Directional Reflection measurements on highly reflecting coatings

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Abstract

We developed spectrophotometry accessories for measuring absolute reflection on highly reflecting samples, not only at near-normal incidence but also at oblique incidence for incident angles up to more than 80 degrees. The accessories are designed for use with a high-performance UV/Vis/NIR industrial spectrophotometer and are widely used for the accurate characterisation of mirrors. Our directional VW accessory is designed to calibrate front- and second-surface reference mirrors based on reflecting metallic layers while our directional IV accessories are specially designed for the characterisation of multilayer dielectric materials like laser mirrors.

In this paper the features of these tools are discussed and an evaluation of the measurement uncertainties is given.

Keywords: spectrophotometry, reflection, laser mirror

1. Introduction

Laser mirrors are multilayer coatings optimized to have absorption close to zero at the wavelength of interest. To quantify the absorption one needs to measure the reflection, a value close to one, extremely accurately. A similar demand for a highly accurate reflectance measurement lies in the calibration of reference mirrors used as reflectance standard in spectrophotometry.

A device commonly used for the calibration of reflectance standards is the so-called VW accessory by Strong [1]. The advantage of a VW reflectometer is that it features a double reflection on the sample so that the square of the reflectance is measured. This reduces the measurement errors in the reflectance by 50% which is a great advantage when high accuracy is important.

A weakness in Strong's original design is, that separate (matched) mirrors are used for setting the 100% and for the sample measurements. Modified versions have been proposed that utilise a moving mirror to solve this problem [2,3].

A relatively complicated design in which sample tilt is compensated by the optics was proposed by Bennet and Koehler [3]. Other reflectometer designs for specular reflectance have been proposed by Voss, Plass and Giesen [4] and by Weidner and Hsia [5].

A commercially available VW accessory for the Lambda 900 spectrophotometer from PerkinElmer is the PELA 1029 developed by Labsphere Inc.. The PELA 1029 is originally intended to be used in combination with the instrument's standard detector and uses the front beam as reference and the back beam as sample beam. Using the standard detector makes the accessory difficult to align, a problem that was also observed in a previous design for the older Lambda 19 [6]. For this reason, we decided to develop a new VW accessory that can be used in combination with an integrating sphere unit. The result is the directional VW accessory discussed in 2.1. This accessory is now the only commercially available VW accessory that can be used at near-normal as well as oblique incidence.

At oblique incidence however, the VW method proved to be less suitable for measurements of laser mirrors and another method, referred to as the IV method, was developed.

In the following sections we will discuss both methods.

2. Absolute measurement of specular reflectance

2.1 The VW method

The measurement principle of the directional VW absolute reflectance accessory is based on a combination of two measurements (see Fig. 1).
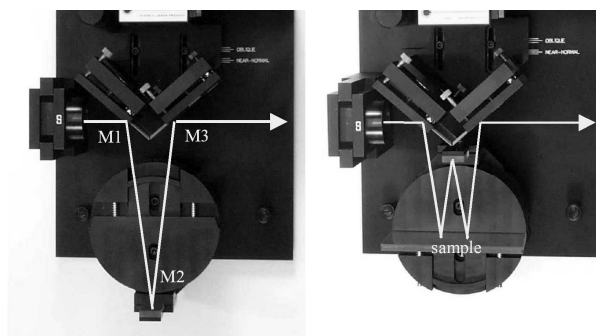

Fig. 1. The directional VW accessory measuring at normal incidence in the V-mode (left) and W mode (right).

In the so-called V-mode the instrument beam is interacting with three mirrors (M1, M2 and M3). In the so-called W-mode the beam additionally interacts twice with the sample. The ratio of the two readings produces the square of the sample reflectance.

In addition to the (near-) normal incidence (8 degrees), the accessory is capable of performing measurements under oblique incidence. In this case, two additional forms of the W -mode are possible, representing "positive" and "negative" angles of incidence (see Fig. 2).

The ability to perform measurements at both "positive" and "negative" angles, is important since it gives the user the possibility for compensating for systematic errors related to beam-shift effects and angular accuracy, by taking the average of these two types of measurements.
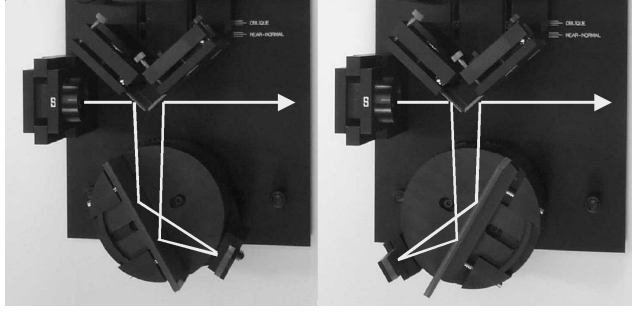
Fig. 2. The directional VW accessory measuring at oblique incidence for positive angle of incidence (left) and for negative angle of incidence (right).

M2 is a spherical mirror that compensates for the elongated optical path. The optical design is optimised to get the smallest possible measurement spots on the sample (approximately 6 mm wide and 10 mm high at 15 degrees).

In the case of a measurement at near-normal incidence, the angle of incidence on mirror M2 must be as small as possible but large enough to make sure that in the W-mode the beam can pass this mirror on either side without vignetting. Typically this angle is 8 degrees and is determined by the position of the mirrors M1 and M3. These mirrors are mounted on the same block which can be shifted backward to get a "sharper" angle on M2. In the case of oblique incidence, the angle of incidence on mirror M3 must be as small as possible. In our case, this angle is 5 degrees and requires the mirrors M1 and M3 to be shifted backward relative to the "near-normal" position. Markers were engraved on the base plate of the accessory to indicate the proper position of the mirror block.

In the W-mode, there are always two reflections on the sample, one coming from mirror M1 (which is reflected towards M2) and one from the V-mirror M2 (which is reflected towards M3). In the case of oblique incidence with the angle set to θ, the average angle of incidence of the beam coming from mirror M1 is actually $\theta_1 = \theta + 5°$, while for the second beam coming from the V-mirror M2 the average is $\theta_2 = \theta - 5°$. This effect is shown in Fig. 3 where the angles are somewhat exaggerated to make the effect more visible.

The systematic error related to this effect is given by

$$\Delta R = R(\theta) - \sqrt{R(\theta + 5^o)R(\theta - 5^o)} \qquad . \qquad (1)$$

In the case of a aluminium coated mirror, this systematic error typically is in the order of only 0.05% - 0.4%, depending on the wavelength, polarisation and θ . An example of a calibration of a second surface reference mirror is shown in Fig. 4.
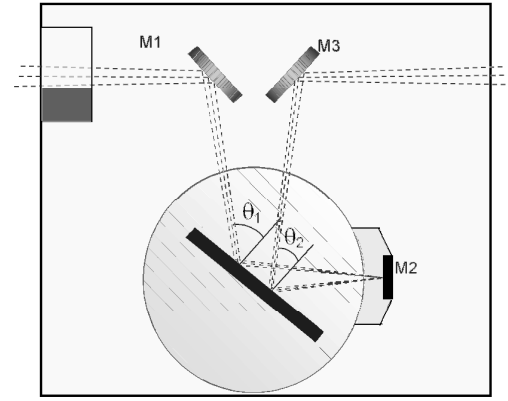

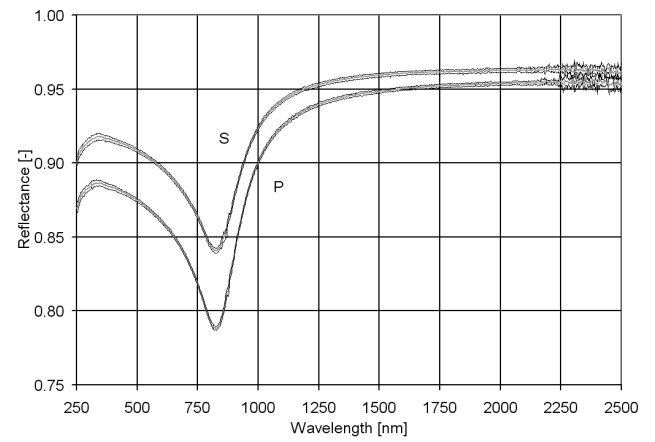Fig. 3. Different angles of incidence on the sample at oblique incidence.

Fig. 4. Calibration results for an aluminium coated second surface mirror measured with P and S polarised radiation at 60°.

2.2 Using an Integrating Sphere detector

We can expect a slight misalignment between the beams in the V and W modes of the VW accessory. The best way to cope with these alignment errors is to use an Integrating Sphere as detector, as shown in Fig. 5. In this case we have installed one of our reflection accessories in the sample compartment of our PerkinElmer Lambda 900 spectrophotometer and used a standard 60 mm integrating sphere as detector, for which the diffuse reflection target at the back is placed under 0° incidence by removing the 8° wedge between target and sphere.

If the sample in the reflection accessory is in some way distorting or slightly changing the direction of the beam, the entrance port still captures all radiation as long as the beam stays centered within the entrance port of the sphere, thereby strongly reducing influence of misalignment.

Fig. 5. Set-up used for reflectance measurements.

2.3 The IV method

In the case of the second surface mirror of Fig. 4 the systematic error cased by the different angles for the two reflections on the sample has proven to be very small for angles below 60 degrees and still quite acceptable at 75 degrees. However, the effect is disastrous in the case of multilayer coatings like laser mirrors.

The shape of the reflectance spectrum of such material is determined by constructive and destructive interference of radiation reflected at the layer interfaces and this effect is sensitive to small changes in the angle of incidence.

In Fig. 5 we compare the true reflectance of a laser mirror for an angle of incidence of 45 degrees with the reflectance as measured using the VW accessory. The results show a large difference between the true values and the ones measured with the VW accessory.
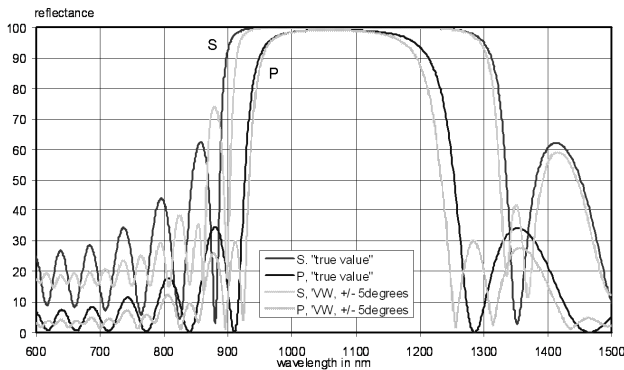

Fig. 6. Simulated true values (ideal measurement with zero beam divergence) versus simulated VW measurements with reflections at 40° and 50° (average 45°).

In order to solve this measurement problem we developed a new technique which we call the IV method. The principle is compared with the VW method in Fig. 6.
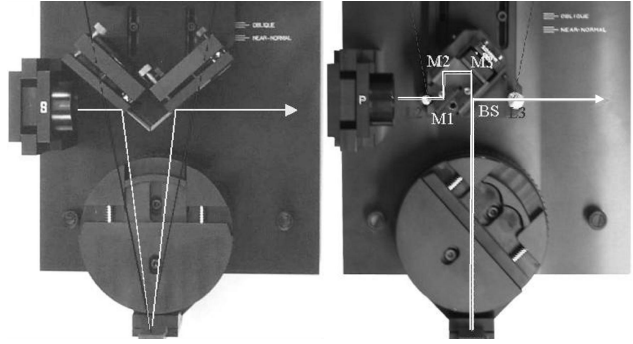

Fig. 7. Top view of the VW accessory (left) and IV accessory (right) in the reference mode (without sample), showing the optical path of the sample beam.

In this top view you see the two accessories in the reference mode, which is the V mode for the VW accessory, and the I mode for the IV accessory

In the IV accessory, the beam coming from the polariser (in Fig. 6 shown in the P position, as indicated by the "P" on top), passes 3 mirrors before it is transmitted by a broadband polkadot beamsplitter (BS) and directed to the mirror on the rotation stage.

This mirror then redirects the beam back towards the beamsplitter where it is reflected 90° towards the exit window of the sample compartment to interact with the detector unit

Fig. 7 shows the IV accessory in the V mode which is the sample measurement mode for this accessory. Not only are the two reflections on the sample at the same spot (in the axis of rotation of the sample), the also have the same angle of incidence which makes the method suitable for multilayer dielectric coatings.
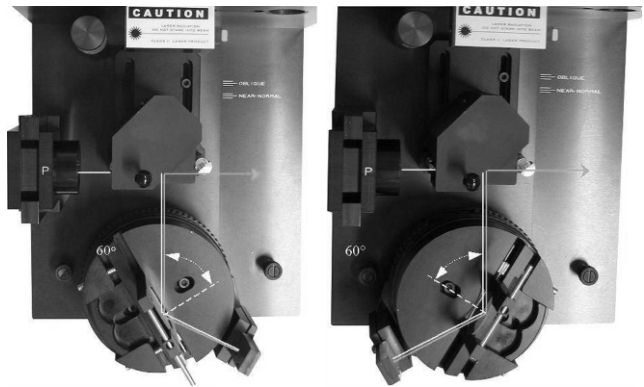

Fig. 8. Top view of the IV accessory in the V-mode, for "Positive" V configuration (left) at +60° incidence and "Negative" V configuration (right), -60° incidence, showing the optical path of the sample beam.

2.4 Laser alignment

Both VW and IV accessories have a build-in laser alignment which makes switching between reference and sample modes easier. The path of the laser beam in the VW accessory is shown in Fig.9.
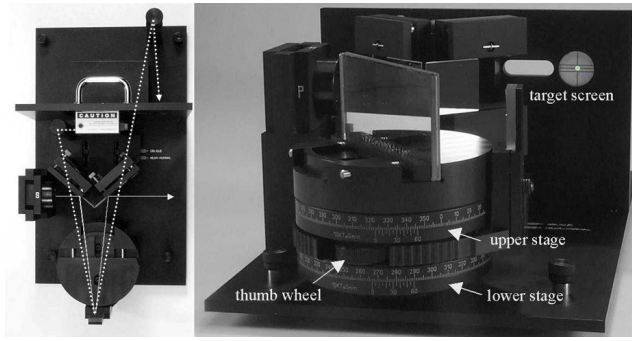
Fig. 9. VW accessory seen from the top (left) in V-mode, showing the optical path of the laser alignment beam, and a view up front, giving a close-up view of the rotation stages and the laser target screen.

The laser module contains a switch to turn of the laser during measurements. Before using the laser, the upper stage which controls the angle of the sample holder is set to the appropriate angle and fixed by turning the thumb wheel. For a reference measurement, the sample is removed and the entire sample stage assembly (upper stage+sample holder+V-mirror) is rotated to get the V-mirror into the reference position. The correct position is obtained when the laser beam hits the target screen in the centre. For a sample measurement, we only need to install the sample and rotate the entire sample stage assembly back into the W-mode position, which is also indicated by the laser hitting the same target position!

In case of the IV accessory, the procedure is quite similar. The only difference is that the laser beam now has to follow the exact part of the sample beam. This is accomplished with the help of two mirrors just before and after the sample beam mirror block. This mirrors are mounted on a vertical sliding mechanism that is fixed to the knob on top of the mirror block. By pulling this knob upwards (see Fig. 10), the alignment mirrors are shifted into the beam path, steering the laser beam through the mirror block.
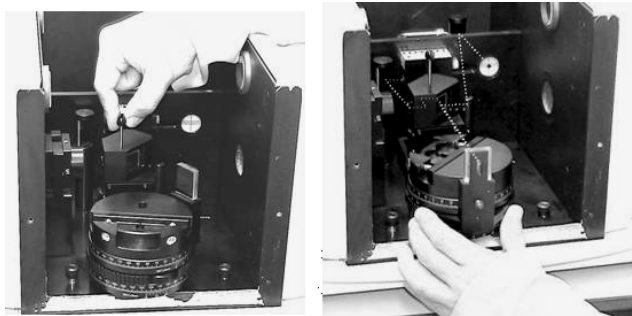

Fig. 10. Using the build-in laser to align the IV accessory. The dotted line in the right photograph shows the optical path of the laser beam.

2.4 Small Spot IV accessory

Our regular IV accessory can easily handle sample as small as 25 mm diameter. This is quite an improvement over the VW accessory, which has two seperated measurement spots on the sample, roughly 25 mm apart.

Nevertheless, some of the laser mirrors on the market have diameters as small as 10 mm. For these products we developed a Small Spot version of the IV accessory. This accessory (see Fig. 11) has the means to adjust the spot size down to 2 x 3 mm. The downside is that due to the reduced intensity we need longer integration times for the detector so measurements require more time.
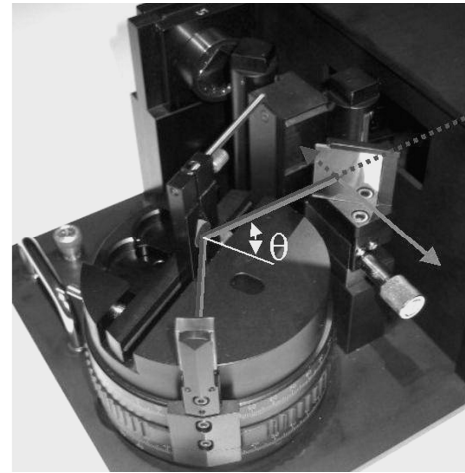

Fig. 11. Small Spot IV accessory with a small laser mirror installed.

The design did not allow for a laser alignment so we optimised the optics to reduce its sensitivity for misalignment. This is obtained mainly by choosing a retro-reflecting mirror (the one connected to the lower rotation stage) with a radius equal to its distance from the axis of rotation.

A comparison of the sizes of the measurement spots for near-normal and 45° incidence is shown for all three accessories in Fig. 12
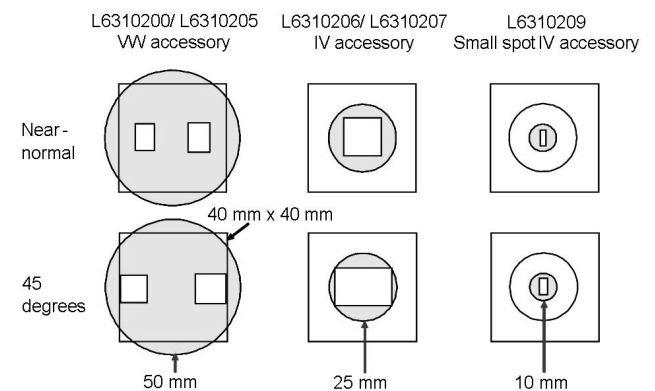

Fig. 12. Spot size comparison of the three accessories

3. Evaluation of measurement uncertainty

3.1 Measurement procedure

Measurements are performed in the sequence

1. 0% (stray light) measurement
2. 100% reference measurement
3. sample measurement 1 (' + ' angle)
4. sample measurement 2 (' - ' angle)
5. 100% reference measurement
6. sample measurement 3 (' + ' angle)
7. sample measurement 4 (' - ' angle)
8. 100% reference measurement
9. sample measurement 5 (' + ' angle)
10. sample measurement 6 (' - ' angle)
11. 100% reference measurement
12. 0% (stray light) measurement

All measurements are performed for P and S-polarisation separately (by repeating each scan after switching the polariser position), resulting in 24 scans per sample. As an example we will discuss the procedure for the IV accessory. This procedure is the same for all three accessories.

The six reflectance values corresponding to each of the V-mode measurements are determined for P and S-polarisation separately, according to:

$$R_i = \sqrt{\frac{2M_{V,i} - M_{0,1} - M_{0,2}}{M_{I,j} + M_{I,j+1} - M_{0,1} - M_{0,2}}} \qquad (2)$$

where $j = 0.5 \, (i + 1)$ for $i = 1, 3, 5$ ("positive" angles) $j = 0.5 \, i$ for $i = 2, 4, 6$ ("negative" angles).

The reflectance of the sample is determined by taking the average of these six values but for the evaluation of the calibration uncertainty we also need the averages of reflectance values obtained at positive and negative angles:

$$< R+ > = \tfrac{1}{3} \sum_{i=1,3,5} R_i \quad \text{and} \quad < R- > = \tfrac{1}{3} \sum_{i=2,4,6} R_i \qquad (3)$$

The procedures below are based on the ISO Guide to the Expression of Uncertainty in Measurement [7].

3.2 Measurement equation

The following measurement equation is valid for the reflection at near-normal incidence of an unknown sample:

$$R = \sqrt{f} \cdot < R > + \Delta_{NL} + \Delta_{WL} + \Delta_A + \Delta_P + \Delta_{NU} \qquad (4a)$$

in which

f is a factor that accounts for differences in alignment between V and W mode,
$<R>$ is the average of the measured reflectance values,
Δ_{NL} is a contribution that accounts for detector non-linearity,

Δ_{WL} is a contribution that accounts for a systematic deviation in the wavelength,
Δ_A is a contribution that accounts for a systematic deviation in the angle of incidence,
Δ_P is a contribution that accounts for a systematic deviation in the polarisation,
Δ_{NU} is a contribution that accounts for a systematic deviation due to sample non-uniformity.

In case of oblique incidence, the measurement equation is

$$R = \sqrt{f} \cdot \left(\tfrac{1}{2} < R+ > + \tfrac{1}{2} < R- > \right) + \\ + \Delta_{NL} + \Delta_{WL} + \Delta_A + \Delta_P + \Delta_{NU} \qquad (4b)$$

in which $<R+>$ and $<R->$ are the averages of the measured reflectance values at positive and negative angles respectively.

3.3 Misalignment

The alignment factor f in (Eq. 4) has a value of one (1) with a standard uncertainty $u_f < 0.001$ (conservative value based on experience). The uncertainty in the reflectance due to misalignment is proportional to u_f according to

$$u_M = \tfrac{1}{2} < R >^2 u_f \qquad . \qquad (5)$$

3.4 Standard uncertainty in the reflectance

The average reflectance values R+ and R- are estimated from 3 independent observations Ri according to (3). The standard uncertainties associated with these observations are the estimated standard deviations (of the mean) according to:

$$u_{R+} = 1.32 \cdot \sqrt{\tfrac{1}{2} \sum_{i=1,3,5} \frac{\left(R_i - < R+ > \right)^2}{3}} \qquad (6a)$$

and

$$u_{R-} = 1.32 \cdot \sqrt{\tfrac{1}{2} \sum_{i=2,4,6} \frac{\left(R_i - < R- > \right)^2}{3}} \qquad (6b)$$

In which the factor 1.32 is the Student-t factor for 2 degrees of freedom and a 68.27% confidence level (1 sigma).

3.5 Detector non-linearity

The detector non-linearity produces a systematic uncertainty component that in principle can be corrected. This requires a thorough investigation of the instrument in use. We found that, when a measurement is made with a background correction (values between 0% and 100%), the non-linearity error of our Lambda 900 spectrophotometer approximates very well the function

$$\Delta_{NL} = C(1 - M)M \qquad (7)$$

where M is the measurement value (between 0 and 1) and C a constant that is wavelength dependent. According to

this equation, the non-linearity error is zero at 0% and 100% and has its maximum at M = 0.5 (50%). The constant C in Eq. 7 can be determined using the Double Aperture Method [8].

If we don't make a non-linearity correction, we take Δ_{NL} = 0 in Eq. 4. The standard uncertainty associated with Δ_{NL} is

$$u_{NL} = \tfrac{1}{4}C(1-R)R \qquad . \qquad (8)$$

We can obtain a safe estimate for C by using the limits of the photometric accuracy of the Lambda 900 according to specifications and experience. Using conservative values, we find C = 0.008 for the UV/Vis range (photometric accuracy = 0.2%) and C = 0.012 for the NIR range (photometric accuracy = 0.3%).

The factor ¼ is a correction for the fact that we measure R^2 and for the coverage factor of 2 that is assumed to be associated with the specified photometric accuracy:

3.6 Wavelength uncertainty

The correction for a systematic deviation in the wavelength Δ_{WL} is assumed to be zero with a standard uncertainty that can be estimated using wavelength standards.

Unless the measured spectrum is flat, the uncertainty in the wavelength will yield an uncertainty in the ordinate. Taking into account that we measure the square of the reflectance, the standard uncertainty due to this effect is given by:

$$u_{WL} = \tfrac{1}{2}\left|\frac{\partial R}{\partial \lambda}\right| u_\lambda \approx \tfrac{1}{2}\left|\frac{\Delta R}{\Delta \lambda}\right| u_\lambda \qquad (9)$$

Typical values for the standard uncertainty in the wavelength scale of the Lambda 900 are u_λ = 0.1 nm in the UV/Vis range and u_λ = 0.15 nm in the NIR range.

3.7 Angular uncertainty

There is a systematic deviation (off-set) in the angle of the rotation stage. This error is corrected for by taking for by taking the average for positive and negative angles of incidence. The standard uncertainty in the reflection that relates to this error is estimated by

$$u_{A,ROT} = \tfrac{1}{2\sqrt{3}}\left|<R+>-<R->\right| \qquad (10)$$

3.8 Uncertainty in the polarisation

At oblique incidence, polarisation becomes more important and we have to take into a deviation φ (< 1°) in the angle of the polariser. This results in an uncertainty in the measured reflectance, which is estimated by

$$u_P = \frac{\sin(\varphi)}{\cos(\varphi)+\sin(\varphi)}\frac{\left|R_S - R_P\right|}{2\sqrt{3}} \qquad (11)$$

3.9 Sample nonuniformity

The sample nonuniformity is expressed in a standard uncertainty u_{NU} of which the best estimate is given by the standard deviation of the mean of N (>2) measurements on different positions on the sample, multiplied by the Student-t factor for N-1 degrees of freedom and a 68.27% confidence level (1 sigma).

3.10 Combined standard uncertainty

The combined standard uncertainty in the measured reflectance is obtained by taking the square root of the sum of squared standard uncertainties [7]. Typical values for the various standard uncertainties and the combined uncertainty are shown for S-polarisation in Fig 12. For P-polarisation the results are similar.
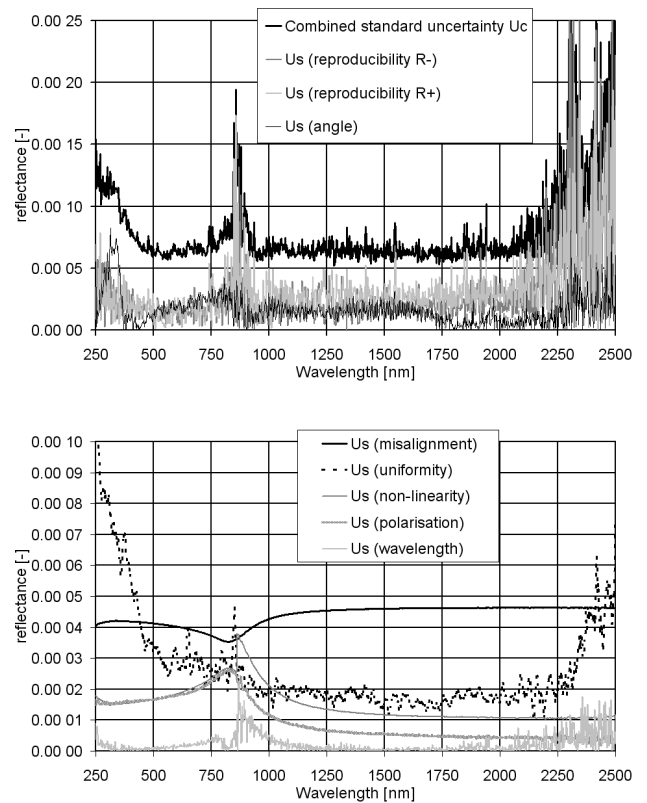

Fig. 13. Standard uncertainty components determined for the spectral reflectance for S-polarization of Fig. 4.

3.10 Expanded uncertainty

The expanded uncertainty U provides an interval R-U to R+U about the result R within which the value of R can be asserted with a high level of confidence.

The expanded uncertainty is determined by multiplying the combined standard uncertainty u_C with a coverage factor k (for which commonly a value k=2 is chosen).

4. Conclusion

We developed a new generation accessories for measuring absolute reflectance of highly reflecting samples. These accessories are capable of measuring reflectance at near-normal as well as oblique incidence for P and S-polarised

radiation separately, at an accuracy level compatible with that of metrology laboratories.

The accessories measure the square of the reflectance and are intended for flat specular samples having reflectance values > 20% (reflectance standards, laser mirrors, optical solar reflectors, beam-splitters, etc).

The expanded uncertainty which can be obtained on reference mirrors with a reflectance > 80% using our VW accessory is typically < 0.2% in the visible range and < 0.4% in the UV and NIR ranges for at angles up to 75°. These calibrations are performed on a regular basis to provide reference mirrors for one of our other tools [9].

With our IV accessories we have been able to measure the reflection of laser mirrors, even at oblique incidence, with expanded uncertainties as low as 0.05%.

For the calibration of metallic reference mirrors the VW accessory offers a faster measurement as compared with the IV accessories, which require longer integration times due to the reduction of the beam intensity by the beam-splitter.

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