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REPORT

Determination of Uncertainties in Diffuse Reflectance and Radiance Factor measurements using the National Reference Reflectometer

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Centre for Optical and Environmental Metrology

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Abstract

Absolute scales of diffuse reflectance and radiance factor have been established at NPL using the National Reference Reflectometer. The basic system uses two concentric rotary tables upon which the detector and a reflecting sample are mounted. This enables any incident and reflected beam combination to be measured. The absolute diffuse reflectance can be calculated by scanning the detector in the horizontal plane and numerical integration of the results.

All sources of uncertainty associated with the measurement have been identified and their contribution to the final uncertainty calculated. Included in the report are the results of a recent comparison of diffuse reflectance and radiance factor measurements with the National Institute of Standards and Technology (NIST), USA which give confidence in this uncertainty evaluation.

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National Physical Laboratory
Queens Road, Teddington, Middlesex, TW11 0LW

Approved on behalf of the Managing Director, NPL
by D H Nettleton, Head, Centre for Optical and Environmental Metrology

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1 Introduction

At present the UK 0°/45° radiance factor scale is directly traceable to the German national standards laboratory, PTB. The UK National Reference Reflectometer has been designed and built to provide the UK's national scale for regular reflectance and for diffuse reflectance and radiance factor. A milestone in the current NMSPU programme for Optical Radiation was to reduce the measurement uncertainty for diffuse reflectance and radiance factor measured on the National Reference Reflectometer.

To obtain the 0°/d diffuse reflectance, the individual radiance factors are measured at 9° intervals in a semi-circle around the sample. This sequence includes the 45° position, commonly used for radiance factor measurements, which will be considered here. The absolute diffuse reflectance can be determined by numerical integration of these measurements.

The following report outlines the processes used to determine the present measurement uncertainty for 0°/45° radiance factor measurements using the National Reference Reflectometer, but is equally valid for determining other incident and reflected beam combinations.

2 Sources of Uncertainty

When calculating measurement uncertainties it is important to firstly identify all possible sources of uncertainty. For radiance factor the sources of uncertainty thought to be significant are as follows:

- Repeatability (Type A)
- Stray light
- Wavelength calibration and its effect on the reflectance
- Uncertainty in the DVM readings from detector signals
- System linearity
- Detector and sample angle readouts and the effect of angle on the reflectance
- Aperture dimensions, diameter and depth of aperture from its holder, and the effect on reflectance
- The distance from the sample surface to the aperture
- Sample thickness

Apart from repeatability, all other sources of uncertainty are Type B. All these must be considered when determining the measurement uncertainty. For all these factors, with the exception of repeatability and stray light, the uncertainty in the measured quantity was determined first and then its effect on the uncertainty of the radiance factor values was calculated. These values were then used in the final uncertainty budget.

2.1 Repeatability

A Spectralon, 23764-1-1, used in a recent intercomparsion with NIST, was measured six times in the 0°/45° geometry. The sample was removed from the sample jaws and replaced for each measurement. The measurements were made on 2 separate days. The sample was measured in nominal 100 nm wavelength steps from 400 - 1000 nm. In all cases measurements were taken with the reflected beam on both sides of the incident beam. The reflectances were then converted into radiance factor values by means of a geometrical factor (see page 4). For each set of 6 results, at each wavelength and angle, the standard deviation and standard deviation of the mean value were calculated using the equations shown below. The highest value for the standard deviation of the mean was used in the final uncertainty calculation. The largest value was 0.065 %.

The standard deviation and standard deviation of the mean value were calculated using the following equations:

Standard Deviation, $$s(q_j) = \sqrt{\frac{1}{(n-1)} \sum_{j=1}^{n} (q_j - \bar{q})^2}$$

Eqn. 1

Standard deviation of the mean value, $$s(\bar{q}) = \frac{s(q_j)}{\sqrt{n}}$$

Eqn. 2

where n is the number of repeated values
q_j is the measured quantity
q is the mean value

2.2 *Stray Light*

Two different tests was used to determine the effect of stray light. The first test measured how much of the light reflected back onto the sample from the surroundings and the second test measured the amount of light that was spilling beyond the edge of the detector aperture and not included when measuring the direct beam.

For the first test two targets were used. One was placed at the sample position and was overfilled by the light patch, the other was positioned between the first target and the detector so as to block the direct beam. Even when a large piece of white card was placed inside the light tight cabinet, the effect was found to be negligible.

For the second test apertures of progressively larger size were fitted in front of the detector. The direct beam was blocked by means of an opaque mask slightly larger than the incident beam. It was found that provided that the aperture diameter is 2 mm larger than the light patch the effect can be minimised. This investigation showed that the stray light component was a maximum of 0.05 %.

2.3 *Wavelength Calibration*

At present the wavelength for radiance factor measurements is determined by a series of interference filters. The uncertainty in the knowledge of the transmittance wavelength of these filters has been determined previously [1]. An uncertainty of ± 0.6 nm is stated. This uncertainty is based on repeatability measurements made on a Cary 5 Spectrophotometer, whose wavelength scale is checked periodically using a wavelength standard calibrated on the National Reference Spectrophotometer, and on the observed change in wavelength over time. The effect of this uncertainty on percentage radiance factor for a neutral, i.e. white sample, however will be negligible. A nominal value of 0.001 % has therefore been assigned.

2.4 DVM Readings

Using data from the most recent DVM calibration (24/08/99) the effect of the uncertainty in the DVM readings on percentage radiance factor has been calculated. A change in the voltage reading would mean a change in the reflectance of the sample. This in turn will change the radiance factor.

The lowest signal level recorded during these measurements was 0.15 V. The corresponding uncertainty associated with this voltage is 0.004 %. The reflected signal voltage was then changed by this uncertainty, as was the incident beam voltage changed by its uncertainty, in this case, at 409 nm, by 0.004 % also. The effect these changes have on the radiance factor value can then be determined.

In this investigation the largest change in radiance factor was calculated to be 0.008%.

2.5 Linearity and amplifier range changing

The amplification of the detector current uses an operational amplifier in the detector head with feedback resistors which can be switched in decades by the computer. By increasing the gain by 10 and reducing the sensitivity by 10 the ratios of the successive resistor values can be determined. Some of the results of which are shown below.

Range change	$10^6/10^7$	$10^7/10^8$	$10^8/10^9$
	9.9938	10.0431	9.9674
	9.9930	10.0222	9.9648
	10.0052	10.0299	9.9612
	10.0049	10.0307	9.9553
	10.0100	10.0354	9.9748
	10.0119	10.0279	9.9575
	9.9999	10.0425	9.9691
	9.9998	10.0357	9.9601
	9.9939	10.0315	
Mean	10.001	10.033	9.964
STD	0.007	0.007	0.006
STD of mean	0.002	0.002	0.002

For the $10^7/10^8$ and $10^8/10^9$ ranges a correction is applied to the results to account of the non-linear behaviour of the gain ratios. Measurements will typically use signal ranges of 1000:1 and 10000:1. The value of the standard deviation of the mean from the data shown above is the uncertainty in the range changing between the resistors. This uncertainty will have an effect on the measured reflectance and radiance factor, which can be calculated. For a white sample with a radiance factor of greater than 100 % the uncertainty due to linearity and range changing is 0.024 %.

3 Calculating Radiance Factor

To calculate the radiance factor β, the ratio of the reflected light to the incident light is multiplied by a geometrical factor F and divided by cos θ, where θ is the detector viewing angle, in this case 45°. The equation is shown below:

$$\beta = \frac{F}{\cos\theta} \times \frac{I_R}{I_o}$$

Eqn. 3

where θ is the detector viewing angle
I_R is the reflected beam flux
I_o is the incident beam flux
F is a geometrical factor

F is calculated using equation 4 below,

$$F = \left[\frac{2 \times \left(M + D_p \right)}{D_a} \right]^2$$

Eqn. 4

where M is the distance from the sample surface to the defining aperture
D_p is the depth of the aperture shim below the holder
D_a is the diameter of the aperture

3.1 Angle

The verification of the turntable readout of angle has been described elsewhere [2] but briefly it is achieved by mounting a pair of mirrors on to the front of the detector and mirror in the sample jaws. A theodolite is then used to monitor the deviations as the sample and detector turntables are moved in discrete steps covering 360°. An example of the results is shown below. In this case the sample turntable was asked to move in 7.826° steps, the detector in steps twice as big, i.e. 15.652°. The actual angle moved and difference from the angle that each turntable was asked to move are shown.

N	Sample turntable 7.826° steps		Detector turntable 15.652° steps	
	angle moved	Diff (moved-asked)	angle moved	Diff (moved-asked)
1	7.823	-0.003	15.648	-0.004
2	7.817	-0.009	15.651	-0.001
3	7.818	-0.008	15.654	0.002
4	7.819	-0.007	15.658	0.006
5	7.823	-0.003	15.659	0.007
6	7.814	-0.012		
7	7.808	-0.018		
8	7.820	-0.006	15.645	-0.007
9	7.814	-0.012	15.663	0.011
10	7.822	-0.004	15.664	0.012
11	7.826	0.000	15.661	0.009
12	7.825	-0.001	15.662	0.010

From the above data the largest difference is 0.018°. Taking this into account the final uncertainty has been assigned a value of ± 0.02°. The effect of this uncertainty on radiance factor has been calculated to be 0.035 %.

3.2 Distance from sample surface to aperture

The distance from the sample surface to the defining aperture must be known in order to calculate the radiance factor. In practice the distance from the rear of the sample to the shoulder of the aperture holder is measured. This is because the surface of the sample being measured must not be touched and also because the detector aperture itself is too delicate to be contacted. Therefore the depth below the shoulder of the aperture shim must also be measured and then added to the distance between sample and aperture.

3.2.1 Distance from rear of sample to aperture mount

The distance from the sample to the aperture shoulder is measured using a stick micrometer. This, in practice, proves awkward to measure. The user must lean into the reflectometer cabinet and measure a distance of approximately 360 mm, whilst being careful not to damage the aperture foil itself. To test the repeatability of the stick micrometer measurements, a test rig was set up on a bench.

A plate was fixed onto the bench as a reference point. Another plate was then mounted onto a linear translation slide with micrometer drive. The distance between the two plates was measured. The translation stage was then driven an unknown amount and the distance re-measured, using the stick micrometer. The difference between the two micrometer readings was recorded. The results of this test are shown below.

	Micrometer readings (mm)			
	Before	After	Distance (mm)	Difference (stick-linear)
Linear Slide (LS)	12.50	11.45	1.05	0.04
Stick micrometer (SM)	330.07	331.16	1.09	
LS	8.75	5.43	3.32	0.02
SM	333.87	337.21	3.34	
LS	7.07	14.19	7.12	0.02
SM	335.61	328.47	7.14	
LS	17.17	9.89	7.28	0.04
SM	325.50	332.82	7.32	
LS	7.40	8.11	0.71	0.02
SM	335.35	334.62	0.73	

The uncertainty of measurement using the stick micrometer has been estimated to be ± 0.2 mm. This includes the correction from the stick micrometer calibration, the repeatability measurements shown above and from experience. The effect of this uncertainty on the radiance factor is 0.1 %.

3.2.2 Depth of Aperture Shim Below Holder

The depth of the aperture shim below the surround was measured using a Societe Genevoise SIP 414M measuring machine. The microscope eyepiece was used to focus on to the aperture itself and then on to the surround. The depth was calculated as the difference between the two readings. The depth of the aperture below the surround, for a number of apertures has been measured. The results are shown below.

Diameter (mm)	6	9	12	13	30
	1.86	1.84	1.96	1.85	1.93
	1.84	1.82	1.99	1.80	1.97
	1.85	1.85	1.93	1.85	1.97
	1.84	1.81	1.95	1.79	1.95
		1.86			1.96
		1.85			2.02
mean	1.848	1.836	1.958	1.823	1.966
STD	0.010	0.021	0.025	0.032	0.030
STD of mean	0.005	0.008	0.013	0.016	0.012

The depth will differ for different apertures. For the repeatability measurements mentioned earlier a 12 mm (nominal) aperture was used. The standard deviation of the mean from the values shown above was used to calculate the effect on the radiance factor. This was found to be 0.007 %.

3.2.3 Sample Thickness

The thickness of the sample can be calculated from the difference between the measured distance, using the stick micrometer, from the front surface of a dummy sample to that of the rear surface, when rotated by 180°, to the detector aperture shoulder. If the value obtained using this method is equal to the thickness measured using a micrometer then the sample surface is centred on the axis of rotation of the sample turntable. The thickness the dummy sample was measured using a micrometer at a number of different places around the samples' circumference. The results are shown below.

	Thickness of sample
	9.27
	9.26
	9.26
	9.27
	9.27
mean	9.266
STD	0.005
STD of mean	0.002

Using the value of the standard deviation of the mean from above, the effect on the radiance factor has been calculated as 0.001 %.

3.3 Diameter of aperture

The diameter of a number of apertures was measured on a Societe Genevoise SIP 414M machine. The microscope eyepiece was used to focus on a point on the circumference of each aperture. The distance to the point on the opposite side of the circumference was then measured. The midway point between these two points then lies on the diameter of the aperture. The distance across the diameter found by this method was then measured. The process was repeated a number of times, across different diameters, the results of which are shown below.

Nominal Diameter (mm)	6	9	12	13	30
	5.997	8.985	11.988	12.970	29.985
	5.999	8.987	11.989	12.972	29.953
	5.999	8.990	11.991	12.970	29.994
	5.999	8.986	11.987	12.974	29.953
		8.988	11.990	12.974	29.951
			11.992	12.971	29.978
Mean	5.999	8.987	11.990	12.972	29.969
STD	0.001	0.002	0.002	0.002	0.019
STD of mean	0.0005	0.001	0.001	0.001	0.008

A 12 mm (nominal) aperture was used for measurements to establish the scale of radiance factor. Using the data shown above, the standard deviation of the mean for the 12 mm aperture was used to calculate the effect on the radiance factor. This was calculated to be 0.01 %.

3.4 Calculating uncertainty of radiance factor

Table 1 sets out the uncertainty budget for the Radiance Factor scale realised on the National Reference Reflectometer. The various contributions to the uncertainty budget have been combined in quadrature to obtain an expanded uncertainty of 0.2% at the 95% confidence level. This is in line with the target set for milestone 1.6.26.

Table 1. Uncertainty budget for NPL radiance factor measurements using the National Reference Reflectometer

Symbol	Source of Uncertainty	Limiting value (%R)	Probability distribution	Divisor	c_i	$u_i(R)$
R	Type A uncertainty	0.065	normal	1.0	1.0	0.065
W_c	Wavelength (neutral samples)	0.001	rectangular	1.732	1.0	0.001
S	Stray light	0.05	rectangular	1.732	1.0	0.029
V	DVM	0.008	rectangular	1.732	1.0	0.005
L	Linearity (amplifier range changing)	0.024	rectangular	1.732	1.0	0.014
A	Angle	0.035	rectangular	1.732	1.0	0.020
M	Sample to aperture distance	0.1	rectangular	1.732	1.0	0.064
D_a	Diameter of diaphragm	0.01	rectangular	1.732	1.0	0.007
D_p	Depth of diaphragm	0.007	rectangular	1.732	1.0	0.004
T	Sample thickness	0.001	rectangular	1.732	1.0	0.001
$u_c(R)$	Combined uncertainty		normal			0.100
U	Expanded uncertainty		normal (k = 2)			0.200

4 Comparison with NIST

In late 1998 and early 1999, NPL conducted a bilateral comparison with the National Institute of Standards and Technology in the USA. Both laboratories used a gonio-reflectometer system [2] to make the measurements. Measurements of individual radiance factors at 9 degree intervals over a full semi-circle were made on a white Spectralon sample over the wavelength range 400 to 1000 nm. Measurements included the 45° position which is the most frequently used radiance factor geometry and is commonly used in the measurement of surface colour. Numerical integration of the individual radiance factors then yields the 0°/d diffuse reflectance value. The results of the comparison are shown in Table 2 on page 10.

Examining the results one finds good agreement over all wavelengths with the maximum difference in diffuse reflectance being 0.2%. The agreement in the radiance factor values is also good, around the 0.2% level. The larger differences found at the larger angles is to be expected as the signal levels are smaller, but the effect of these on the overall diffuse reflectance values are minimal. The NIST uncertainties are not currently available, but as they use a similar system the uncertainties can be expected to be at a similar level. Thus the differences between the measurements made at the two laboratories fall within the combined expanded uncertainty.

Table2. Results of comparison of diffuse reflectance and radiance factor measurements between NPL and NIST.

NPL Results

	Radiance factor (%)						
Angle (°)	400 nm	500 nm	600 nm	700 nm	800 nm	900 nm	1000 nm
9	106.85	106.88	106.91	106.93	106.93	106.93	106.87
18	105.82	105.84	105.84	105.82	105.80	105.77	105.70
27	104.69	104.71	104.70	104.64	104.63	104.60	104.54
36	103.31	103.29	103.27	103.22	103.21	103.18	103.15
45	101.48	101.48	101.38	101.43	101.40	101.36	101.29
54	99.04	99.06	99.04	99.01	99.00	98.95	98.93
63	95.63	95.71	95.67	95.67	95.65	95.65	95.56
72	90.74	90.71	90.74	90.75	90.70	90.73	90.70
81	81.72	82.18	82.31	82.29	82.29	82.34	82.27
0°/d value	99.75	99.78	99.77	99.76	99.74	99.72	99.66

NIST Results

	Radiance factor (%)						
Angle (°)	400 nm	500 nm	600 nm	700 nm	800 nm	900 nm	1000 nm
9	106.72	107.02	107.12	106.92	107.02	106.92	106.92
18	105.66	105.96	105.96	105.86	105.86	105.76	105.56
27	104.46	104.76	104.76	104.66	104.66	104.56	104.46
36	103.12	103.32	103.22	103.32	103.22	103.12	102.92
45	101.25	101.45	101.35	101.55	101.45	101.35	101.05
54	98.82	98.92	98.82	99.12	99.02	98.92	98.62
63	95.52	95.62	95.42	95.82	95.72	95.62	95.22
72	90.58	90.68	90.48	90.98	90.88	90.78	90.38
81	82.82	82.62	82.22	82.82	82.52	82.42	82.22
0°/d value	99.63	99.80	99.69	99.88	99.80	99.70	99.45

Difference (NPL - NIST)

	Radiance factor (%)						
Angle (°)	400 nm	500 nm	600 nm	700 nm	800 nm	900 nm	1000 nm
9	0.13	-0.14	-0.21	0.01	-0.09	0.01	-0.05
18	0.16	-0.12	-0.12	-0.03	-0.06	0.01	0.14
27	0.23	-0.05	-0.06	-0.02	-0.03	0.04	0.08
36	0.19	-0.04	0.05	-0.10	-0.02	0.06	0.23
45	0.23	0.03	0.03	-0.12	-0.05	0.01	0.24
54	0.22	0.13	0.22	-0.11	-0.02	0.03	0.31
63	0.11	0.09	0.25	-0.16	-0.08	0.02	0.34
72	0.17	0.03	0.26	-0.22	-0.18	-0.05	0.32
81	-1.10	-0.44	0.09	-0.53	-0.23	-0.08	0.05
0°/d value	0.12	-0.02	0.08	-0.12	-0.06	0.02	0.21

5 References

[1] G.H.C. Freeman, A. J. Deadman, Reference Reflectometer Report No. 9, December 1994

[2] D. C. Williams, Anal. Chim. Acta 380 (1999) 165-172