# Appendix B: Facility Descriptions

*CENAM's Measurement Technique:*
The measurement sequence is detailed in Table B1. Measurements at 900 nm and 1000 nm were not reported because of the noise of our lead sulfide photodiode.

Figure B1. Schematic diagram of CENAM's spectrophotometer.

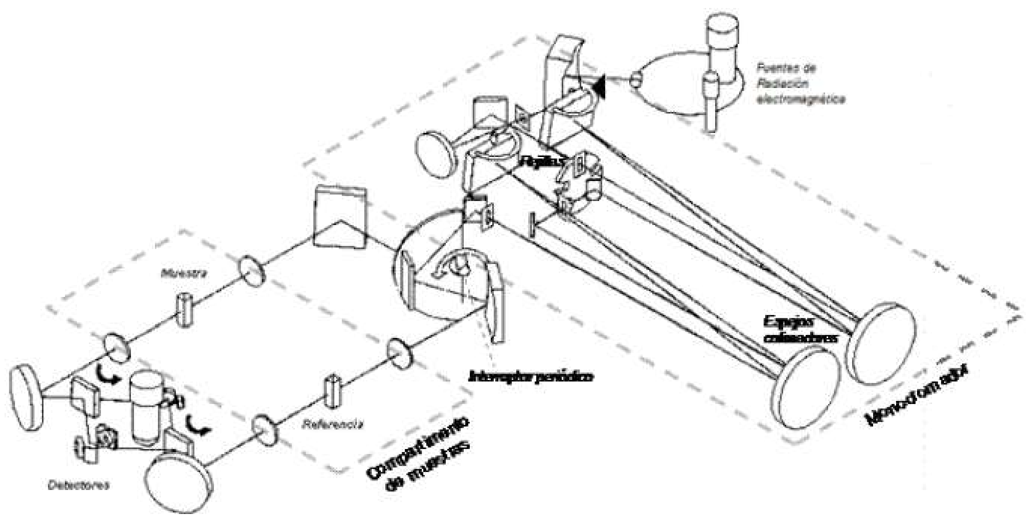



Table B1. Details of CENAM's Measurement Setup

| | |
|---|---|
| Make and Type of Spectrophotometer | Spectrophotometer UV-VIS-NIR, Varian Cary 5e |
| Additional Stray Light Rejection | ASTM E 387, The Opaque filter method |
| Source Drift Monitoring | Signal Stability by 10 minutes |
| Source | Tungsten Halogen Lamp |
| Detector | R928 PMT |
| Temperature | Place near to spectrophotometer (23.2 °C; max. variation: 0.3 °C) |
| Humidity | Place near to spectrophotometer (RH 48.7 %; max. variation: 8.7 %) |
| Beam Size | 1 mm x 10 mm (W x H) |
| Beam Collimation | Unchanged, Factory default |
| Measurement Sequence | For each filter: 4 measurements, Filter rotation of 90 degrees, sense clockwise; three times |
| Bandwidth | 1 nm |

*INM's Measurement Technique:*
The transmittance measurements were performed on a Cary 4000 (Agilent Technlogies) double bean spectrophotometer, with suitable photometric performance in the 175 nm to 900 nm range.

At each wavelength the following measurements were performed:

1. Set the zero of the measurement
2. Measurement of the transmittance at 100% (clear beam) ($\tau_z$)
3. Blocking the sample beam and measurement of the transmittance ($\tau_0$)
4. Measurement of the filter ($\tau_m$)
5. Blocking the sample beam and measurement of the transmittance ($\tau_0$)
6. Measurement of the transmittance al 100% (clear beam) ($\tau_z$)
7. The spectral transmittance was obtained from:

$$\tau = \frac{\tau_m - \tau_0}{\tau_z - \tau_0}$$

Figure B2. Schematic diagram of INM's spectrophotometer. (Adapted from: Fundamentos de la espectroscopía UV- Visible moderna. Conceptos básicos AGILENT TECHNOLOGIES.)

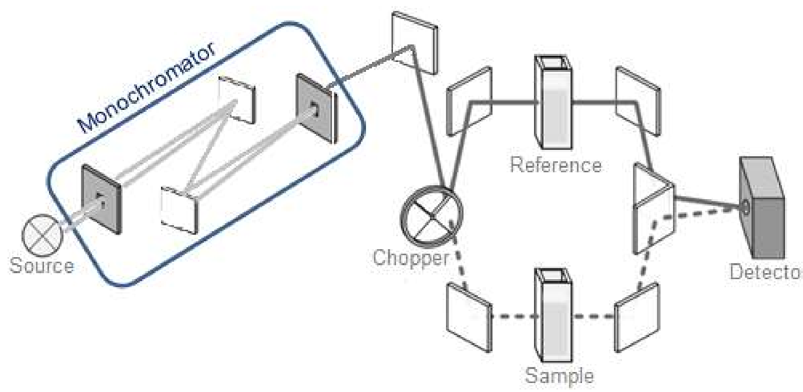


Table B2. Details of INM's Measurement Setup

| | |
|---|---|
| Make and Type of Spectrophotometer | The measurements were done with the reference spectrophotometer Agilent Cary 4000 UV-Vis. This spectrophotometer has a double beam, a double out of plane Littrow monochromator, a 2 mm by 400 mm focal length and two gratings with 1200 lines/mm. The detector is a R928 photomultiplier tube. The instrument has three sources, a tungsten halogen visible source with quartz window, a deuterium arc UV source, and a mercury lamp used for wavelength accuracy validation. The spectrophotometer has a beam splitter, with a speed of 300 Hz. The wavelength range is 175 nm to 900 nm. Variable spectral bandwidth between 0.01 nm to 5.00 nm with 0.01 nm steps. |
| Additional Stray Light Rejection | At 220 nm, the spectrophotometer has a stray light < 0.00007%. At 370 nm. it has a stray light < 0.00007%. |
| Source Drift Monitoring | In order to monitor the drift, we did three measurements per point: clear beam, blocked beam, and the filter, and we corrected each transmittance (see Description of measuring technique) |
| Source | All measurements were performed with the tungsten halogen visible source. |
| Detector | R928 photomultiplier tube. |
| Temperature | The measurements were done between 22.5 °C and 23.2 °C. |
| Humidity | The relative humidity during the measurements was within 39.6 % RH and 52.4% RH. |
| Beam Size | Slit at full height (5 nm SBW): 13.35 mm high by 5.11 mm wide<br>Slit at reduced height (5 nm SBW): 9.08 mm high by 5.1 mm wide |
| Beam Collimation | The spectrophotometer has 2 collimating mirrors, one in each monochromator. |
| Measurement Sequence | For each filter, we performed the following sequence:<br>1. We removed dust with dry nitrogen.<br>2. The filter was placed in the sample holder with its identification number in front of the incident beam and upright with a normal angle of incident.<br>3. The measurements were done at 380 nm, 400 nm, 500 nm, 600 nm, 700 nm, 800 nm, and 900 nm.<br>4. The filter was measured 30 times.<br>5. After these measurements, the filter was rotated 90° and measured 30 times more.<br>Each filter was measured in two different days. The regular transmittance reported is the mean of all the measurements. The filters were measured in the following order A, E, D, C, B. |
| Bandwidth | The measurements were done with a bandwidth of 1 nm. |

*INMETRO's Measurement Technique:*
Inmetro transmittance measurement system (TMS) is designed for the measurement of absolute regular spectral transmittance above 0.1 in the wavelength range from 400 nm to 1000 nm. The system consists of a 24 V/250 W quartz-tungsten-halogen lamp, a Czerny-Turner monochromator (Jobin Yvon HR 250 M), a light-tight box with the sample and a silicon photodiode (Hamamatsu S1227), as shown in Figure B3.

The quartz-tungsten-halogen lamp mounted inside the housing and operated in current-mode (10 A) is focused onto the entrance slit of the monochromator. A cut-off filter (Schott OG 550) is placed in front of the entrance slit to reject higher order wavelengths in measures above 590 nm. The light exiting the monochromator is collimated by a concave mirror and directed onto an iris that adjusts the beam diameter to about 17 mm. After the sample, the beam is focused by a concave mirror on the surface of the detector. A translation stage is used to interchange the sample (filter) and the reference (empty) in the beam path. The photocurrent is amplified by a transimpedance amplifier (LabKinetics SP042) and read with a digital voltmeter (Agilent 34420A).

Figure B3. Schematic diagram of INMETRO's transmittance measurement system.

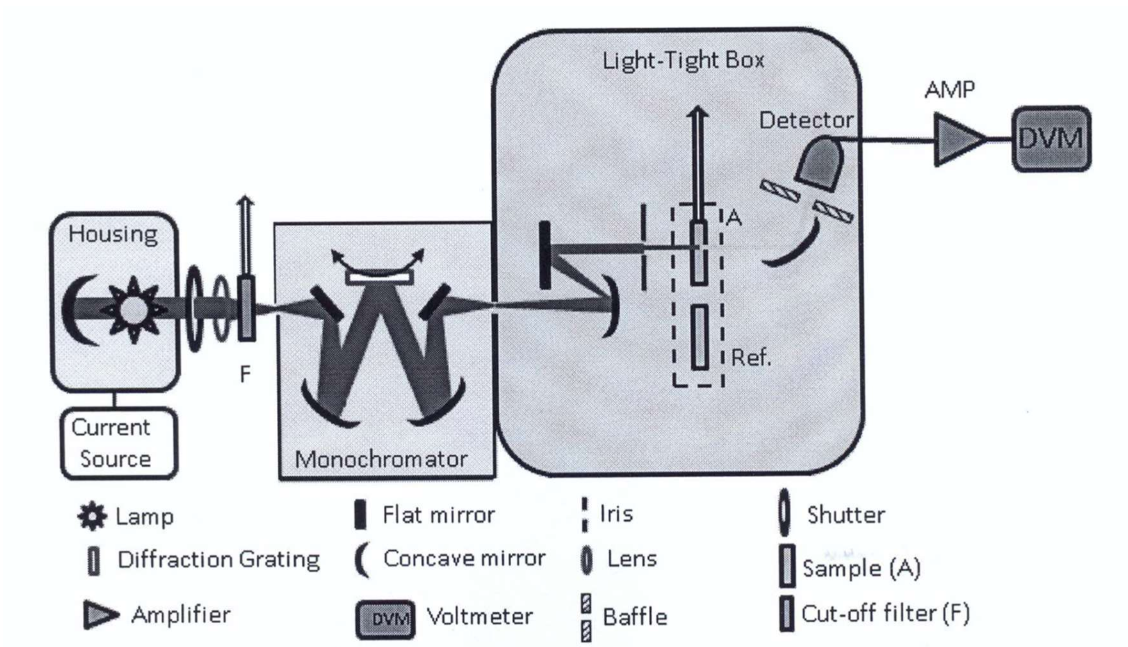


Table B3. Details of INMETRO's Measurement Setup

| | |
|---|---|
| Make and Type of Spectrophotometer | Custom-made spectrophotometer based on a single beam Czerny-Turner monochromator (Jobin Yvon HR 250 M). |
| Additional Stray Light Rejection | Cut-off filter in front of the entrance to the monochromator to minimize high order in measurements above 590 nm. Sample and detector are placed in a light-tight box. |
| Source Drift Monitoring | Source drift is corrected along measurement by measuring the reference signal prior and after the measurement with the filters. |
| Source | 24 V/250 W quartz-tungsten-halogen lamp |
| Detector | Silicon photodiode (Hamamatsu S1227) |
| Temperature | (24.5 ± 0.4) °C measured inside the light-tight box with a thermo-hygrometer traced to national metrology standards. |
| Humidity | < 60.0 % measured inside the light-tight box with a thermos-hygrometer traced to national metrology standards. |
| Beam Size | 17 mm in diameter, rounded beam |
| Beam Collimation | Beam divergence < 0.7° at the sample |
| Measurement Sequence | Measurements were taken for each filter following the sequence: 0 % T (blocked beam), 100 % T (no filter), filter positioned 0°, and filter positioned 90°. At each wavelength measurement follows the sequence: reference (no filter), with filter, reference. |
| Bandwidth | Approximately 1 nm |

*CMS/ITRI's Measurement Technique:*
CMS uses a double beam spectrometer to measure the transmittance of samples. The light passes through the filters vertically. The transmittance of the filters is calculated with respect to the reference beam and is corrected by separate baseline measurements.

The baseline measurements consist of 100% and 0% transmittance measurements. For 100% transmittance measurement, the sample beam passes through an empty sample holder. For the 0% transmittance measurement, the sample beam is blocked by an opaque sample.

To increase the sensitivity of low transmittance samples, the reference beam was attenuated. Specifically, the C filter was used to attenuate the reference beam when measuring the D filter, and the D filter was used to attenuate the reference beam when measuring the E filters. No filters were used in the reference beam during measurement of the A, B, and C filters.

Figure B5. Schematic diagram of the CMS/ITRI's spectrophotometer.

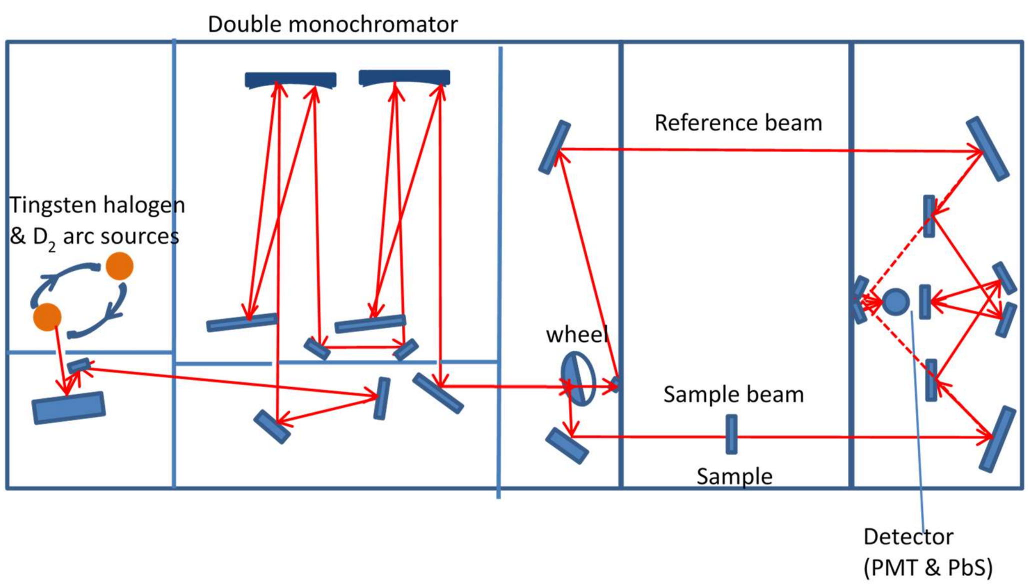


Table B4. Details of CMS/ITRI's Measurement Setup

| | |
|---|---|
| Make and Type of Spectrophotometer | Agilent/Cary 5000: A UV-Vis-NIR spectrophotometer with photometric performance in the 200 nm to 3000 nm range. |
| Additional Stray Light Rejection | The signal of the solution of NaI at 220 nm and of $K_2Cr_2O_7$ at 370 nm was measured to determine the stray light effect. Stray light contribution was added to measurement uncertainty. |
| Source Drift Monitoring | A double-beam spectrophotometer was used. The reference and sample beam are continually compared to produce final measurement results, and the chopper cycle is 0.033 s. |
| Source | QTH lamp |
| Detector | The detector was PMT (Hamamatsu/R928) for the measurement below 800 nm, and cooled PbS for the measurement at 900 nm and 1000 nm |
| Temperature | (23.0 ± 1.5) °C |
| Humidity | (45 ± 10) % |
| Beam Size | An around 13 mm by 1 mm rectangular area |
| Beam Collimation | Maximum half angle of cone in the incident angle beam: 3.8° |
| Measurement Sequence | Each filter has been measured within three days and been repeated three times in each day. |
| Bandwidth | 1 nm |

*NIM's Measurement Technique:*
The measurements were conducted using a spectrophotometer based on a single beam, single monochromator (Type 2061, McPherson) with 2400 g/mm diffraction gratings. A tungsten halogen lamp (Type Osram 300 W) was used as the light source for the measurements over the whole spectral range. A series of band-pass filters were placed at the exit slit of the monochromator. A small portion of the light from the band-pass filters was directed onto a monitor detector (silicon detector, Type S1337-1010BQ, Hamamatsu) using a beam splitter. A silicon detector of the same type was used as the main detector. The monitor detector was used to minimize the impact from the drift of the source. The photocurrents of both detectors were measured using two Keithley 6485 Picoammeters synchronously. The filter was mounted with a sample holder on a translation and rotary stage.

Figure B6. Schematic diagram of the spectrophotometer at NIM.

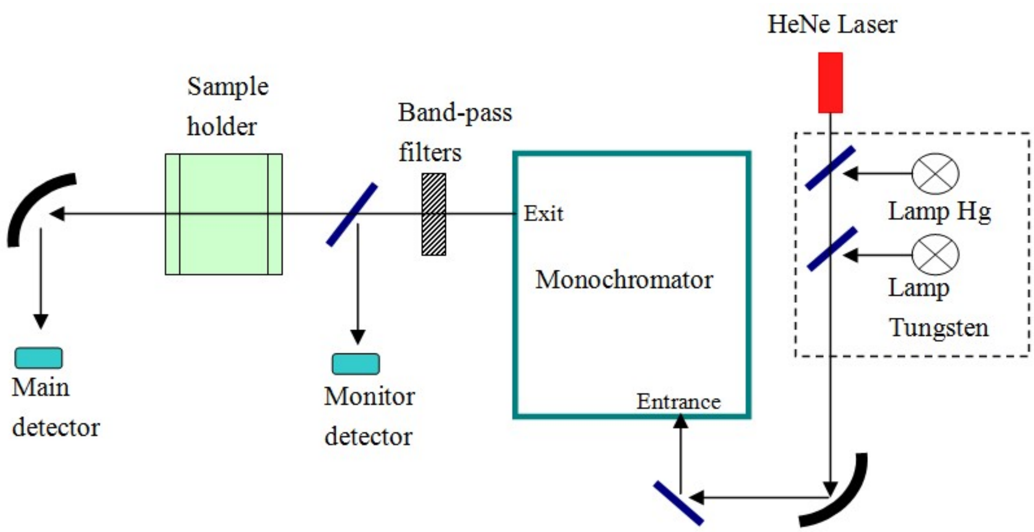


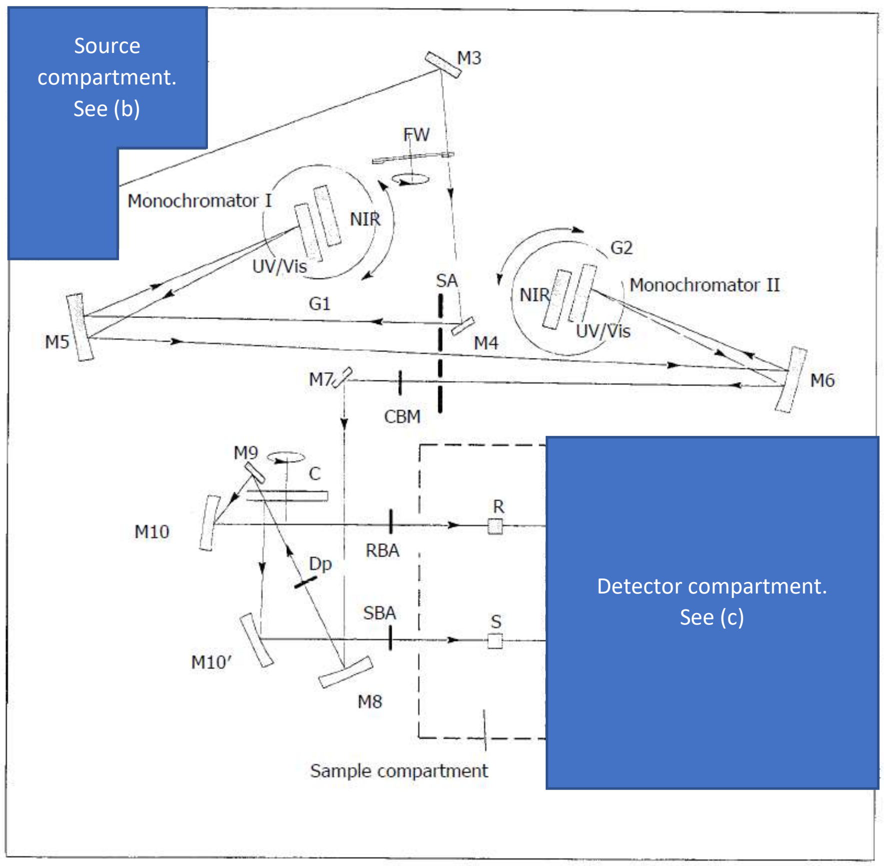
Table B5.  Details of NIM's Measurement Setup

| | |
|---|---|
| Make and Type of Spectrophotometer | Made by NIM |
| Additional Stray Light Rejection | Stray Light Filter |
| Source Drift Monitoring | Yes |
| Source | QTH Lamp, 300 W |
| Detector | Si, 1337 |
| Temperature | (23 ± 1) ºC  (4 Wire, PT100) |
| Humidity | < 40 % RH |
| Beam Size | Φ17 mm |
| Beam Collimation | 0.3º |
| Measurement Sequence | 100 %, Sample, Zero (Six times, no changes between measurements) |
| Bandwidth | 1 nm |

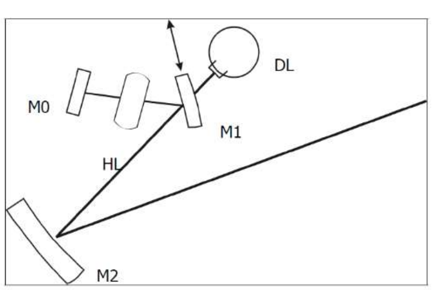
*NIMT's Measurement Technique:*
Even though the spectrophotometer used for the measurement is a double-beam spectrophotometer, the single beam method is adopted. In each series (round), the signal (which is the ratio of the sample beam to the reference beam) is measured in the series: dark, sample, blank, sample. The transmittance is calculated for each round of measurement by dividing the average dark corrected signal of the sample by the dark corrected signal of the blank. The diagrams of the spectrophotometer used are as followed:

Figure B7. (a) Schematic diagram of NIMT's spectrophotometer. (b) Source compartment showing the source doubling mirror M0. (c) Detector module with 3 detectors.

(a)




(b)




(c)

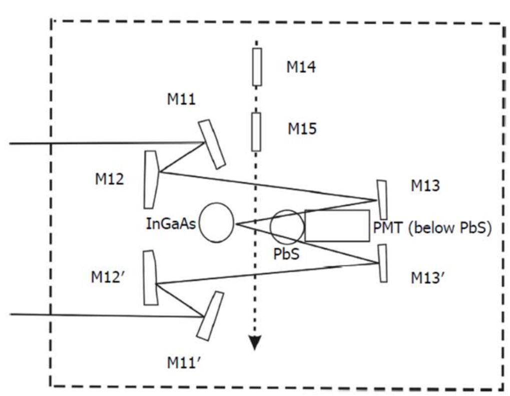


Table B6. Details of NIMT's Measurement Setup

| | |
|---|---|
| Make and Type of Spectrophotometer | Perkin Elmer Lambda 1050 |
| Additional Stray Light Rejection | $10^{-8}$ |
| Source Drift Monitoring | Yes, using the ratio of the sample beam to the reference beam. |
| Source | 100 W Tungsten Halogen Lamp 1 |
| Detector | PMT |
| Temperature[(a)] | The temperature on the filter was not monitored. Only the ambient temperature of the room (near the spectrophotometer) was controlled and monitored to be within (23 ± 2) °C. |
| Humidity | (50±15) % RH |
| Beam Size | Approx. 1 mm width by 13 mm height |
| Beam Collimation | No, the beam is a focusing one |
| Measurement Sequence[(b)] | Measurement were performed in total 12 rounds. In each round, the signals were measured in the sequence, dark→Sample→Blank (Ref)→Sample. Eight rounds were measured with the mark on the filter facing the beam while the other four rounds were measured with the other side of the filter facing the beam. For the first eight rounds, the filter was oriented in four different directions, north, south, east, and west with respect to the beam, two rounds for each orientation. Similar orientations were applied for the other four rounds. |
| Bandwidth | 1 nm |

*NRC's Measurement Technique:*
The filters were mounted in an automated 6-position filter wheel sample holder. A precision aperture was mounted in front of the filter wheel to limit the beam size to 17 mm diameter at the sample position, as specified in the technical protocol. In general, three filters were measured in any given measurement cycle at a given set of experimental conditions, with 2 open beam positions for the 100 % reference readings bracketing each filter reading. A dark signal (0 % reading) with the shutter closed was also recorded before each reference reading and the mean dark signal for each measurement cycle was subtracted from each raw signal measurement. For the higher density filters, D and E, a reference beam attenuation technique was used to reduce the uncertainty measurement. When using the reference beam attenuation technique, the automated measurement sequence was modified so that the measurement of the attenuating filter (and dark signal) bracketed the measurement of the filter under test in a time-symmetrical sequence and the filter wheel was rotated first in a clockwise and then a counter-clockwise direction. The measured signal for the filter under test was then referenced to the average of the two time-bracketing measurements of the attenuating filter, after each reading had been corrected for the mean dark signal. The transmittance of the filter under test was then calculated by multiplying this apparent transmittance by the known transmittance of the attenuating filter calibrated on the Reference Spectrophotometer under the same measurement conditions.

For the SIM.PR-K6 measurements, the filters D and E were measured at all wavelengths from 380 nm to 800 nm using a reference beam attenuation technique. Filter D was measured against filter C and Filter E was measured against filter D. For the wavelengths of 900 nm and 1000 nm, these two filters were measured relative to an open beam position.

The Type A uncertainty due to long-term measurement reproducibility and influence of sample non-uniformity were assessed from the experimental standard deviation of the mean of a minimum of 3 independent runs recorded on different days over a two month period and for which the filter was repositioned and/or the detection system was changed. This uncertainty component includes the influence of short term repeatability, where the result of one measurement run was typically obtained from the mean of 8 repeat measurement cycles recorded over a total elapsed time of 20 minutes.

All five filters were calibrated at the eight specified wavelengths from 380 nm to 1000 nm, with a bandwidth of (1.0 ± 0.03) nm. The measurements were performed using a 200 W tungsten-halogen lamp, and a minimum of two different types of the detectors identified above. For the wavelengths of 900 nm and 1000 nm, only the two different types of silicon photodiode detectors were used, whereas for the intermediate wavelengths of 400 nm to 700 nm, all 4 different types of detectors were used (two PMTs and the two Si detectors). For the wavelength of 380 nm, only the two PMTs were used for the highest density filter E.

The relative humidity during the measurements varied from a minimum of about 25 % RH to a maximum of about 45 % RH. The NRC Reference Spectrophotometer has a calibrated RTD element installed in the sample compartment. The temperature of the sample compartment was recorded with a calibrated digital thermometer Fluke Model 1529-R (uncertainty is ± 0.0025 °C at 25 °C) during the filter measurements and was used to correct the transmittance results with the relative temperature coefficients, $\kappa$, and the equation provide in the technical protocol. The laboratory is equipped with an electronic air cleaner and a positive air flow system. Prior to each measurement run, a jet of purified nitrogen gas was used to blow any dust off the surfaces of the filters. No other cleaning of the filters was carried out.

The NRC Reference spectrophotometer was used for this comparison. It is a single-beam instrument with a highly-collimated beam design using all-reflective optics and a prism-grating monochromator in conjunction with a deuterium and tungsten-halogen source to cover the spectral range 200 nm to 2500 nm. The detectors that have been used for this key comparison over the spectral range 380 nm to 1000 nm are two different side-on PMTs: a Hamamatsu R6872 and Hamamatsu R928; and two different types of silicon photodiode detectors: a custom-designed silicon sphere detector comprising two Hamamatsu S1337 photodiodes mounted in a sintered PTFE integrating sphere; and a large area silicon photodiode (LASD) detector, a Hamamatsu S6337. To minimize inter-reflection errors and improve spatial uniformity of response, the Hamamatsu R6872 and R928 PMTs are used behind a ground quartz diffuser and the Hamamatsu S6337 photodiode is slightly tilted. The PMTs are thermoelectrically cooled to (-13 ±1) °C to increase measurement sensitivity and to lower dark current signal. The linearity of the PMTs and the silicon photodiode detection systems has been tested over more than 3 decades using the NRC automated high-precision variable aperture device.

Figure B4. Schematic diagram of the NRC Reference Spectrophotometer.

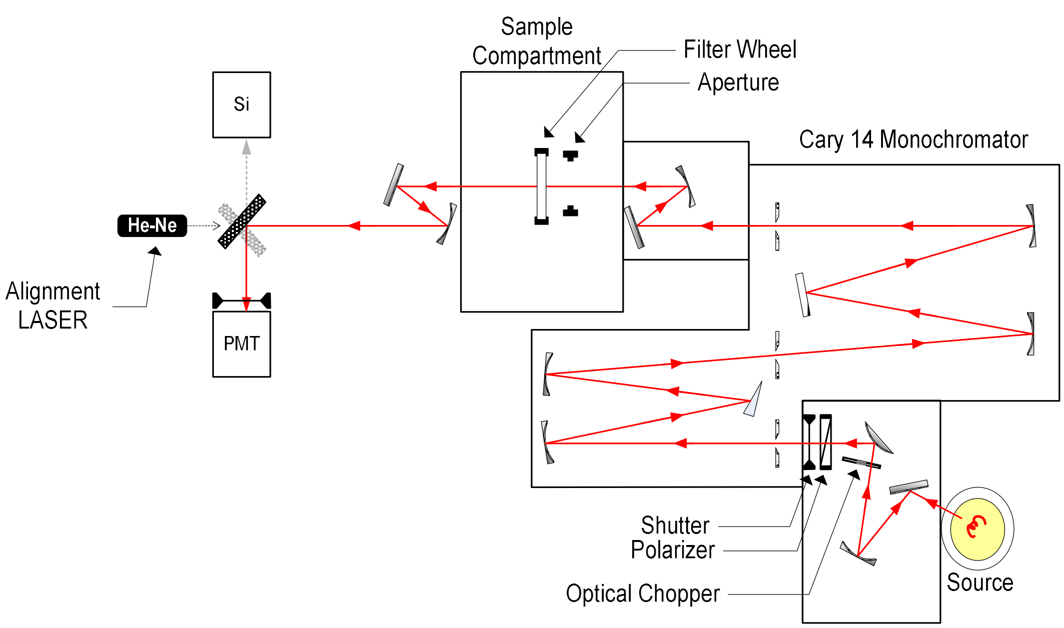


Table B7. Details of NRC's Measurement Setup

| | |
|---|---|
| Make and Type of Spectrophotometer | Custom-built (J.C. Zwinkels and D.S. Gignac, "Design and testing of a new high-accuracy ultraviolet-visible-near-infrared spectrophotometer" *Appl. Opt.*, **31**, 1557-1567 (1992)) |
| Additional Stray Light Rejection | Fused-silica prism predisperser, minimum monochromator slit height of 7 mm, and instrument operated in fully darkened room. |
| Source Drift Monitoring | Automated time-symmetrical measurements of sample and reference (either open beam or calibrated attenuator filter). |
| Source | 200 W tungsten-halogen lamp (Q6.6A/T4/1CL) in a custom-designed and built chimney-type lamp housing. |
| Detector | Custom-designed and built silicon sphere detector (2x HMT 1337), large-area silicon detector (HMT S6337), two different thermoelectrically-cooled (-13° C) PMTs used behind a Suprasil quartz diffuser (HMT R928, HMT R6872). |
| Temperature | Monitoring with a calibrated digital thermometer (Fluke Model 1525-R) mounted in the sample compartment; range of measured temperatures: 22.91° C to 23.67° C, corrected to 23° C using temperature coefficients for the filters provided by pilot lab. |
| Humidity | 25 % RH to 45 % RH |
| Beam Size | 17 mm diameter (using a precision diaphragm mounted in front of the sample wheel) |
| Beam Collimation | Maximum degree of convergence is 0.7°. |
| Measurement Sequence | 8 repeat measurements of filter in a time-symmetrical sequence, bracketing filter measurements with open beam measurements for each repeat measurement run. |
| Bandwidth | 1.0 nm |

*NIST's Measurement Technique:*
The transmittance measurements were performed on a custom-made spectrophotometer, which consists of a lamp-based source, a prism-grating monochromator, a sample carriage, and an optical detector attached to an integrating sphere. Details concerning the source, monochromator and detector are provided in Table B8. A schematic of the instrument is provided in the figure below.

After cleaning with an air bulb, each sample was mounted in a holder on the sample carriage with the identification number facing the incident beam in one of two possible sample orientations (see Table B8 Measurement Sequence). The sample was centered on the incident beam and aligned normal to the beam by retroreflecting a laser beam collinear with the axis of the incident beam. The maximum deviation from normal was 0.1º. The diameter of the incident beam was 17 mm.

The sample carriage consists of two incident positions for the beam: clear and sample. In each position, a shutter is used as a light trap for a dark signal. For each wavelength, the following signals were measured in this order: signal in the clear position, dark signal in the clear position, signal in the sample position, dark signal in the sample position, and signal in the clear position.

Net signals for the clear and sample positions are obtained by subtracting the dark signals, and the net clear signals, the first taken before the sample signal and the second taken after the sample signal, are averaged to minimize the effects of source drift and fluctuations on the timescale of a single transmittance measurement. The spectral transmittance of the sample was given by the net sample signal divided by the average net clear signal. Measurements were performed at wavelengths of 380 nm, 400 nm, 500 nm, 600 nm, 700 nm, 800 nm, 900 nm, and 1000 nm.

Figure B8. Schematic diagram of the NIST Reference Transmittance Spectrophotometer.

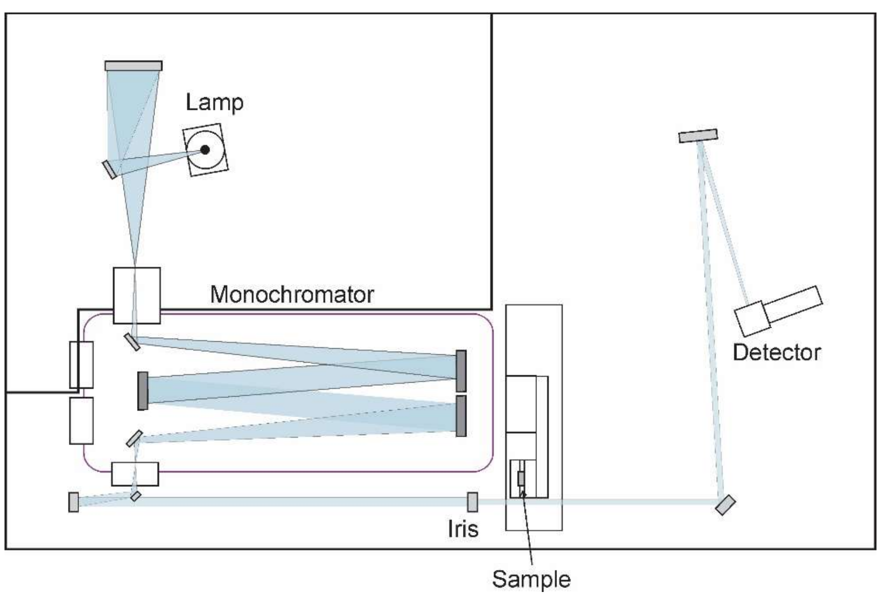


Table B8. Details of NIST's Measurement Setup

| | |
|---|---|
| Make and Type of Spectrophotometer | Custom-made spectrophotometer consisting of a 1 m, prism-grating monochromator (McPherson 2051). Light from the source is focused by a spherical mirror onto the 1 mm entrance slit of the monochromator. The grating has 600 lines/mm and is blazed at 200 nm. The exit aperture of the monochromator is 1 mm diameter circle. The slit, apertures, and grating yield a triangular slit function with a nominal bandwidth of 1.5 nm. The light exiting the monochromator is collimated by an off-axis parabolic mirror, reduced in diameter by an iris, and is incident upon the sample plane. Light passing through the sample plane is focused by a spherical mirror into an averaging sphere attached to a detector. The current from the detector is amplified and read by a digital voltmeter. |
| Additional Stray Light Rejection | None |
| Source Drift Monitoring | The incident beam is measured in the clear position before and after measurement in the sample position at each wavelength. The average clear signal is used to minimize the effects of source drift on the timescale of a single transmittance measurement. |
| Source | 150 W quartz-tungsten-halogen incandescent lamp |
| Detector | Silicon photodiode |
| Temperature | Calibrated temperature probe (Fluke 1620A Thermo-hygrometer). The average temperature was 23.98 °C. |
| Humidity | Calibrated hygrometer probe (Fluke 1620A Thermo-hygrometer). The range was from 5.3 % RH to 49.5 % RH. |
| Beam Size | 17 mm diameter |
| Beam Collimation | Collimated (maximum deviation from collimation at the sample is 0.2°) |
| Measurement Sequence | The spectral transmittance of each sample was measured at two different orientations to remove any effects from the slight polarization of the incident beam: (1) upright, with the filter identifier facing the incident beam and located at the upper right corner and (2) rotated 90º about its normal so that the filter identifier was facing the incident beam and was located at the lower right corner. In each orientation, the transmittance was measured 3 times at each wavelength and averaged. The unpolarized transmittance is the average of transmittance at each orientation. Measurements were repeated on 3 different days for each sample over 14 weeks. |
| Bandwidth | 1.5 nm |

**Disclaimer**

Certain commercial equipment, instruments, or materials are identified in this appendix in order to specify the measurement procedure adequately. Such identification is not intended to imply recommendation or endorsement by NIST, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.