

Calibration report

Calibration of Front Surface Gold Mirror no. XXXXX-XX:

Direct reflectance at 10° incidence in the wavenumber range $200 \text{ cm}^{-1} - 4,000 \text{ cm}^{-1}$

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1. Introduction

1.1 Details

The sample is a 50 mm x 50 mm front surface gold reference mirror no. XXXXX-XX.

The sample was calibrated at 10° incidence for wavenumbers in the range $200 \text{ cm}^{-1} - 4,000 \text{ cm}^{-1}$ using a PerkinElmer 983 IR spectrophotometer equipped with a reflectometer.

Date of calibration:

Person performing the calibration:

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

1.2 Applicable documents

AD1

- 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 NPL Calibration certificate for OM&T-IR-17025 gold reference mirror, NPL164/05

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2. Measurements

2.1 Equipment and conditions

Measurements have been performed in the wavenumber range 200 cm⁻¹ - 4,000 cm⁻¹ with intervals of 1 cm⁻¹ using a Perkin Elmer 983 Infrared Spectrophotometer equipped with a specular reflectance accessory.

The angle of incidence is approximately 10° and the illuminated sample area is approximately $7 \times 12 \text{ mm}^2$. The sample temperature during the measurement was 25 ± 5 °C.

2.2 Measurement sequence

Three types of readings were taken in a time sequence of the following 15 scans:

- 1. M_{D,1} : 0% (stray light) measurement 1
- 2. M_{R,1}: Reference mirror measurement 1
- 3. M_{s,1}: Sample mirror measurement 1
- 4. M_{R,1}: Reference mirror measurement 2
- 5. M_{S,1}: Sample mirror measurement 2
- 6. M_{R,1}: Reference mirror measurement 3
- 7. M_{S,1}: Sample mirror measurement 3
- 8. M_{R,1}: Reference mirror measurement 4
- 9. M_{S,1}: Sample mirror measurement 4
- 10. M_{R,1}: Reference mirror measurement 5
- 11. M_{s,1}: Sample mirror measurement 5
- 12. M_{R,1}: Reference mirror measurement 6
- 13. M_{S,1}: Sample mirror measurement 6
- 14. M_{R,1}: Reference mirror measurement 7
- 15. M_{D,2} : 0% (stray light) measurement 2

2.3 Calculations

The reflectance is calculated for N=6 measurements as follows:

$$R_{s,i} = \frac{M_{s,i} - \frac{1}{2}(M_{D,1} + M_{D,2})}{\frac{1}{2}(M_{R,i} + M_{R,i+1}) - \frac{1}{2}(M_{D,1} + M_{D,2})} R_{REF} ,$$
(1)

In which i = 1 to N refers to the number of sample mirror measurement and R_{REF} is the reflectance of our primary standard OM&T-IR-17025, NPL164/05 [AD4].

The reflectance of the sample mirror 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°) of an unknown sample mirror:

$$\mathbf{R} = \mathbf{f}_{\mathrm{M}} \cdot \mathbf{f}_{\mathrm{D}} \cdot \mathbf{f}_{\mathrm{P}} \cdot \mathbf{f}_{\mathrm{A}} \cdot \mathbf{f}_{\mathrm{S}} < \mathbf{R} > +\Delta_{\mathrm{W}} \qquad , \tag{2}$$

in which

 f_{M} is a mis-alignment factor that accounts for differences in alignment between the sample mirror and the reference mirror

 $f_{\mbox{\scriptsize D}}$ is a factor that accounts for detector non-linearity

 f_{P} is a factor that accounts for the effect of polarization

 $f_{\text{A}}\xspace$ is a factor that accounts for a systematic deviation in the angle of incidence

 $f_{S}\xspace$ is a scale factor that accounts for the calibration of the reflectance scale

<R> is the average of the measured reflectance values,

 Δ_W is a contribution that accounts for the systematic deviation in the wavenumber scale

3.2 Misalignment

The alignment factor f_M in Eq.(2) has a value of one with an estimated standard uncertainty $u_{f,M} < 0.0005$ (conservative value based on experience). The uncertainty in the reflectance due to misalignment is proportional to $u_{f,M}$ according to

$$u_{\rm M} = < R > u_{\rm f,M}$$
 ,

(3)

3.3 Detector non-linearity

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

We have investigated the detector non-linearity of our PE 983 IR spectrophotometer using the Double Aperture Method described in AD3 and found the effect to be < 0.0002 for most of the wavenumber range, as shown in Figure 3.1 below. Since the investigated sample mirror and our reference mirror have reflectance values that are very close, we may safely assume that $f_D = 1$ with a negligible uncertainty!

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3.4 Uncertainty in the polarisation

Measurements have been performed without a polarizer. For Aluminium front surface mirrors at 10° incidence the difference between the reflectance at respectively p and s polarization is < 0.001. Assuming that the degree of polarization of the beam is < 80% over the measured wave number range (estimate based on previous measurements with a polarizer), the factor $f_P = 1.0000$ with a standard uncertainty of $u_{f,P} = 0.0002$.

The standard uncertainty in the measured reflectance due to this effect is

 $u_{\rm P} = < R > u_{\rm f,P} \qquad . \tag{4}$

3.5 Angular uncertainty

The average angle of incidence of our beam is $10^{\circ} \pm 2^{\circ}$ which in the case of a metallic mirror with reflectance > 0.95 results in a maximum standard uncertainty < 10^{-5} . Therefore, we may assume that $f_A = 1$ with a negligible uncertainty!

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3.6 Standard uncertainty associated with the scale factor

The scale factor $f_s = 1$ with standard uncertainty u_{REF}/R_{REF} where u_{REF} is the standard calibration uncertainty of the reference mirror with reflectance R_{REF} . The standard uncertainty in the reflectance of the calibrated sample mirror associated with the scale factor is:

$$u_{s} = \langle R \rangle \frac{u_{REF}}{R_{REF}}$$
(5)

3.7 Standard uncertainty in the reflectance measurement

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:

$$u_{R} = 1.11 \cdot \sqrt{\frac{1}{5} \sum_{i=1,3,5} \frac{\left(R_{i} - \langle R + \rangle\right)^{2}}{6}}$$
(6)

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

3.8 Wavenumber uncertainty

The correction Δ_W for the systematic deviation in the wavenumber setting is assumed to be zero with a standard uncertainty u_W .

Unless the measured spectrum is flat, the uncertainty in the wavenumber will yield an uncertainty in the Reflectance. The standard uncertainty due to this effect is given by:

$$\mathbf{u}_{W} = \left| \frac{\partial \mathbf{R}}{\partial \boldsymbol{\omega}} \right| \mathbf{u}_{\boldsymbol{\omega}} \approx \left| \frac{\Delta \mathbf{R}}{\Delta \boldsymbol{\omega}} \right| \mathbf{u}_{\boldsymbol{\omega}}$$
(7)

In the case of the PE 983 IR spectrophotometer, we determined the standard wavenumber uncertainty to $u_{\omega} = 1 \text{ cm}^{-1}$.

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3.9 Combined standard uncertainty

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

$$u_{\rm C} = \sqrt{(u_{\rm M})^2 + (u_{\rm P})^2 + (u_{\rm S})^2 + (u_{\rm R})^2 + (u_{\rm W})^2}$$
(8)

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. (8) with a coverage factor k (for which commonly a value k = 2 is chosen).

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4 Conclusion

4.1 Calibration results

The calibration results of the gold mirror are shown in Fig. 4.1 below. A table of the Reflectance and Expanded Uncertainty with a coverage factor k = 2 is given in the Excel file.

4.2 Using the calibrated mirror

If the mirror gets contaminated or damaged, the calibration values may become invalid. Contaminations may be removed by cleaning in a bath of isopropyl alcohol after which the mirror is blow-dried with a stream of clean air. However, this does not guarantee that its condition after cleaning is the same as during our calibration!



Figure 4.1 Near-normal (10°) Reflectance and calibration uncertainty of the Gold Mirror

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5 Authorization

	Name	Signature
Calibration performed by		
Authorized by		