength, nm (Maximum Spectral Bandpass, nm)	**Transmittance Density ($-\log_{10}T$) Filter Number - Set Identification**		
440.0 (2.2)	± 0.0055	± 0.0054	± 0.0020
465.0 (2.7)	± 0.0055	± 0.0054	± 0.0020
546.1 (6.5)	± 0.0055	± 0.0054	± 0.0020
590.0 (5.4)	± 0.0055	± 0.0054	± 0.0020
635.0 (6.0)	± 0.0055	± 0.0054	± 0.0020

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

Wavelength, nm (Maximum Spectral Bandpass, nm)	**Transmittance (T) Filter Number - Set Identification**		
440.0 (2.2)	±	±	±
465.0 (2.7)	±	±	±
546.1 (6.5)	±	±	±
590.0 (5.4)	±	±	±
635.0 (6.0)	±	±	±

Date of Recertification:

Table 2. Transmittance Density Uncertainty for SRM

Item	**Transmittance Density Uncertainty**		
	Filter Number		
	1	**3**	**50**
Combined Type A	0.0009	0.0012	0.0002
Combined Type B	0.0026	0.0024	0.0010
Combined Uncertainty (u_c)	0.0028	0.0027	0.0010
Coverage Factor	2	2	2
Expanded Uncertainty (U)	0.0055	0.0054	0.0020

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

Source	**Filter Number**			**DF**
	1	**3**	**50**	
Repeatability	0.0009	0.0012	0.0002	60

Table 4. Type B Components of Transmittance Uncertainty for SRM

Source	**Filter Number**			
	1	**3**	**50**	
Homogeneity	0.0023	0.0020	0.0005	∞
Transmittance Stability	0.0013	0.0013	0.0007	∞
Temperature	0.0005	0.0005	0.0005	∞
Linearity/Geometry	0.0003	0.0003	0.0003	∞
Combined Type B Uncertainty	0.0026	0.0024	0.0010	

Effective Degrees of Freedom > 30