National Institute of Standards & Technology

Certificate

Standard Reference Material 930d

Glass Filters for Spectrophotometry

This Standard Reference Material (SRM) is intended for use in the verification of the transmittance and absorbance scales of spectrophotometers in the visible spectral domain. SRM 930d consists 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 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 a set identification number and a filter number (10, 20, or 30), which corresponds to the nominal % transmittance (100 x transmittance) of the filter.

Certified Transmittance Values: Certified transmittance values independently determined for each filter at five wavelengths in the visible portion of the electromagnetic spectrum are given in Table 1. The relative uncertainty of the certified transmittance values is $\pm$ 0.5% at 21 $\pm$ 2 °C for a period of two years from the date of certification specified below in Table 2. This uncertainty includes the effects of the random and systematic errors of the calibration procedure, as well as estimated systematic errors associated with alignment of the filters and material properties (e.g. aging of the glass which may cause some filters to change transmittance by about + 0.25% over the two-year period). Uncertainty estimation is described in the NIST Special Publication 260-116. [1]

Certified Transmittance Densities: The transmittance densities given in Table 2 are calculated from the certified transmittance (T) as $-\log_{10} T$. These values should be indicated by the absorbance (A) scale of the spectrophotometer if the filters are measured against air. The overall uncertainty in transmittance density is $\pm$ 0.002 absorbance units (AU) for a period of two years from the date of certification below Table 2.

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

The transmittance measurements were performed in the NIST Inorganic Analytical Research Division by J.C. Travis, M.V. Smith, and N.K. Winchester.

The overall direction and coordination of technical measurements leading to certification were performed in the NIST Inorganic Analytical Research Division by J.C. Travis and R.L. Watters, Jr.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
October 18, 1993
(Revision of certificate dated 9-27-93)

Thomas E. Gills, Acting Chief
Standard Reference Materials Program

(over)

NOTICE AND WARNINGS TO USERS

Storage and Handling: SRM 930d is stored in a black anodized aluminum container fitted with a threaded cap made of the same metal. Contamination of the glass filter surface with particulate matter due to static charge is minimized by the metallic nature of the container. 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 which is inserted into the cylindrical cavity to minimize damage during transportation. These springs can be removed during normal use in the laboratory. Improper storage or handling of the filters may cause changes in the transmittance. [1] It is recommended that the filters in the holders be handled only by the edges with soft plastic (polyethylene) gloves and optical lens tissue. When not in use they should be stored in their holders and in the container provided for this purpose. Extended exposure to laboratory atmosphere and dusty surroundings should be avoided. Should the surface of the glass filter become contaminated, no attempt should be made to clean it unless the user has the facilities to demonstrate that the cleaning treatment has not altered the glass surface or compromised the certified values. As this SRM is a transfer standard, the only means available to verify its integrity is by remeasuring its transmittance with a primary standard instrument similar to that used in this certification. [2-4]

Expiration of Certification: This certification is valid for 2 years from the date of certification specified below in Table 2.

Recalibration: The filters should be returned to NIST for cleaning and recalibration at two-year intervals to revalidate the filters. For recalibration, please contact M.V. Smith at (301) 975-4115 or N.K. Winchester at (301) 975-3152. Filters for recalibration should be shipped to M.V. Smith, NIST, Bldg. 222, Rm. B222, Gaithersburg, MD 20899.

Instrument Dependence Warning: Some samples of SRM 930d may cause a small (<0.02 AU) increase in the apparent absorbance, resulting from a minor deviation of the optical axis, in instruments for which wavelength dispersion occurs after the light has passed through the filter. If such effects are detected or suspected, the user should contact J.C. Travis, NIST Inorganic Analytical Research Division at (301) 975-4117, for assistance and instructions.

Source and Preparation of Material: The neutral glasses for the filters were provided by Schott of Mainz, Germany, and are designated as "Jena Color and Filter Glass." Glass material of types NG-4 and NG-5 were selected for best homogeneity and a minimum of inclusions and striae. The filters were cut from plates which were ground and polished in the NIST optical shop to appropriate thicknesses to achieve the nominal transmittances of 0.1, 0.2, and 0.3. [1,5] 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. [1]

Determination of Transmittances: The transmittance measurements were made against air (an empty filter holder) at an ambient temperature of 21.0 ± 1.0 °C using the high-accuracy spectrophotometer designed and built in the NIST Inorganic Analytical Research Division. [2] This instrument represents the primary transmittance standard; its transmittance accuracy was established using the double-aperture method of linearity testing. [1,2,6,7] The effective spectral bandpass used to determine the certified values was 0.8 nm. The transmittance measurements were made by producing the vertical image of the slit (about 8 mm by 1 mm), using a convergent beam geometry with an aperture ratio f:10, in the middle of the entrance face of the glass filter. The filter is mounted in a multiple-filter carousel in the spectrophotometer. Each transmittance value reported in Table 1 is the average of three transmittances determined over an eight-min period required for three carousel rotations. The transmittance is measured in this way several times during an aging period of at least six months, and only the final measurement is reported. Each transmittance measurement 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 and a signal integrating time of approximately 1 s for each measurement. The filters were measured in the spectrophotometer in a position perpendicular to the incident light beam as shown in figure 1.

Uniformity: The transmittance uniformity of each filter comprising SRM 930d was established over an area 5 mm wide by 24 mm long and located symmetrically about the center face of each filter. The transmittance was required to vary by less than the estimated systematic error component for uniformity of 0.3%, relative, over the specified area.

Instructions for Use: The transmittance of the filters depends upon the intrinsic properties of the material, wavelength, spectral bandpass, geometry of the optical beam, and can be affected by other factors such as stray light, temperature, and positioning of the filter. A change of ambient temperature of $\pm$ 2 °C from 21.0 °C will not significantly affect the calibration. [1] 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 under "Storage and Handling". [1,3,5,6] The spectral bandpass values indicated in parentheses in this certificate are maximum values that should not be exceeded when accurate measurements are contemplated. The empty filter-holder is provided 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, and the optical geometry described under "Determination of Transmittances", will produce transmittance values that might differ from the certified data.

The cooperation of G.N. Bowers, Jr., M.D., of Hartford Hospital, Hartford, CT; R.N. Rand, Ph.D., of the Eastman Kodak Co. Research Laboratories, Rochester, NY; D.S. Young, M.D. and Ph.D., of the Mayo Clinic, Rochester, MN; and B. Mueller, Ph.D, of Hewlett-Packard GmbH, Waldbronn, Germany are gratefully acknowledged.

REFERENCES

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[3] Collected Papers from NBS Conference on Accuracy in Spectrophotometry and Luminescence Measurements, NBS Journal of Research 76A, No. 5, pp. 375-510, (1972).

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

[5] Mavrodineanu, R., Solid Materials to Check the Photometric Scale of Spectrophotometers, NBS Tech. Note 544, Menis, O., and Shultz, J.I., eds., pp. 6-17, (Sept. 1970); ibid, NBS Tech. Note 584, pp. 2-21, (December 1971).

[6] Gibson, K.S., Spectrophotometry, NBS Circ. 484, (Sept. 1949).

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

Figure 1. Metal Holder for the Colored Glass Filters