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Comparison of NRC goniometric and integrating sphere methods for realizing an absolute diffuse reflectance scale

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ABSTRACT

At the National Research Council of Canada (NRC), the scale of diffuse spectral reflectance in the UV and visible range is realized using an integrating sphere method known as the Modified Sharp-Little method and has been well-characterized and validated. More recently, a gonireflectometer has been designed to capture the bidirectional reflectance of reflecting objects. This system incorporates a diode-array spectroradiometer and is prone to non-linearity errors. In this paper the magnitude of this non-linearity error is evaluated using several diffusely reflecting materials by comparing the angular-integrated reflection indicatrix measured with the gonireflectometer with results obtained using a transfer spectrophotometer that is traceable to the sphere-based NRC Absolute Reflectometer.

Keywords: diffuse reflectance, BRDF, goniometric method, sphere method, uncertainty

1. INTRODUCTION

Absolute measurements of diffuse reflectance are generally performed using integrating sphere methods although in recent years, several national measurement institutes have developed gonireflectometer instruments for this purpose. At the National Research Council of Canada (NRC), the scale of diffuse spectral reflectance in the UV and visible range is realized using an integrating sphere method known as the Modified Sharp-Little method and has been well-characterized and validated through international measurement comparisons. More recently, a gonireflectometer has been designed and built that incorporates a uniform light source for the sample illumination and a commercial diode-array spectrometer for the detection. It has been designed to capture the bidirectional reflectance of reflecting objects for dense wavelength sampling over the visible range, and for large numbers of in-plane and out-of-plane illumination and detection directions. The inherent parallel processing of the diode-array detectors makes these acquisitions practical in reasonable times. BRDF is obtained from the ratio of the radiance reflected by the sample to the radiance of the source itself. As the difference between these two radiances exceeds three orders of magnitude, the result can be prone to nonlinearity errors. In this paper the magnitude of this non-linearity error is evaluated using several diffusely reflecting materials by comparing the angular-integrated reflection indicatrix measured with the gonireflectometer with results obtained using a transfer spectrophotometer that is traceable to the sphere-based NRC Absolute Reflectometer. The differences are found to vary spectrally with a fixed pattern superimposed on a small offset that is only sample-dependent. A simple method is proposed to correct the fixed pattern deviations. Comparison of this corrected goniometric method with the NRC sphere method shows generally good agreement for realizing an absolute diffuse reflectance scale.

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Figure 1. Arrangements for the source and sample radiance measurements.

2. GONIOREFLECTOMETRIC MEASUREMENTS

2.1 Principle of the measurement

The NRC gonireflectometer is designed to measure the bidirectional reflectance distribution function (BRDF) of reflecting samples^{**1}. It comprises three main elements: a sphere source for the illumination, a spectroradiometer for light detection, and a five-axis robot and gear system for holding the sample and controlling its orientation relative to the illumination and reflection directions. The sphere source is internally coated with barium sulphate and houses a tungsten halogen lamp behind a white baffle to provide uniform radiance over the extent of an exit port delimited by a precision aperture. The spectral radiance at this exit port, L_s , is first measured by pointing the spectroradiometer directly towards it (Figure 1 - left). The sample is then brought in with the proper orientation and measured with the same spectroradiometer (Figure 1 - right) for its reflected spectral radiance L_r . The BRDF f_r , can then be obtained as

$$f_r(\theta_i, \varphi_i, \theta_r, \varphi_r) = \frac{1}{\pi} \frac{1}{\cos(\theta_i)} \frac{d^2 + r^2}{r^2} \frac{L_r(\theta_r, \varphi_r)}{L_s(\theta_i, \varphi_i)}, \quad (1)$$

where d is the source to sample distance, r is the radius of the exit port, and the θ, φ are the realized elevation and azimuth angles for the incident and reflection directions as measured from the normal to the surface.

The BRDF f_r expresses the ratio of the reflected radiance in direction (θ_r, φ_r) to the irradiance from direction (θ_i, φ_i) . A closely related concept is the radiance factor β which is defined as the ratio of the radiance reflected by the sample in the reflection direction to the radiance that would be reflected in that direction by a perfect white diffuser under the same illumination conditions. For directional illumination, one has

$$\beta(\theta_i, \varphi_i, \theta_r, \varphi_r) = \pi f_r(\theta_i, \varphi_i, \theta_r, \varphi_r). \quad (2)$$

It can be more intuitive to think about bidirectional reflectance in terms of β than in terms of f_r as $\beta=1$ for a perfect white diffuser.

2.2 Uncertainties

The influential factors that affect the uncertainty of the BRDF measurement can be classified in two categories: geometry-related and sensor-related.

The geometry-related measurement errors include the source-to-sample distance d , the aperture port radius r , the incidence angle θ_i , and sphere port non-uniformity. A type B evaluation of these geometry related errors leads to a

combined contribution to the standard uncertainty of typically 0.18% for 0° incidence at wavelength 550 nm, and 0.27% for 45° incidence.

The sensor-related errors relate to the diode array spectroradiometer used for the measurements of L_r and L_s . The earliest version of the setup used a Minolta CS-1000* covering the range 380nm to 780nm. This was then replaced by a Photoresearch PR-715 that extended the range to the near infrared up to 1068 nm. The system currently uses a Konika-Minolta CS-2000 for the range 380nm to 780nm. The repeatability and reproducibility of the CS-2000 have been tested using stabilized quartz halogen spectral irradiance standard lamps. A Type A estimation of the repeatability error is done by irradiating a pressed PTFE diffuser with one standard lamp and measuring repeatedly the spectral radiance reflected from the PTFE. The standard deviation of the spectral radiance is taken as the standard uncertainty due to repeatability errors. This uncertainty component is found to be 0.1% for a single measurement. This uncertainty can be reduced by averaging multiple measurements, in principle by a factor equal to the square root of the number measurements. The reproducibility error is estimated by substituting one spectral irradiance lamp for another one of the same type and looking at the stability of the ratio of the reflected radiances. This reproducibility error is found to be 0.1% and cannot be reduced by statistical averaging.

Excluding sample-related uncertainties, the combined standard uncertainty of the above mentioned components add up, in the quadratic sense, to 0.21% for 0° incidence at a wavelength of 550 nm. The break-down of the uncertainty components is 0.18% for geometry-related components, 0.1% for reproducibility, and 0.05% for repeatability assuming averages of four measurements. However, there could be other sources of uncertainty, and in particular possible non-linearity of the spectroradiometer.

2.3 Unknown uncertainties and need for validation

A possible source of uncertainty is non-linearity of the spectroradiometer for the 3 decade difference between the reflected radiance L_r and the source radiance L_s . This is difficult to estimate without an exact knowledge of what is actually occurring inside the spectroradiometer, which is a commercial instrument with proprietary hardware and software. From what we know of the spectroradiometer, the bright source measurement is achieved by the instrument choosing a short exposure time, typically 5 ms, while the sample measurement is done at longer integration time, typically 5s. These integration times are automatically chosen to produce digital counts close to saturation on the photodiode array. Dark noise subtraction is also automatically performed. The number of photoelectrons left after this subtraction should in principle be proportional to the spectral radiance multiplied by the integration time, and the instrument responsivity should then be linear. Other factors like charge leakage could make the raw responsivity nonlinear, but if these factors are present they could have been measured and corrected for by the manufacturer. It is possible to imagine a setup to test the linearity where the uniform light source would be attenuated by a known factor, say 1000. The ratio of the direct to the attenuated spectral radiance could serve to derive a correction factor if nonlinearity was observed. But without knowledge of the data processing inside the spectroradiometer, such correction factor might not be valid when the conditions are changed even slightly.

Translucency of the sample is another possible source of uncertainty. For translucent samples, the location at which light reflection occurs does not correspond with the physical surface but spreads inside the sample, possibly leading to a mis-estimation of the “effective” source-to-sample distance d . Translucency can also cause diffused photons to leak into and out of the measurement area in non-equivalent ways. Fluorescence can be also be a problem. When this is present for a sample, part of the incident power at a given wavelength in the excitation range, say blue, can be emitted at longer wavelengths, say red. As the NRC gonireflectometer uses polychromatic illumination (white light illumination) and monochromatic detection, this can lead to an overestimation of reflectance at the reemission wavelengths (reds). In comparison, a system that uses monochromatic illumination and polychromatic detection can overestimate the reflectance at the excitation wavelengths (blues). Sample translucency and fluorescence effects can be problems not just for BRDF measurements but also for sphere-based spectrophotometric measurements.

Comparing gonireflectometric measurements with data measured with other proven techniques is needed to validate the system and to understand better its behavior. This is the objective of the next section where we compare angle-integrated data to measurements obtained with a traceable sphere-based method.

* Certain commercial equipment, instruments, or materials are identified in this paper. Such identification does not imply recommendation or endorsement by the National Research Council of Canada, nor does it imply that the products identified are necessarily the best available for the purpose.

2.4 Angular integration of the hemispherical reflectance factor

The reflectance factor R can be defined as the ratio of the radiant flux reflected in the directions delimited by a given cone to that reflected in the same directions by a perfect reflecting diffuser identically irradiated. For directional irradiation this can be expressed mathematically using the radiance factor:

$$R = \frac{\iint_{\Omega_r} \beta(\theta_i, \phi_i, \theta_r, \phi_r) \sin(\theta_r) \cos(\theta_r) d\theta_r d\phi_r}{\iint_{\Omega_r} \sin(\theta_r) \cos(\theta_r) d\theta_r d\phi_r}, \quad (3)$$

where the integrals are carried over the extent of the given cone. As the solid angle of the cone approaches 2π sr, the reflectance factor approaches the reflectance for the same conditions of irradiation. Figure 2 shows the reflection indicatrix measured from a pressed PTFE sample. The incidence angle is here 0° and θ_r takes on values between -85° and $+85^\circ$ in steps of 2.5° . The region between -15° and $+15^\circ$ is where the sphere source occludes the detection path and the reflection indicatrix can be extrapolated in that region. For 0° incidence and for an isotropic sample the reflection indicatrix should not depend on ϕ_r and the integrals in (3) can be carried as a discrete summation over θ_r .

Figure 2. Reflection indicatrix of a pressed PTFE sample for 0° incidence and for wavelength 550 nm.

This calculation closely approximates the hemispherical diffuse reflectance factor for 0° incidence. This can be compared with measurements taken with sphere based spectrophotometers.

3. INTEGRATING SPHERE METHOD

3.1 NRC sphere-based absolute reflectometer

The NRC scale of absolute near-normal hemispherical reflectance factors over the wavelength range 360 nm to 850 nm is realized using the custom-built and well-characterized NRC absolute reflectometer, based upon the Sharp-Little method^{**2}, modified by Budde^{**3}. This instrument measures absolute spectral radiance factors in the d:0 or 0:d geometry utilizing an integrating sphere of 200 mm diameter coated with barium sulphate paint providing diffuse irradiation of the sample mounted at a 28 mm diameter port of the sphere. A schematic diagram of this instrument design is shown in

Figure 3 configured for d:0 geometry where the diffuse irradiation condition is achieved by the external tungsten lamp being focused onto the sphere wall and producing a secondary diffuse source within the sphere. A baffle coated with barium sulphate paint is mounted in the sphere to screen the sample from direct irradiation by this secondary diffuse source, so that it only receives indirect radiation from the sphere wall. The radiance factor of the sample is measured by the ratio of the radiance from the sample (Position B_s) to that of the unscreened sphere wall (Position B_w) after applying geometric corrections for aperture losses. The spectral selection is accomplished using a series of narrowband (about 10 nm bandpass) interference filters in conjunction with selected absorbing glass blocking filters. The uncertainty budget for these absolute d:0 reflectance factor measurements in the visible range (400 to 800 nm) is shown in Table 1. The metrological equivalence of the absolute diffuse reflectance factor scale realized using this instrument with that of other national measurement institutes (NMIs) has been demonstrated via international measurement comparisons^{**4}. The most recent of these comparisons has been CCPR K5. In all cases, the results have been in good agreement with the stated uncertainties.

Figure 3. Schematic diagram of NRC Absolute Reflectometer

Table 1. Uncertainty in spectral diffuse reflectance factor measurements ($k=1$) of white transfer standards in the visible range (400 nm to 800 nm) using the NRC Absolute Reflectometer.

Uncertainty Component	Type	Standard Uncertainty ($k=1$)	Relative Uncertainty in R
Signal-to-noise	A	0.02%	0.02%
Instrument stability (short term)	A	0.02%	0.02%
Instrument stability (long term)	A	0.05%	0.05%
Wavelength	B	0.15 nm	<0.01%
Detector linearity	B	<0.01%	<0.01%
Modified Sharp-Little method			
$R > 0.85$	B	0.05%	0.05%
$0.75 < R < 0.85$	B	0.10 – 0.05%	0.10 – 0.05%
$0.60 < R < 0.75$	B	0.15 – 0.10%	0.15 – 0.10%

3.2 NRC diffuse reflectance measurements transfer procedure

The NRC absolute diffuse reflectance factor scale is transferred from the NRC Absolute Reflectometer to other spectrophotometers using white diffuse reflectance transfer standards. The most important of these is pressed PTFE ($1.0 \pm 0.1 \text{ gm/cm}^3$) which is prepared according to an NRC quality system procedure to ensure reproducible pressings within 0.1 gm/cm^3 . This reproducible and controlled pressing of the PTFE reflectance standard is critical since its absolute reflectance has been found to be very dependent upon the pressing density^{**6}.

The main transfer spectrophotometer for NRC routine diffuse reflectance factor calibration services is a Perkin-Elmer Lambda-19 (PE19) spectrophotometer equipped with a 150-mm diameter integrating sphere accessory, coated with barium sulphate paint. This is a double-beam instrument where the signals from the test sample and the PTFE reflectance standard mounted at the reflectance sphere port are ratioed to the signal from a second pressed PTFE sample that is mounted at a fixed comparison sphere port. Since the test sample and reflectance standard are substituted at the same sample port and ratioed to the signal from the comparison port, this compensates for changes in the sphere's efficiency for these two measurement conditions.

The dark sample reading error is determined and corrected for by measuring a diffuse reflectance black cavity and these readings are subtracted from the raw measured signals for the sample and white reflectance standard. The additional uncertainties associated with the transfer of the NRC absolute diffuse reflectance scale to the PE19 spectrophotometer are mainly due to the reproducibility of the white transfer standard and the stability and linearity of the PE19 instrument. The uncertainty in the reproducibility of the transfer standard was evaluated by measurements on seven different pressed PTFE tablets. At 550 nm, this reproducibility component is 0.0076%. The quality control of the long-term instrument stability is verified at the time of each test sample measurement by also measuring the reflectance of two stable NRC reflectance standards (99% reflecting Spectralon standards) whose historical results have been monitored for several years. The nonlinearity of the photometric scale has been determined using calibrated neutral density filters inserted at the entrance port to the sphere accessory in the PE19. For high quality (nonscattering) neutral density filters, it is assumed that the total (regular and diffuse) transmittance measured by the integrating sphere should agree with the calibrated regular transmittance values on the NRC Reference Spectrophotometer under the same conditions of measurement. In the visible range, it has been determined that non-linearity error and instrument stability are 0.021% and 0.02% respectively. Thus, the overall Type B standard uncertainty ($k=1$) for NRC diffuse reflectance measurements in the visible range (380 to 800 nm) using the PE19 transfer spectrophotometer is estimated to be 0.20%. This error estimate excludes sample-induced effects such as: nonuniformity, translucency, scattering, temperature-sensitivity, hygroscopic, fluorescent and polarization sensitivity.

Figure 4. Sample recess correction error for the measurement of Spectralon-type samples using the PE19 equipped with a 150 mm sphere coated with sintered PTFE.

4. COMPARISON

Figure 5. Comparison of the uncorrected angle-integrated reflectance factor to the PE19 measurement for a PTFE sample.

4.1 Fixed pattern noise and correction

The comparison begins with pressed PTFE. A reflection indicatrix shown in Figure 2 was obtained at every nanometer from 380 nm to 780 nm following the method described in paragraph 2.1. The reflection indicatrix was then interpolated between -15° and $+15^\circ$ and the 0° diffuse reflectance was obtained as described in paragraph 2.4. When compared with the PE19 measurement (Figure 5), the angle-integrated reflectance is found to exhibit local deviations of about 1% in the near infrared, and as large as 6.5% at 380nm. We choose to name this spectral deviation “fixed pattern noise” (FPN) because the same pattern of deviation has been consistently observed at all the angles and for a variety of other white and grey samples. Even the small deviations in the region 450 nm to 650 nm seem repeatable. The origin of this FPN is unclear and is possibly due to local nonlinearities of the photodetection. Attempts to calibrate this effect in the lab have been unsuccessful.

The fixed pattern noise in the goniometric data can be eliminated by appropriate scaling to the PE19 data. Our method assumes that the PE19 data has negligible FPN and starts by adjusting a gain factor g between the PE19 reflectance factor R_{PE19} and the angle-integrated reflectance factor R_{gonio} by minimizing the Euclidian norm $\|R_{\text{gonio}} - gR_{\text{PE19}}\|$. This least-square adjustment is performed for the range 450 nm to 600nm, where the FPN is small, and it aims to compensate for errors that affect the scaling of the spectra independently of wavelength, like the geometry-related errors mentioned in Section 2.2. A FPN correction function C_{FPN} is then established as

$$C_{\text{FPN}}(\lambda) = \frac{g R_{\text{PE19}}(\lambda)}{R_{\text{gonio}}(\lambda)}, \quad (4)$$

to be applied to all BRDFs calculated with (1). Figure 6 shows the result for C_{FPN} . The correction oscillates around 1 for the region 450 nm to 600nm with a standard deviation of 0.001 which is taken as the standard uncertainty component for FPN correction. This component is added in quadrature to the other uncertainty components to give a combined standard uncertainty to 0.23%. By assuming a normal distribution and by multiplying the combined standard uncertainty by a coverage factor $k=2$, the expanded uncertainty for these BRDF measurements is estimated to be 0.46%, representing a level of confidence of approximately 95%.

Figure 6. Fixed-pattern noise correction factor to be applied to spectral BRDF data.

4.2 Comparison for reference materials

Angle-integrated reflectance factors corrected for FPN are compared here with those obtained with the sphere-based method for three different reference standards consisting of pressed PTFE, Spectralon (99%), and a matte Russian opal glass. The expected difference between the compared measurements is 0 with a ($k=2$) expanded uncertainty of 0.007 (quadratic sum of the uncertainties for the two measurements).

Figure 7 is the result for pressed PTFE. It can be seen that the fixed pattern in the gonireflectometer data has disappeared at the cost of a small increase of the local fluctuations. The difference between the gonireflectometer measurement and the PE19 is approximately 0.003 and constant throughout the spectral range. NIST 6°:d data for pressed PTFE published by Weidner and Hsia^{**6} is also shown for comparison. All these spectral measurements appear consistent with the stated uncertainties.

Figure 7. Angle-integrated reflectance factor after correction for the fixed-pattern noise in comparison with sphere-based measurements for a pressed PTFE sample.

Figure 8. Angle-integrated reflectance factor after correction for the fixed-pattern noise in comparison with sphere-based measurements for a Spectralon sample.

Figure 9. Angle-integrated reflectance factor after correction for the fixed-pattern noise in comparison with sphere-based measurements for an opal glass sample.

This result for PTFE also suggests that there could be a systematic bias of 0.003 between the two instruments, independent of wavelength.

Figures 8 and 9 are the results for the Spectralon and the opal glass samples. In the case of Spectralon the angle-integrated reflectance is found to be consistently 0.007 higher than the sphere-based measurement, at the very edge of the 95% confidence interval. This consistently higher difference for results obtained with a goniometric method compared with a sphere-based method for 0:d radiance factor measurements was also reported by Chunnillal^{**5} for a comparison of NPL scales. In this case, it was suggested that these discrepancies might be due to the different manner in which the samples are probed in the two techniques. However, it was subsequently reported by Holopainen^{**7}, that light scattering around the main beam which is always present in a goniometric measurement system, might be primarily responsible for these observed differences between the goniometer and integrating sphere based measurement methods.

In our comparison of the NRC absolute diffuse reflectance scales, it was found that for opal glass this difference increases to 0.001 and is not fully explained by the estimated combined uncertainty. If we accept that there is a 0.003 bias between the two techniques used here, the residual bias, 0.004 for Spectralon and 0.007 for opal glass, appear to be related to sample-induced effects related to geometry alone as they do not depend on wavelength. One plausible explanation is differences in sample translucency.

The impact of sample translucency on diffuse reflectance measurements is illustrated in Figure 4 for a series of grey Spectralon-type samples measured using the PE19 spectrophotometer equipped with a second integrating sphere accessory with a sintered PTFE coating. This coating is much thicker (about 5-10 mm) compared with the integrating sphere accessory with the barium sulphate paint (about 1-1.5 mm) in order to achieve totally opaque wall reflectance. The magnitude of this error is not wavelength dependent but is very dependent on the type of sample and, in particular, the sample translucency.

It is tempting to attribute the observed differences to an uncorrected linearity error of approximately 0.005 for the CS-2000 measurements. After such correction, gonireflectometer measurements would be reduced by 0.5% and would be consistent with the PE19 within the stated uncertainties. The problem with this explanation is that in a recent intercomparison with PTB Germany⁸, NRC gonireflectometric measurements were found to be consistently lower than PTB gonireflectometric measurements by approximately 0.75%, and the aforementioned possible linearity correction would raise that difference to 1.25%. We prefer to trust the linearity of the CS-2000 and attribute the observed bias to sample-induced effects, which we have consistently observed over recent years for other reference materials and with different spectroradiometers used on the gonireflectometer.

5. CONCLUSION

A gonireflectometer designed to measure BRDF and radiance factors has been described, and the uncertainties associated with this instrument have been quantified. For validation purposes, angular-integrated BRDFs have been compared with hemispherical radiance factors measured with a well established NRC sphere-based technique. The comparison indicates that the measured BRDFs are affected by a high-frequency fixed-pattern spectral noise which may originate from local non-linearities of the diode-array detector. This spectral noise is removed by scaling to the sphere-based measurement in a way that preserves the overall scaling of the BRDF. The corrected angularly-integrated data is consistently higher by a small amount that is consistent with previously reported comparisons between goniometer and sphere-based methods for realizing absolute diffuse reflectance factor scales. Furthermore, the comparison data appear to be affected by an additional small systematic bias which is attributed to sample-induced effects. This is affecting the gonireflectometer, the sphere-based spectrophotometer, or both, and should be investigated further.

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