

<b>OMT Solutions BV</b> <i>Optical Measurements and Testing</i> <i>Materials for optical applications</i> <i>Thin Film Analysis</i>	DOC. NO. :
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# Calibration report

**Direct reflectance at 8° incidence in the wavelength  
range 220 nm – 2,500 nm of XXXXX-XX  
(50 mm × 50 mm second surface mirror)**

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# 1. Introduction

## 1.1 Applicable documents

- AD1 OMT Solutions, Quotation OFF-209059-04, 13.07.2009.
- AD2 Guide to the Expression of Uncertainty in Measurement, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland (1993).
- AD3 Mielenz, K.D. and Eckerle, K.L., Spectrophotometer at the National Physical Laboratory, J. Res. Of the National Bureau of Standards – A. Physics and Chemistry, Vol. 76A, 1972.
- AD4 Nijnatten, P.A. van, Calibration of neutral density glass filters to produce transmittance standards, 5th ESG Conference “Glass Science and Technology for the 21st Century”, Prague, 1999. AD3

## 1.2 Details

The sample is a 50 mm × 50 mm second surface mirror with label “SSM-XXXXX-XX”.

The sample was calibrated at 8° incidence for wavelengths in the range 220 nm – 2,500 nm using a PerkinElmer Lambda 900 UV/Vis/NIR spectrophotometer equipped with a VW reflectometer and an integrating sphere unit as detector.

Date of calibration: XXXX

Person performing the calibration: XXXXX

This report gives a detailed description of the calibration procedure and evaluation of the calibration uncertainty.

## 2. Measurements

### 2.1 Equipment and conditions

Measurements are performed using a Perkin Elmer Lambda 900 UV/Vis/NIR spectrophotometer equipped with a 150 mm InGaAs RSA assy integrating sphere detector and the L631 200 Directional VW absolute reflectometer.

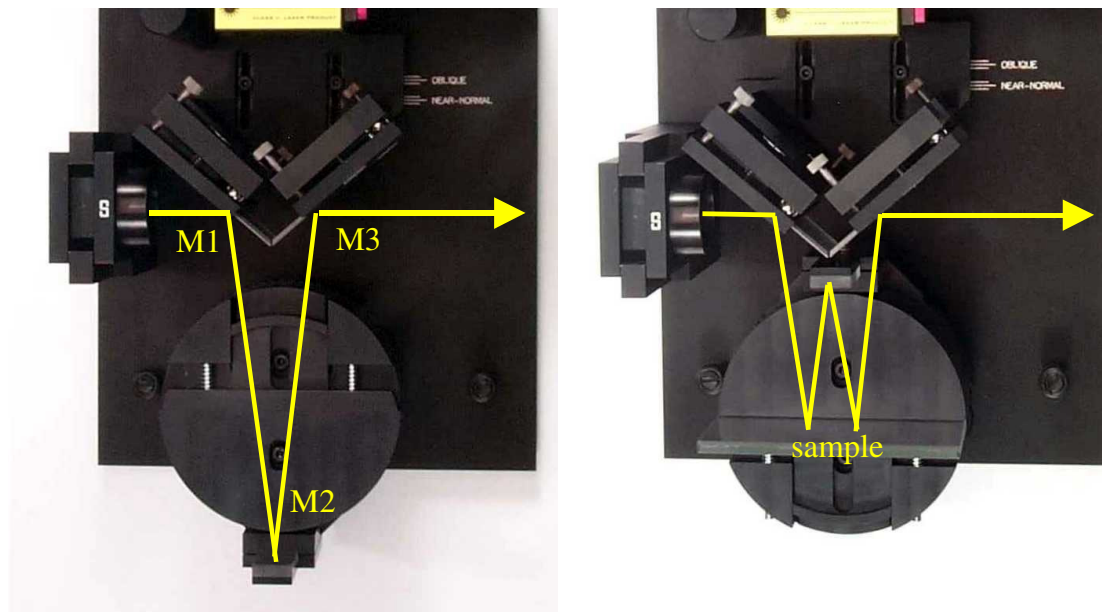
The following slit program was used:

- 5 nm slit in the wavelength range from 220 nm – 860.6 nm,
- “Servo” in the wavelength range from 860.6 nm - 2,500 nm.

The sample temperature during the measurements was  $21 \pm 1$  °C.

### 2.2 Measurement principle

The measurement principle of the VW absolute reflectance accessory is based on a combination of two measurements. In the so-called V-mode the instrument beam is interacting with three mirrors (M1 - M3). In the so-called W-mode the beam additionally interacts twice with the sample. The ratio of the two scans produces the square of the sample reflectance. This method is an absolute one since a calibrated reference is not needed



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### **2.3 Cleaning procedure before calibration**

Before calibrating the mirror was cleaned using Berkshire POLX1200 tissues and isopropyl alcohol after which the mirror is blow-dried with a stream of clean air.

### **2.4 Measurement sequence**

The following measurement sequence of scans is applied :

1. 0% (stray light) measurement
2. 100% measurement, V-mode
3. Sample measurement 1 (near normal), W-mode
4. Sample measurement 2 (near normal), W-mode (after repositioning the sample)
5. 100% measurement, V-mode
6. Sample measurement 3 (near normal), W-mode
7. Sample measurement 4 (near normal), W-mode (after repositioning the sample)
8. 100% measurement, V-mode
9. Sample measurement 5 (near normal), W-mode
10. Sample measurement 6 (near normal), W-mode, (after repositioning the sample)
11. 100% measurement, V-mode
12. 0% (stray light) measurement

## 2.5 Calculations

The measurement sequence results in the series  $M_{0,1}$ ,  $M_{V,1}$ ,  $M_{W,1}$ ,  $M_{W,2}$ ,  $M_{V,2}$ ,  $M_{W,3}$ ,  $M_{W,4}$ ,  $M_{V,3}$ ,  $M_{W,5}$ ,  $M_{W,6}$ ,  $M_{V,4}$ ,  $M_{0,2}$ , from which 6 reflectance values corresponding to each of the W-mode measurements are determined, according to:

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

where  $j = 0.5 (i + 1)$  for  $i = 1, 3, 5$  and  $j = 0.5 i$  for  $i = 2, 4, 6$ .

The reflectance of the sample is determined by taking the average of these six values.

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### 3. Uncertainty analysis

#### 3.1 Evaluation and Expression of Uncertainty

The procedures below are based on AD2.

The following measurement equation is valid for the reflection at near-normal incidence ( $8^\circ$ ) of an unknown sample:

$$R = \sqrt{f} \cdot \langle R \rangle + \Delta_{NL} + \Delta_{WL} + \Delta_A + \Delta_P + \Delta_{NU} \quad (2)$$

in which

$f$  is a factor that accounts for differences in alignment between V and W mode

$\langle R \rangle$  is the average of the measured reflectance values,

$\Delta_{NL}$  is a contribution that accounts for detector non-linearity

$\Delta_{WL}$  is a contribution that accounts for a systematic deviation in the wavelength

$\Delta_A$  is a contribution that accounts for a systematic deviation in the angle of incidence

$\Delta_P$  is a contribution that accounts for a systematic deviation in the polarisation

$\Delta_{NU}$  is a contribution that accounts for a systematic deviation due to sample non-uniformity

#### 3.2 Misalignment

The alignment factor  $f$  in Eq.(2) has a value of one 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

$$u_M = \frac{1}{2} \langle R \rangle^2 u_f \quad (3)$$

#### 3.3 Standard uncertainty in the reflectance

The average reflectance values are estimated from 6 independent observations  $R_i$  according to Eq.(1). The standard uncertainty associated with these observations is the estimated standard deviation (of the mean) according to:

$$u_R = 1.11 \cdot \sqrt{\frac{1}{5} \sum_{i=1,3,5} \frac{(R_i - \langle R \rangle)^2}{6}} \quad (4)$$

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In which the factor 1.11 is the Student-t factor for 5 degrees of freedom and a 68.27% confidence level (1 sigma).

### 3.4 *Detector non-linearity*

The detector non-linearity produces a systematic uncertainty component that in principle can be corrected (see AD3 and AD4). This requires a thorough investigation of the instrument in use.

Our research (see AD4) has shown that, when a measurement is made with a background correction (values between 0% and 100%), the non-linearity error of the Lambda 900 approximates the function

$$\Delta_{NL} = C(1 - M)M \tag{5}$$

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 (6) can be determined using the Double Aperture Method as described in AD4.

If we don't make a non-linearity correction, we chose  $\Delta_{NL} = 0$  in equation (3). The standard uncertainty associated with  $\Delta_{NL}$  is then

$$u_{NL} = \frac{1}{4}C(1 - R)R \tag{6}$$

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:

In the UV/Vis range (photometric accuracy = 0.2%) → C = 0.008

In the NIR range (photometric accuracy = 0.3%) → C = 0.012

The factor  $\frac{1}{4}$  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:

Given the reflectance range in which the VW accessory operates, the non-linearity errors according to these specifications and Eq. (6), follow the curves shown in Fig. 3.1 below.



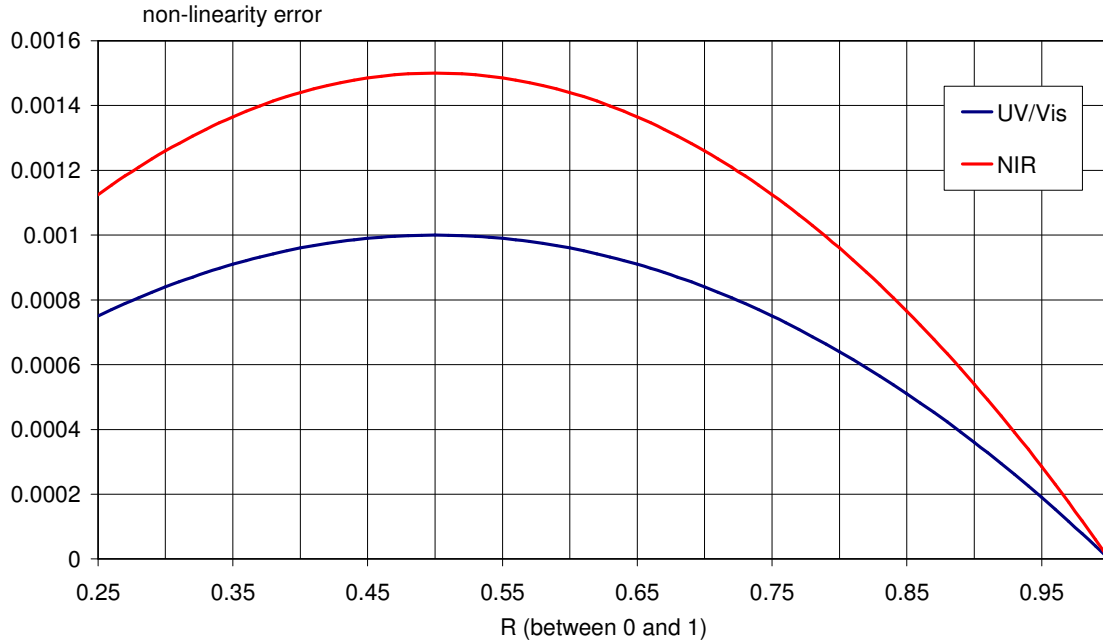


Figure 3.1 Worst-case detector nonlinearity (2 x standard uncertainty)

### 3.5 Wavelength uncertainty

The correction for a systematic deviation in the wavelength  $\Delta_{WL}$  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:

$$u_{WL} = \frac{1}{2} \left| \frac{\partial R}{\partial \lambda} \right| u_{\lambda} \approx \frac{1}{2} \left| \frac{\Delta R}{\Delta \lambda} \right| u_{\lambda} \quad (7)$$

Typical values for the standard uncertainty in the wavelength scale of the Lambda 900 are:

- In the UV/Vis range                      →  $u_{\lambda} = 0.1 \text{ nm}$
- In the NIR range                            →  $u_{\lambda} = 0.15 \text{ nm}$

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### 3.6 Angular uncertainty

The standard uncertainty due to the uncertainty in the angle of incidence is assumed to be negligible!

### 3.7 Uncertainty in the polarisation

The deviation  $\varphi$  in the angle of the polarizer is  $< 1^\circ$ . This results in an uncertainty in the measured reflectance, which is estimated by

$$u_p = \frac{\sin(\varphi)}{\cos(\varphi) + \sin(\varphi)} \frac{|R_s - R_p|}{2\sqrt{3}} \quad (8)$$

In our case ( $8^\circ$  incidence)  $u_p$  is assumed to be negligible!

### 3.8 Sample non-uniformity

The sample non-uniformity is based on a separate VW measurement sequence of 8 measurements containing 5 measurements on different positions on the sample:

1. 0% (stray light) measurement
2. 100% measurement, V-mode
3. Sample measurement position 1 (near normal), W-mode
4. Sample measurement position 2 (near normal), W-mode
5. Sample measurement position 3 (near normal), W-mode
6. Sample measurement position 4 (near normal), W-mode
7. Sample measurement position 5 (near normal), W-mode
8. 100% measurement, V-mode

The sample non-uniformity associated with these observations is the estimated deviation (of the mean) according to:

$$u_{NU} = 1.14 \cdot \sqrt{\frac{\sum_{i=1}^N (R_i - \langle R \rangle)^2}{N \cdot (N - 1)}} \quad (9)$$

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In which the factor 1.14 is the Student-t factor for 4 degrees of freedom and a 68.27% confidence level (1 sigma).

### **3.9 Combined standard uncertainty**

The combined standard uncertainty in the measured reflectance is obtained according to

$$u_c = \sqrt{(u_R)^2 + (u_M)^2 + (u_{NL})^2 + (u_{WL})^2 + (u_{NU})^2} \quad (10)$$

### **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  $u_c$  of Eq. (10) with a coverage factor k (for which commonly a value  $k = 2$  is chosen).

## 4 Conclusion

### 4.1 Calibration results

The calibration results of the reflectance standard “SSM-XXXXX-XX” which has been calibrated in the wavelength range from 220 nm to 2,500 nm at an angle of incidence of 8° are shown in Figs. 4.1 and 4.2 below.

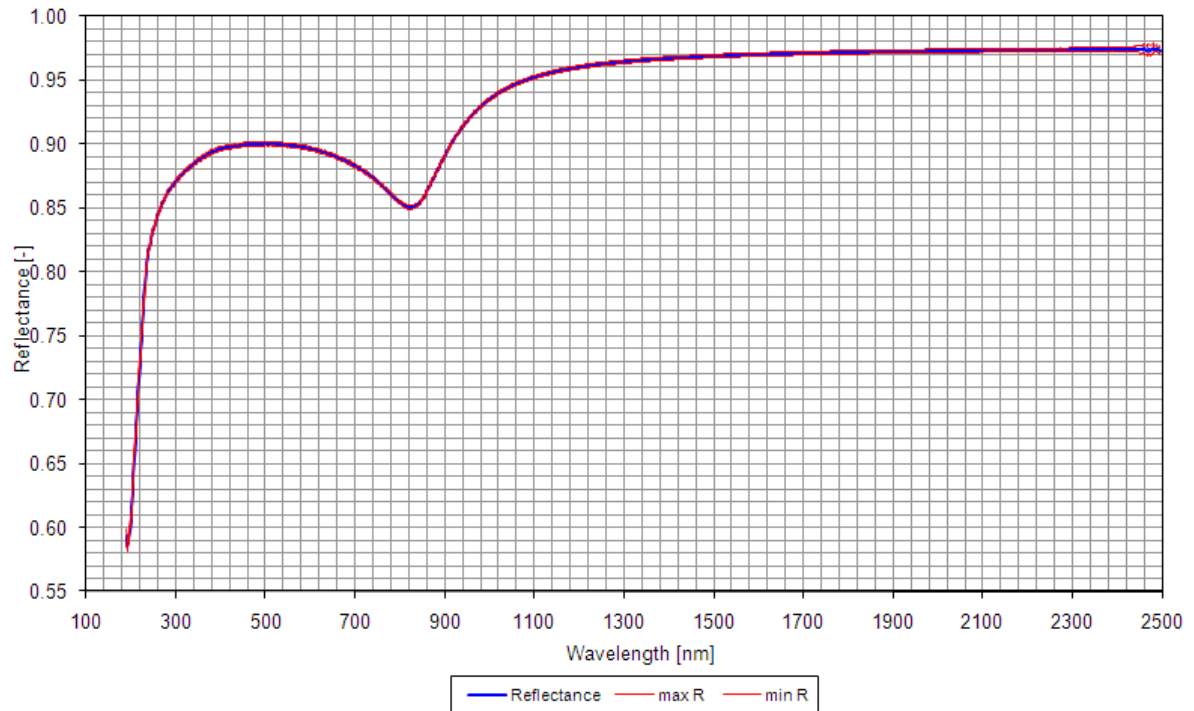


Figure 4.1 Near-normal (8°) Reflectance of the mirror.

Expanded Uncertainty, coverage factor  $k = 2$

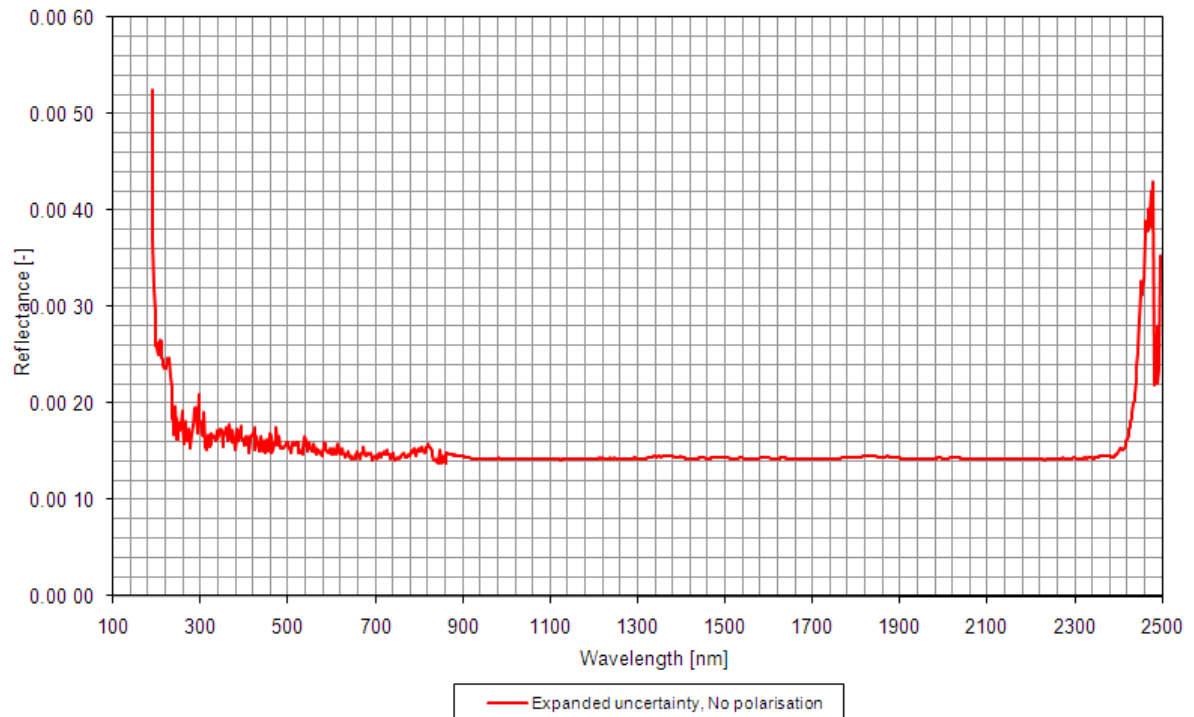


Figure 4.2 Expanded uncertainty, coverage factor  $k = 2$ .

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## 5 Authorization

	Name	Signature
Calibration performed by		
Checked by		