National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2055

Infrared Transmittance Standard

Sample

This Standard Reference Material (SRM) is intended for use in checking the accuracy of the transmittance (ordinate) scale of spectrophotometers in the infrared (IR) spectral region from 2 µm to 25 µm (400 cm^{-1} to 5000 cm^{-1}). SRM 2055 is a neutral-density transmitting filter with an optical density (OD) near 3.0 (OD = $-\log_{10}$(T), where T is the spectral transmittance). It consists of an approximately 77 nm thick copper-nickel film on a 250 µm thick, 25 mm diameter, high-resistivity, single-crystal <100> silicon substrate. The metal film is overcoated with a thin (30 nm) layer of silicon oxide to protect it against oxidation from the atmosphere. SRM 2055 also includes a CD that contains the certified transmittance values and associated expanded uncertainties.

Certified Transmittance Values: The transmittance of this filter was measured at equally spaced wavenumber values from 400 cm^{-1} to 5000 cm^{-1} (2 µm to 25 µm) with an apodized resolution of 8 cm^{-1} and a data spacing interval of approximately 4 cm^{-1}. The transmittance was measured at the center of the filter using a 6 mm diameter spot size with the filter at the focus of an f/3 optical beam in the spectrophotometer. The certified transmittance values and associated expanded uncertainties at each wavelength are listed in a text file on the accompanying CD and plotted in Figure 1. These values and their associated uncertainties are valid for normal incidence transmittance within a 5 mm radius of the center of the filter and at temperatures from 20 °C to 30 °C.

Expiration of Certification: The certification of this SRM is deemed to be valid until **31 December 2010**, provided the SRM is stored and handled in accordance with the *Storage and Handling* section of this certificate. Certification will be nullified if the SRM is damaged, contaminated, or exposed to excessive temperatures or humidity.

Maintenance of SRM Certification: The neutral-density filter has been measured over a period of several years, and no significant change in transmittance has been noted. A reference filter is kept at NIST and will be monitored over time. Users will be notified if a significant change occurs before the stated expiration date. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of the technical measurements were performed by R.U. Datla of the NIST Optical Technology Division.

The technical measurements leading to certification were performed by S.G. Kaplan and L.M. Hanssen of the NIST Optical Technology Division.

Statistical consultation was provided by M.C. Croarkin of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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Optical Technology Division

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Gaithersburg, MD 20899
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Source of Material: The silicon substrates used in making the filters were obtained from Virginia Semiconductor Corporation[1] of Fredericksburg, VA. The metal films were deposited on the substrates by Luxel Corporation[1] of Friday Harbor, WA.

CERTIFICATION ANALYSIS

Measurement Conditions: The calibration measurements were made using a Bio-Rad FTS-60A[1] Fourier transform infrared (FT-IR) spectrophotometer. The temperature in the filter compartment of the instrument was approximately 29 °C, and the spectrophotometer was purged with dry, carbon dioxide free air. A neutral-density filter with optical density near 1 (SRM 2053) was used as the reference in measuring the transmittance of this SRM. The transmittance of the filter under test was calculated from

$$T_{filter} = T_{relative} \times T_{reference} \qquad (1)$$

Details of the measurement and analysis can be found in Reference [1].

Determination of Uncertainty in Transmittance: The combined uncertainty includes "Type A" uncertainties, which are evaluated by statistical methods, and "Type B" uncertainties, which are determined by other means. Type A uncertainties include the reproducibility, listed in Table l, and the repeatability, listed on the accompanying CD. Each filter was measured three times in one day, then removed from the spectrophotometer. Each of the 10 nominally identical filters in this batch was again measured twice, approximately six months after the first set of measurements. Each set of measurements was evaluated for repeatability variance, s_r^2. The day-to-day variance was also computed for each filter, and no evidence of long-term drift in the measurements was found. The maximum daily variance of all the filter measurements was used as the value of reproducibility variance, s_{days}^2, which was averaged over several different wavelength bands. Values for a number of systematic (Type B) sources of uncertainty were also estimated (Table 1) and added in quadrature to produce a variance component s_B^2. The variance in transmittance of the OD 1 reference is $s_{reference}^2$. The combined uncertainty for the filter was calculated from

$$\begin{aligned} s_{meas}^2 &= \frac{1}{3} s_r^2 + s_{days}^2 + s_B^2 \\ s_{filter}^2 &= \left(\frac{s_{meas}^2}{T_{filter}^2} + \frac{s_{reference}^2}{T_{reference}^2}\right) \times T_{filter}^2 \end{aligned} \qquad (2)$$

The expanded uncertainty (coverage factor of 2) is $2s_{filter}$. Representative curves for the certified transmittance values and associated expanded uncertainties in transmittance are shown in Figures 1 (a) and (b). The certified values and uncertainties are listed in the data file on the accompanying CD.

INSTRUCTIONS FOR USE

Storage and Handling: The SRM is quite fragile and should be handled with care when mounting and dismounting it from the measurement apparatus. The filter may be handled by wearing clean plastic or thin rubber gloves and holding it gently by the edge. The surfaces should never be touched. Dust can be removed by gently blowing the surface with clean, dry air. When not in use, SRM 2055 should always be kept in its accompanying protective box, which has the SRM serial number engraved on the bottom. It is recommended that the SRM be stored in a dessicator cabinet, if available.

Test Measurements: The spectrophotometer system should be set up under the following conditions. The spectral resolution should ideally be 8 cm^{-1} in order to match that used in the SRM calibration measurements. Lower resolution, (i.e. larger cm^{-1} value), may be used and the certified values averaged to match the resolution of the measurement. At resolutions higher than 8 cm^{-1}, Fabry-Perot fringes will appear in the spectrum due to interference in the silicon substrate. In addition, the depth and width of the prominent silicon absorption features near 9 µm and 16 µm wavelengths, and weaker structures at intervening wavelengths, will be affected by choice of resolution. It is recommended that transmittance levels away from these features be used to compare the user's data with the SRM calibration data if the user's resolution is different from 8 cm^{-1}.

[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order 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 this purpose.

The IR beam parallelism may range from collimated up to f/3 focusing geometry. The beam should be centered on the filter with a spot size less than 10 mm in diameter. For most FT-IR spectrophotometers, a spot size in the 1 mm to 5 mm diameter range will be most convenient. The average angle of incidence at the filter surface should be less than 6°. The filter should be reproducibly moved in and out of the beam path and the spectra, with and without the filter in place, ratioed to produce a transmittance spectrum. The measurements should be repeated several times in order to assess their reproducibility.

Steps should be taken to reduce the effects of detector nonlinearity, ambient thermal emission, and inter-reflections involving the filter, spectrophotometer, and detector. The magnitude of each of these effects will depend on the user's system and may be reduced by using additional filters and/or stops in the beam path, tilting the filter slightly or blocking half of the beam, and performing additional measurements to test for the effects of ambient thermal emission not coming from the source. A measurement should be made with the beam path at the filter position blocked with an opaque object in order to test for stray light or electronic offset which could produce a false transmittance signal. If a significant signal is found, it should be subtracted from both the filter and reference measurements before ratioing them to obtain the filter transmittance.

Comparison with Certified Values: The primary purpose of this SRM is to check the accuracy of the transmittance (ordinate) scale of the user's spectrophotometer system. In conjunction with SRMs 2053 and 2054, transmittance measurements of this filter can be used to assess the linearity of the instrument response scale. The user's transmittance spectrum should be compared with the certified values at the wavelength(s) of interest. If the user's values differ from the certified values by less than the quadrature sum of the certified expanded uncertainties and the expanded uncertainty of the mean of the user's values, then the user's values are accurate to this level and no correction should be attempted on the basis of comparison with this SRM.

If the user's measured values differ significantly from the certified values, then additional steps along the lines suggested in the previous section may be taken in order to attempt to improve the transmittance (ordinate) scale accuracy of the instrument. Because of the wide variety of possible spectrophotometer systems and the number and complexity of the various sources of error that may be present in different systems, it is not possible to give a general correction algorithm that will work for all systems. The user of FT-IR systems is referred to the literature on the accuracy of measurements with these instruments [3]. In particular, it should be noted that different types of filters will introduce very different sources of error into the measurements. This SRM is highly reflective and has a transmittance significantly less than 1, so it will tend to reveal errors due to detector nonlinearity and inter-reflections. However, it is optically quite thin and will not yield much difference in the beam geometry, with and without the filter in place, which can be a significant source of error for thicker filters.

REFERENCES

[1] Kaplan, S.G.; Hanssen, L.M.; *Standard Reference Materials: Infrared Transmittance Standards – SRMs 2053, 2054, 2055, and 2056*; NIST Special Publication 260-123, U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

[2] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed. ISO; Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

[3] Kaplan, S.G.; Hanssen, L.M.; Datla, R.U.; *Testing the Radiometric Accuracy of Fourier Transform Infrared Transmittance Measurements*; Appl. Opt. Vol. 36, p. 8896 (1997) and references therein.

Certificate Revision History: 08 March 2006 (Extension of certification period and editorial changes); 25 August 2002 (Extension of certification period and editorial changes); 09 August 2000 (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 at http://www.nist.gov/srm.

Table 1. Standard uncertainty components, in percent of measured transmittance value, for systematic uncertainty sources as well as reproducibility in the measured values.

Uncertainty source	Value (%)
Type B	
Inter-reflections	0.30
Detector nonlinearity	0.05
Detector nonequivalence	0.10
Non-source emission	0.15
Beam nonuniformity	0.20
Beam displacement, deviation, focus shift	0.10
Beam geometry, polarization	0.20
Filter vignetting	0.03
Filter scattering	0.02
Phase errors	0.10
Filter nonuniformity	2.89
Filter temperature	0.20
Quadrature sum, s_B	2.93
Type A (Reproducibility, s_{days})	
Daily variation 2 µm to 4 µm	2.70
Daily variation 4 µm to 12 µm	2.20
Daily variation 12 µm to 20 µm	3.00
Daily variation 20 µm to 25 µm	3.50

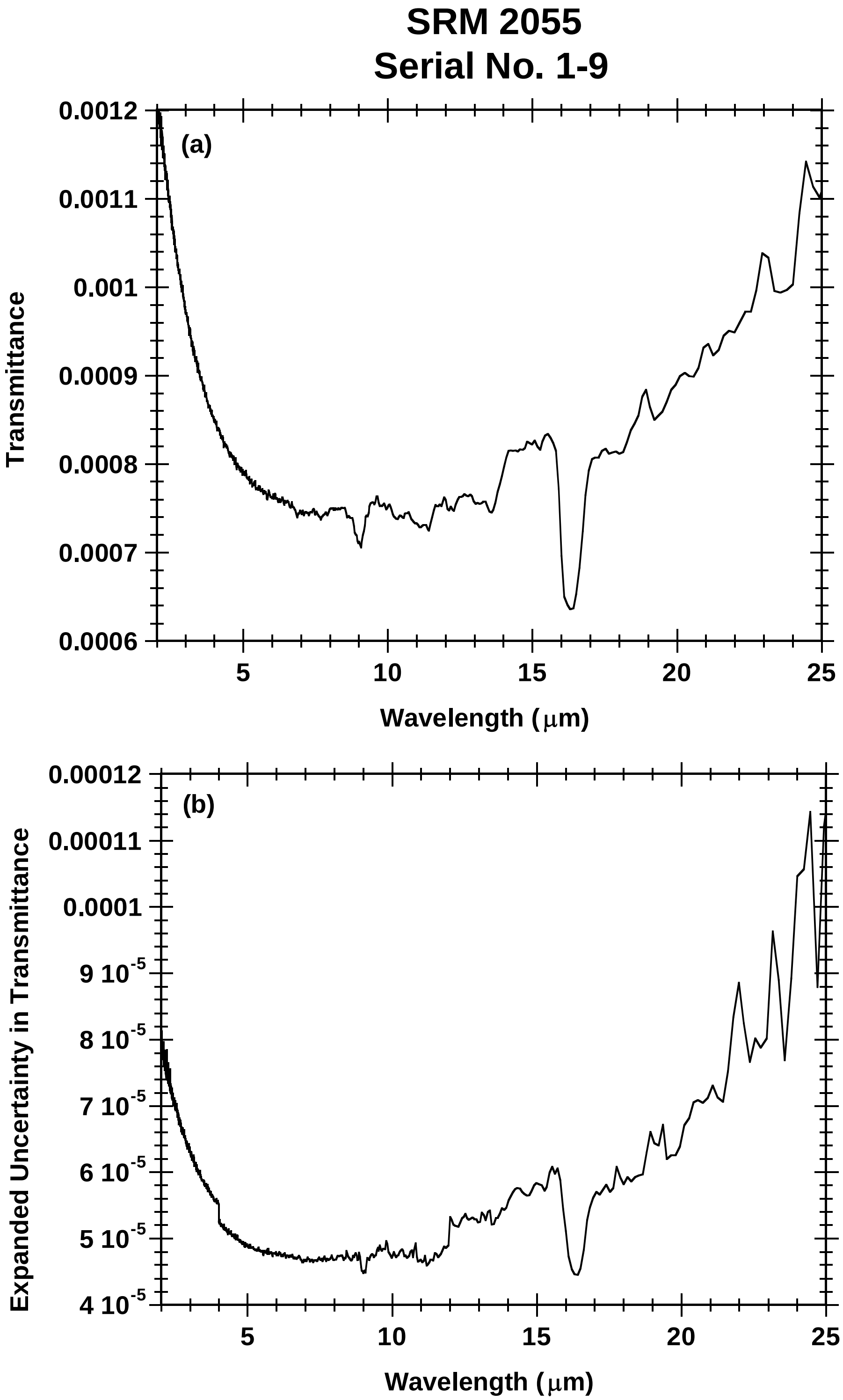

Figure 1. (a) Certified transmittance versus wavelength, and (b) expanded uncertainty in transmittance of the neutral-density filter. The graphs should not be scaled to produce data for the filter. Data should be taken from the file on the accompanying CD.