



Guidelines

METHOD TO EVALUATE THE REFLECTANCE, ABSORPTANCE AND EMMITTANCE OF PARTICLES FOR CONCENTRATING SOLAR POWER TECHNOLOGY



Version 1

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1 Foreword

SolarPACES is an international cooperative network bringing together teams of national experts from around the world to focus on the development and marketing of concentrating solar power (CSP) systems.

The creation of this guideline has been funded by SolarPACES within the Task III group. The funded project was entitled “Characterization of optical properties of particles for CSP” and was carried out from November 2021 to October 2022. The funded institutes were DLR, CIEMAT, ENEA, Universitat Barcelona, NREL and SANDIA.

The guideline is open to amendments and updating as the state-of-the-art advances. Please send general comments, amendments or suggestions to florian.sutter@dlr.de. Questions or suggestions regarding the optical model and correction functions depicted in chapter 4 can be sent to marco.montecchi@enea.it.

2 Symbols

| | |
|--------------------------|--|
| $\langle \rho_w \rangle$ | Window reflectance averaged over the reflectance angle of the specimen behind the window |
| $\langle \tau_w \rangle$ | Window transmittance averaged over the reflectance angle of the specimen behind the window |
| A_s | Near-normal hemispherical spectral absorptance of the specimen (=the particle layer) |
| $A_{s,h}$ | Solar-weighted hemispherical absorptance of the specimen (=the particle layer) |
| c | Speed of light in vacuum, $2.997 \cdot 10^8$ m/s |
| E_s | Thermal emittance of the specimen (=the particle layer) |
| h | Planck constant, $6.63 \cdot 10^{-34}$ Js |
| $I_{\#1}$ | Detector intensity for the reference coupon #1 |
| $I_{\#2}$ | Detector intensity for the reference coupon #2 |
| I_w | Detector intensity for the window |
| $I_{w,\#1}$ | Detector intensity for the reference coupon #1 measured behind the window |
| $I_{w,\#2}$ | Detector intensity for the reference coupon #2 measured behind the window |
| $I_{w,s}$ | Detector intensity for the specimen (=the particle layer) measured behind the window |
| I_z | Detector intensity for the zeroline |
| k | Boltzmann constant, $1.38 \cdot 10^{-23}$ J/K |
| M_{bb} | Blackbody spectral emittance |
| $R_{\#1}$ | Certified near-normal hemispherical spectral reflectance of reference coupon #1 |
| $R_{\#2}$ | Certified near-normal hemispherical spectral reflectance of reference coupon #2 |
| R_s | Near-normal hemispherical spectral reflectance of the specimen (=the particle layer) |
| $R_{s,h}$ | Solar-weighted hemispherical reflectance of the specimen (=the particle layer) |
| T | Particle temperature |
| θ_0 | Near-normal incidence angle |
| ρ_w | Near-normal hemispherical reflectance of the window |
| $\rho_{w,\#1}$ | Near-normal hemispherical reflectance of reference coupon #1 measured through the window |
| $\rho_{w,\#2}$ | Near-normal hemispherical reflectance of reference coupon #2 measured through the window |
| $\rho_{w,s}$ | Near-normal hemispherical reflectance of the specimen (= the particle layer) measured through the window |
| τ_w | Near-normal hemispherical transmittance of the window |

3 Scope

Particles can be used in CSP applications as solid heat transfer and storage medium, heating them directly e.g. in a falling particle receiver. Since the particles interact directly with solar radiation, it is relevant to measure their optical properties accurately. This guideline details the measurement process to determine solar absorptance and thermal emittance of the particle layer.

The optical measurement of particles is quite challenging because spectrophotometers usually require to place the specimen in vertical or downfacing position. The most common trick is to pour particles in a sample holder, of which one face is made of a transparent material, like glass, fused-silica, ZnSe, etc. Of course, the measured reflectance will be a combination of window and particle-reflectance, and the experimental results must be properly treated to get the intrinsic absorptance or emittance of the particle-layer.

In chapter 4 the optical model of the window-particle system is outlined, from which correction formulas were obtained for different scenarios. The classical approach (section 4.2) consists of determining the complex refractive index of the window and averaging its predicted reflectance and transmittance over a Lambertian incidence angle distribution. For the ease of application, an alternative method is described (section 4.4), which requires two certified reference coupons of different reflectance characteristics. For the special cases of low absorption windows (section 4.5) and windows with anti-reflective coatings (section 4.6) only one reference coupon is required.

While chapter 4 is intended to provide a background for the comprehensive understanding of the applied correction formulas, the more practical section of this guideline is detailed in chapter 5, in which the measurement protocol is described step by step for the UV/VIS/NIR and IR range.

4 Theoretical background of the window-particle optical model and correction functions

4.1 The optical model: incoherent bi-layer

The irregularity of the air-gap between particles and window (due to the morphology of the particle-layer surface) and the millimeter-size of the window thickness allows one to sum-up the multiple reflections among the interfaces of the system in incoherent manner (interference is not considered).

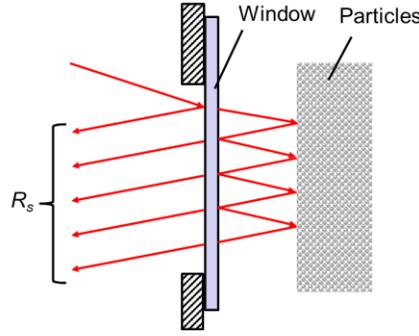


Figure 1: Illustration of particle measurement through a window

The window can be sketched as single interface having transmittance $\tau_w(\lambda, \theta)$ and reflectance $\rho_w(\lambda, \theta)$. Let us assume forward and back reflectance of the window were identical, thanks to the symmetry of a bare substrate.

With these premises, the window-particles system can be sketched like a pure bi-layer. The same simple model is adopted for predicting the optical behavior of architectural double-glazing window; its main relationships are well established and reported in ISO 9050 [1].

With minor adaptations, from the double-glazing reflectance relationship we get

$$\rho_{w,s}(\lambda, \theta_0) = \rho_w(\lambda, \theta_0) + \frac{\tau_w(\lambda, \theta_0) \langle \tau_w(\lambda, \theta) \rangle R_s(\lambda)}{1 - \langle \rho_w(\lambda, \theta) \rangle R_s(\lambda)} \quad (1)$$

Note that because of $T_s(\lambda) = 0$, the absorptance of the particle-layer is simply given by $A_s(\lambda) = 1 - R_s(\lambda)$.

Equation 1 can be rearranged to express the particle reflectance R_s as a function of the window and the window-particle bi-layer

$$R_s = \frac{\rho_{w,s} - \rho_w}{\tau_w \langle \tau_w \rangle + \langle \rho_w \rangle (\rho_{w,s} - \rho_w)} \quad (2)$$

where the arguments of the functions are omitted for the sake of readability.

All terms at the right side of Eq. 2 are easily experimentally measurable except for the two ones in the angular average brackets $\langle \rangle$: their direct determination would require a quite cumbersome procedure such as the experimental measurement of $\tau_w(\lambda, \theta)$ and $\rho_w(\lambda, \theta)$ spectra over the solar wavelength range (320,2500) nm and the incidence angle range $(0, \pi/2)$ (see Method #1, chapter 4.2).

Alternatively, one can follow one of the simpler methods reported in chapter 4.4, 4.5 or 4.6.

4.2 Method #0: flat angular behavior

The simplest approach is to assume $\langle \tau_w \rangle \approx \tau_w$ and $\langle \rho_w \rangle \approx \rho_w$, so that Eq. 2 becomes

$$R_s = \frac{\rho_{w,s} - \rho_w}{\tau_w^2 + \rho_w(\rho_{w,s} - \rho_w)} \quad (3)$$

where all the terms are directly measurable.

4.3 Method #1: optical characterization of the window

As general rule, any optical feature of an optical device can be predicted on the basis of its exhaustive optical characterization. When the window consists of a bare substrate, the main parameters are the complex refractive index of the composing material and its thickness.

The thickness can be easily measured with a caliper.

The complex refractive index $n - ik$ of the window material can be calculated from the transmittance and reflectance spectra measured at near normal incidence by means of the relationships outlined by Nichelatti [1].

Hence, the off-normal spectra of the window can be computed by the relationships reported in [3][4], paying attention to separately treat the case of the two polarizations s and p , to then consider their average value. More precisely spectra shall be numerically evaluated on a suitable set of incidence angles, then they should be weighted for the intensity angular-distribution of the particle reflectance. The latter is unknown; for simplicity, at least initially, we can assume the Lambertian distribution, i.e. $R(\theta) \propto \cos(\theta)$, thus

$$\langle M(\lambda, \theta) \rangle = \frac{1}{\mathcal{N}} \int_0^{2\pi} d\varphi \int_0^{\pi/2} \sin(\theta) \cos(\theta) M(\lambda, \theta) d\theta = 2 \int_0^{\pi/2} \sin(\theta) \cos(\theta) M(\lambda, \theta) d\theta \quad (4)$$

being the normalization factor $\mathcal{N} = \pi$.

The Lambertian hypothesis should be confirmed by crosschecking the result with that got with another method among those here proposed, at least in the part of the wavelength region where the second method is valid. In the case of scarce agreement, one should repeat the window computing by considering $R(\theta) \propto \cos^\alpha \theta$ where α should be adjusted for optimizing the agreement.

Finally, if the window consists of a substrate with anti-reflection coatings, its optical characterization is more complex because one has to also determine complex refractive index and thicknesses of the thin films composing the AR treatment; that operation requires a good expertise in thin film optical characterization.

4.4 Method #2: measurements of a couple of reference-coupons

Let us apply Eq. 1 on a couple of reference-coupons with certified reflectance $R_{\#1}$ and $R_{\#2}$ (as an example the so called “white” and “gray” diffuse-reflectance references); by setting $x = \tau_w(\lambda, \theta_0) \langle \tau_w(\lambda, \theta) \rangle$ and $y = \langle \rho_w(\lambda, \theta) \rangle$, Eq. 1 becomes

$$\rho_{w,\#j} = \rho_w + \frac{x R_{\#j}}{1 - y R_{\#j}} \quad (5)$$

By considering the system composed by two references (Eq. 5 applied to $R_{\#1}$ and to $R_{\#2}$), it is easy to obtain the solutions

$$\begin{aligned} x &= \frac{\rho_{w,\#1} - \rho_w}{R_{\#1}} \left[1 - \frac{R_{\#1}}{\rho_{w,\#2} - \rho_{w,\#1}} \left(\frac{\rho_{w,\#2} - \rho_w}{R_{\#2}} - \frac{\rho_{w,\#1} - \rho_w}{R_{\#1}} \right) \right] \\ y &= \frac{1}{\rho_{w,\#2} - \rho_{w,\#1}} \left(\frac{\rho_{w,\#2} - \rho_w}{R_{\#2}} - \frac{\rho_{w,\#1} - \rho_w}{R_{\#1}} \right) \end{aligned} \quad (6)$$

To have a good accuracy, the two values $R_{\#1}$ and $R_{\#2}$ must be as different as possible. Please note that $x = x(\lambda)$ and $y = y(\lambda)$.

The correction function (Eq. 3) can now be rewritten as

$$R_s = \frac{\rho_{w,s} - \rho_w}{x + y(\rho_{w,s} - \rho_w)} \quad (7)$$

In conclusion, at the price of having to measure two different reference-coupons through the window, it is possible to obtain the correction function without any optical characterization of the window.

Method #2 is certainly simpler than method #1, but to be completely correct, the angular distribution of the diffuse reflectance of the two reference-coupons as well as the specimen should be quite similar to each other.

4.5 Method #3: for low absorbing windows

When the window absorption is much less than 1, the unknowns of Eq. 2 are reduced to one, being

$$\langle \rho_w \rangle \approx 1 - \langle \tau_w \rangle \quad (8)$$

Therefore, one just needs to measure a single reference coupon; Eq. 2 can be rewritten for the reference coupon with certified reflectance $R_{\#1}$ so that

$$\langle \tau_w \rangle = \frac{(\rho_{w,\#1} - \rho_w)(1 - R_{\#1})}{R_{\#1}(\tau_w + \rho_w - \rho_{w,\#1})} \quad (9)$$

where $\rho_{w,\#1}$ is the experimental hemispherical reflectance of the reference coupon when measured through the window, and ρ_w is the window reflectance measured with the integrating sphere.

Finally, the correction function is given by Eq. 2 by considering Eqs. 8 and 9.

4.6 Method #4: flat angular behavior of reflectance

In the case of low reflecting windows (typically for windows provided of anti-reflection coatings),

$$\langle \rho_w \rangle \approx \rho_w \quad (10)$$

By applying Eq. 2 to the case of the reference coupon $R_{\#1}$ one gets

$$\langle \tau_w \rangle = \frac{(1 - R_{\#1} \rho_w)(\rho_{w,\#1} - \rho_w)}{R_{\#1} \tau_w} \quad (11)$$

Finally, the correction function is given by Eq. 2 by considering Eqs. 10 and 11.

4.7 Method #5: flat angular behavior of transmittance

Just for completeness we also include the case where

$$\langle \tau_w \rangle \approx \tau_w \quad (12)$$

By applying Eq. 2 to the case of the reference coupon $R_{\#1}$ one gets

$$\langle \rho_w \rangle = \frac{\rho_{w,\#1} - \rho_w - R_{\#1} \tau_w^2}{R_{\#1} (\rho_{w,\#1} - \rho_w)} \quad (13)$$

Finally, the correction function is given by Eq. 2 by considering Eqs. 12 and 13.

4.8 Additional remarks

In case inconsistencies of the measurements arise, the following two points should be examined:

- ρ_w should be measured by means of a specular reference coupon because of the window specularly: integrating spheres are not perfect, thus it is recommended to use always a reference coupon with angular diffusion distribution similar to the specimen one. For the same reason, a diffuse reference coupon should be used to measure the particle film.
- If a thick window is used, in order to compensate the optical path in the window, the baseline used for any measurements through the window should be accomplished by inserting a spacer between the port of the integrating sphere and the reference coupon; the spacer thickness should be

$$d_{spacer} = d_{air-gap} + d_w / n_w \quad (14)$$

where

- $d_{air-gap}$ is the eventual air-gap between the first surface of the window and the port of the integrating sphere
- d_w the window thicknesses
- n_w the window refractive index (i.e. its mean value in the considered wavelength range)

For the IR range, measurements conducted within this group on a 2 mm thick ZnSe window with anti-reflection coating, it was shown that the influence of using a spacer is negligible for this specific window type. Moreover, it was shown that the measured particle reflectance R_s obtained by means of the simpler Method #4 flat angular behavior of reflectance (section 4.6) was identical to the one obtained by the Method #2 with 2 reference coupons (section 4.4).

For the UV/VIS/NIR range, the Method #3 for low absorption windows (section 4.5) has been tested successfully with a 1 mm thick quartz window and the obtained result was practically identical to the result obtained by Method #1.

5 Practical application

5.1 UV/VIS/NIR measurement in spectrophotometers

Typically employed spectrophotometers (such as the PerkinElmer Lambda series) employ integrating spheres, which require vertical sample positioning. For this reason, the measurement of the particle film needs to be accomplished through a window.

In case that a direct measurement of particles is possible (without any window) the subsequent chapters can be omitted and the particles should be measured as a regular sample according to the actual SolarPACES reflectance guideline [5].

5.1.1 Recommendations for the sample holder

The sample holder for the particles should contain a UV/VIS/NIR transparent window. It is recommended to use a fused silica or quartz window due to its low absorption in the solar wavelength range. The thickness of the window should be low (1 or 2 mm), so that the distance between the specimen and the integrating sphere is minimized. For the same reason, one should try to minimize the air gap between the sample port and the window. The window should be easily removable from the holder, since it needs to be measured separately and in combination with the reference coupon. The window should be handled with care to avoid scratching due to the contact with the particles. Also, before measurements, the window should be blown dry with filtered pressurized air to remove eventually deposited dust particles. An exemplary sample holder is shown in Figure 2. Note that the removable ring leads to an air gap between the integrating sphere port and the window. As mentioned, the thickness of the removable ring should be as low as possible.

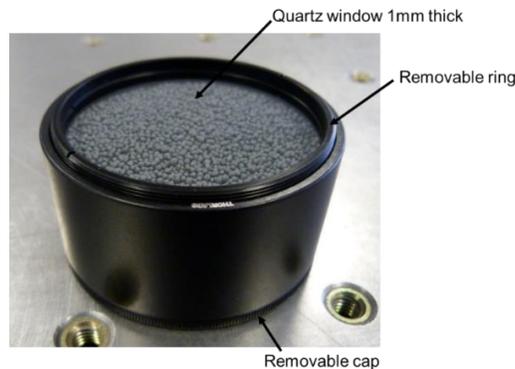


Figure 2: Exemplary sample holder for UV/VIS/NIR measurements

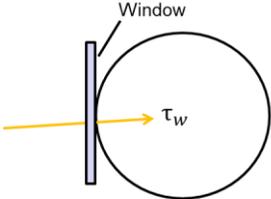
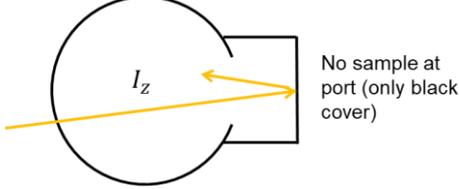
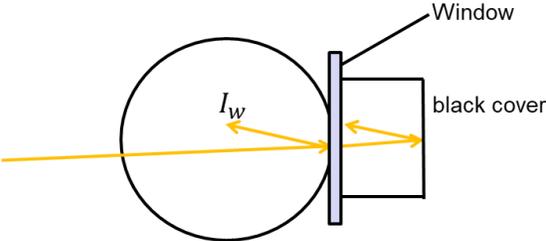
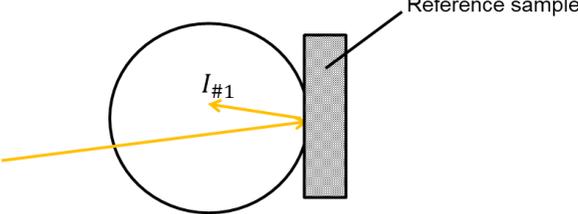
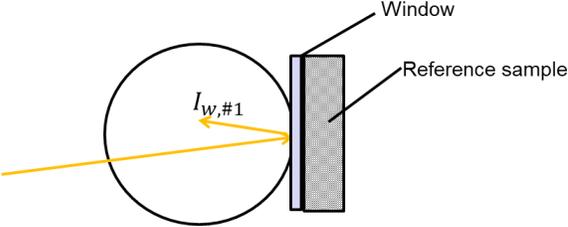
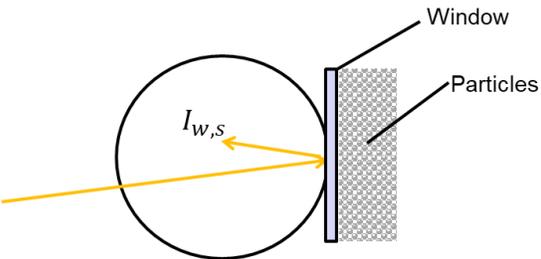
5.1.2 Recommendations for the reference coupon

It is recommended to use a reference coupon of similar reflectance characteristics than the particles to be measured. As general advice a diffuse spectralon coupon of 10 – 20 % reflectance should be used.

5.1.3 Measurement protocol

Table 1 details the required measurements, which need to be collected at the different port configurations. The table also includes the formulas to compute the reflectance for each step. In modern UV-VIS-NIR spectrophotometers zero-line correction can be activated to be accomplished automatically. Users should only take care about centering the measurement beam well in the port of the integrating sphere to be sure that the entire light beam crosses the port without losses. Note that an additional zero-line displayed in Table 1 needs to be collected with the light source turned on, in order to correct for backscattered light of the port during the reflectance measurement of the window. Be aware that the automatic zero-line correction does not correct this artifact.

Table 1: Protocol to determine UV/VIS/NIR reflectance with spectrophotometers with integrating spheres

| Description | Measured quantity | Port configuration | Reflectance calculation |
|---|-------------------|--|---|
| Window transmittance | τ_w |  | - |
| Zeroline with light source turned on for ρ_w | I_z |  | - |
| Window reflectance | I_w |  | $\rho_w = \frac{I_w - I_z \cdot \tau_w^2}{I_{\#1}} \cdot R_{\#1}$ |
| Baseline of grey spectralon | $I_{\#1}$ |  | Reflectance $R_{\#1}$ given by calibration certificate |
| Window + Reference | $I_{w,\#1}$ |  | $\rho_{w,\#1} = \frac{I_{w,\#1}}{I_{\#1}} \cdot R_{\#1}$ |
| Window + Particles | $I_{w,s}$ |  | $\rho_{w,s} = \frac{I_{w,s}}{I_{\#1}} \cdot R_{\#1}$ |

5.1.4 Window correction

The spectral particle reflectance can be computed according to Eq. 3 (Method #0). The spectral particle absorptance is then given by $A_s = 1 - R_s$.

Note: the correction method of choice is still under scientific investigation and may be subject of change in future versions of this guideline. A Round Robin Test of different particle types between different institutes has shown acceptable agreement between corrected window measurements according to Method #0 and windowless direct measurements [8].

5.1.5 Solar-weighting of particle reflectance/ absorptance

Following ASTM Standard E903-82 (92) [6], the solar-weighted hemispherical reflectance $R_{s,h}$ can be calculated by weighting the spectral hemispherical reflectance $R_s(\lambda)$ with the solar direct irradiance G_b on the earth surface for each wavelength according to Eq. 14.

$$R_{s,h} = \frac{\int_{\lambda_{min}}^{\lambda_{max}} R_s(\lambda) \cdot G_b(\lambda) d\lambda}{\int_{\lambda_{min}}^{\lambda_{max}} G_b(\lambda) d\lambda} \quad (15)$$

Where $\lambda_{min} = 300\text{nm}$ and $\lambda_{max} = 2500\text{nm}$

For European and North American latitudes typical solar irradiance spectra are given by the current standard ASTM G173-03 [7] (direct irradiance) for air mass AM 1.5.

The wavelength range of the solar spectral irradiance provided in [7] is $\lambda = [280,4000]$ nm. Because the far UV, UV-B and mid-infrared range have a negligible impact on $R_{s,h}$ and it is practically more convenient for the measurement equipment, the relevant measurement range for reflectance evaluation can be resized to $\lambda = [320,2500]$ nm.

The solar-weighted hemispherical particle absorptance is given by $A_{s,h} = 1 - R_{s,h}$.

5.2 IR measurement in Fourier-Transform infrared (FTIR) spectrometers

For integrating spheres with downward configuration, the particles can just be measured as a normal sample according to common measurement protocols.

An alternative option to avoid measuring through a window in upward configured integrating sphere, is to carefully pour the particles with a funnel into the bottom of the sphere and to close the sample port of the sphere with a gold reference standard. This approach has been tested by CIEMAT. However, it required special care not to damage or contaminate the integrating sphere. A small vacuum cleaner was used to remove all particles from the sphere after the measurement.

In the typical case, the employed gold integration spheres for FTIR spectrometers have an upward configuration of the sample port, requiring to measure the particle film through a window. The following chapters deal with the step-by-step measurement protocol for the latter case.

5.2.1 Recommendations for the sample holder

The sample holder for the particles should contain an IR transparent window for the wavelength range of interest (typically 2-20 μm). The recommended materials are ZnSe and Ge windows, while the latter one has lower transmittance. Anti-reflection coatings are recommended to boost transmittance. However, care must be taken not to scratch the coating when handling the window with the particles on top.

The thickness of the window should be low, so that the distance between the specimen and the integrating sphere is minimized.

The window needs to be measured on its own and in combination with reference coupons. For this reason, it should be removable from the sample holder or embedded in a flat plate, as shown in Figure 3. This Figure also illustrates how to carefully pour the particles onto the window by using a funnel, directing the particles to the metallic part of the holder. In this way a direct impact of particles onto the thin coatings of the window is minimized.

In general, the window should be handled with care. Fingerprints must be avoided. Before measurements, the window should be blown with filtered pressurized air to remove eventually deposited dust particles.

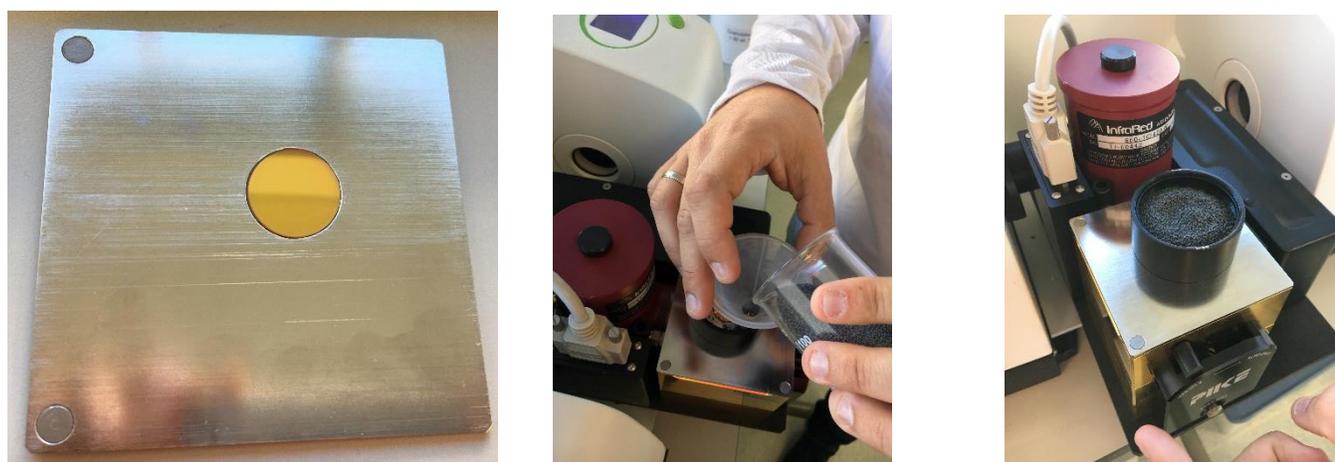


Figure 3: Exemplary sample holder for FTIR measurements. The shown window on the left is consists of ZnSe with a thickness of 2 mm and anti-reflection coatings, reaching around 90% transmittance.

5.2.2 Recommendations for the reference coupon

As general rule, it is recommended to use a certified diffuse gold reference coupon to conduct the reference measurement according to Table 2.

If the employed window does not contain an anti-reflection coating, then an additional certified reference coupon needs to be employed. The reflectance characteristics of the second coupon should be substantially different from the gold coupon, although it should be diffuse as well. For instance, a certified coupon with a diffuse solar absorber coating (like Pyromark 2500) could be used.

As mentioned in section 0, it can be helpful to use a certified specular gold reference coupon to measure the reflectance of the specular window. Although for the herein tested golden integrating sphere from Pike Technologies the window reflectance obtained with a diffuse and specular gold standard was identical, every sphere can be different and as general rule specular samples should be measured with specular standards.

5.2.3 Measurement protocol

Table 2 details the required measurements, their baseline correction and port configurations. The table also includes the formulas to perform a manual computation of the reflectance, in case the instrument is not configured to output reflectance but intensity values.

Typically, the integrating sphere has an internal reflector which can be positioned with a lever to “reference” or “sample” position. In “reference” position, the beam is facing the bottom of the integrating sphere, while in “sample” position the beam is facing the sample positioned at the sample port. There are two options to conduct measurements, by means of the “Taylor” or the “Substitution” method. It is recommended to follow the “Taylor” method, since the substitution error is smaller than for the “Substitution” method. The tests conducted by the authors of this guideline revealed that the differences between both methods are not negligible. Thus, the depicted protocol in Table 2 is based on the “Taylor” method.

Before conducting measurements, it needs to be made sure that the FTIR spectrometer is well aligned. It is recommended to conduct the following two checks at the beginning of each measurement day:

1. With no sample mounted on the sample port and the lever in “sample” position, the screw of the lever stop should be adjusted until the signal (detector energy) is minimized. This procedure will make sure that the beam is well aligned onto the sample port (see Figure 4).

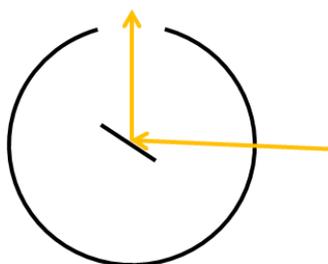


Figure 4: Alignment of upper internal reflector stop until signal is minimized

2. With the diffuse gold standard mounted on the sample port, the lever should be switched to “reference” position and a background correction should be performed. Then, the lever should be switched to “sample” position and the gold standard should be measured. It needs to be checked that the measured intensity of the gold standard $I_{\#1}$ is close to 100% (since the gold standard has a similar reflectance as the bottom of the integrating sphere). Otherwise the lower lever stop needs to be adjusted and the described steps need to be repeated. This procedure will make sure that the beam is well aligned to the bottom of the sphere.

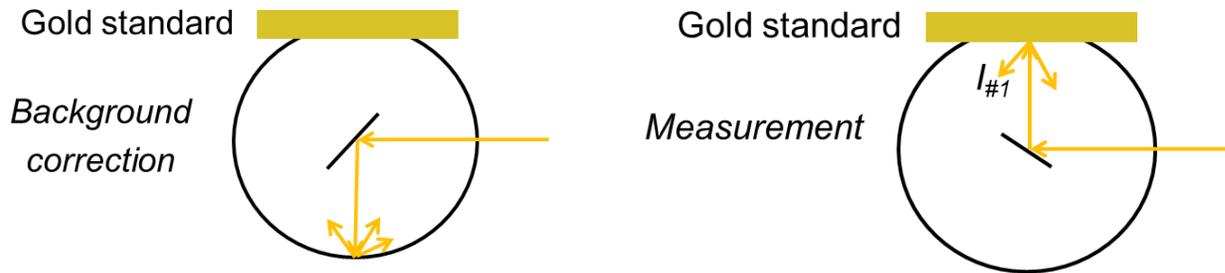
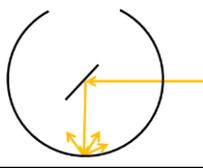
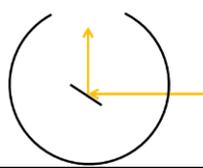
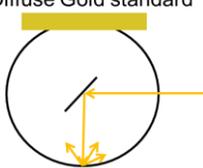
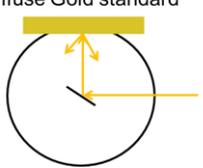
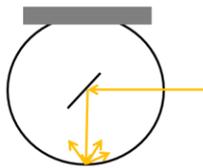
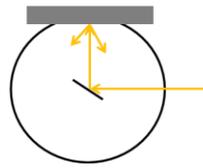
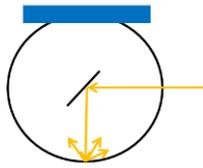
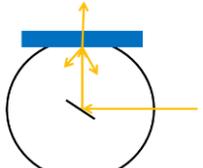
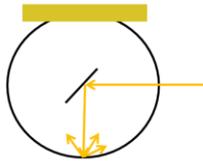
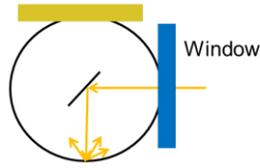
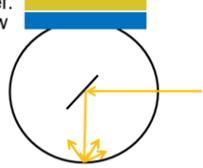
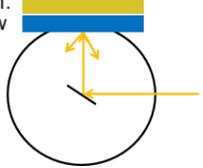
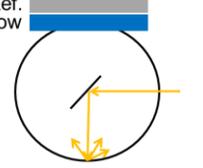
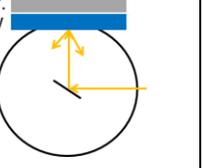
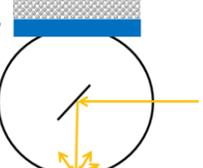
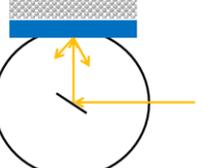


Figure 5: Alignment of lower internal reflector stop until measurement of gold standard is $\approx 100\%$.

Table 2: Measurement protocol to determine IR reflectance with FTIR spectrometers with golden integrating spheres

| Description | Measured quantity | Background correction configuration | Measurement configuration | Reflectance calculation |
|-----------------------------|-------------------|--|---|--|
| Zeroline | I_z | Empty sample port  | Empty sample port  | - |
| Diffuse Gold Reference | $I_{\#1}$ | Diffuse Gold standard  | Diffuse Gold standard  | Reflectance $R_{\#1}$ given by calibration certificate |
| Diffuse Absorber Reference | $I_{\#2}$ | Diffuse absorber standard  | Diffuse absorber standard  | Reflectance $R_{\#2}$ given by calibration certificate |
| Window | I_w | Window  | Window  | $\rho_w = \frac{I_w - I_z}{I_{\#1} - I_z} \cdot R_{\#1}$ Note: if results are inconsistent a specular gold reference should be used |
| Window | τ_w | Diffuse Gold standard  | Diffuse Gold standard Window  | - |
| Window + Gold Reference | $I_{w,\#1}$ | Gold Ref. Window  | Gold Ref. Window  | $\rho_{w,\#1} = \frac{I_{w,\#1} - I_z}{I_{\#1} - I_z} \cdot R_{\#1}$ |
| Window + Absorber Reference | $I_{w,\#2}$ | Absorber Ref. Window  | Absorber Ref. Window  | $\rho_{w,\#2} = \frac{I_{w,\#2} - I_z}{I_{\#2} - I_z} \cdot R_{\#2}$ |
| Window + Particles | $I_{w,s}$ | Particles Window  | Particles Window  | $\rho_{w,s} = \frac{I_{w,s} - I_z}{I_{\#1} - I_z} \cdot R_{\#1}$ |

5.2.4 Window correction

The spectral particle reflectance can be computed according to Eq. 3 (Method #0). The spectral particle emittance at room temperature is then given by $E_s = 1 - R_s$.

Note: the correction method of choice is still under scientific investigation and may be subject of change in future versions of this guideline. A Round Robin Test of different particle types between different institutes has shown acceptable agreement between corrected window measurements according to Method #0 and windowless direct measurements [8].

5.2.5 Thermal emittance calculation

The thermal emittance of the particle layer $E_s(T)$ is calculated at a receiver temperature T according to

$$E_s(T) = \frac{\int_{\lambda_{min}}^{\lambda_{max}} [1 - R_s(\lambda)] \cdot M_{bb}(\lambda, T) d\lambda}{\int_{\lambda_{min}}^{\lambda_{max}} M_{bb}(\lambda, T) d\lambda} \quad (16)$$

$$M_{bb}(\lambda, T) = \frac{2\pi hc^2}{\lambda^5 \left[e^{\left(\frac{hc}{\lambda kT}\right)} - 1 \right]} \quad (17)$$

where M_{bb} is the blackbody spectral emittance in $W/(m^2 \cdot \mu m)$, c , h and k are universal physical constants, which respectively correspond to the speed of light in vacuum ($2.997 \cdot 10^8$ m/s), Planck's constant ($6.63 \cdot 10^{-34}$ Js) and Boltzmann's constant ($1.38 \cdot 10^{-23}$ J/K).

The wavelength ranges from $\lambda_{min} = 300$ nm and $\lambda_{max} = 16$ μm. As such, both UV/VIS/NIR and IR data are required to evaluate the thermal emittance.

The thermal emittance of the particle layer $E_s(T)$ shall be evaluated at a temperature of 900°C.

5.3 Handheld reflectometers for particle characterization

In lieu of traditional laboratory spectroscopy, particle emittance and absorptance can be captured using handheld instruments. Surface Optics Corporation manufactures the 410-Solar to measure hemispherical and specular reflectance from 335-2500 nm and the ET100 to capture thermal emittance from 1.5 to 21 micron. These devices are easy to calibrate, portable for use in various environments, and can be used in any orientation, including face-down to accommodate an open container of particles. When the experiment is configured for an open container, the instruments can measure particle samples without a window covering, negating the need for additional correction methods to compensate for window transmissivity effects. A summary of the instruments' specifications is available below in Table 3.

Table 3. Handheld reflectometer specifications.



| SOC 410-Solar | | SOC ET-100 | |
|-------------------|---|-------------------|--|
| Measures: | Hemispherical and specular reflectance, absorptance | Measures: | Directional and hemispherical thermal emittance from absolute reflectance |
| Wavelength Bands: | 335-2500 nm 1. 335-380 nm 2. 400-540 nm 3. 480-600 nm 4. 590-720 nm 5. 700-1100 nm 6. 1000-1700 nm 7. 1700-2500 nm | Wavelength Bands: | 1.5-21 micron 1. 1.5-2.0 um 2. 2.0-3.5 um 3. 3.0-4.0 um 4. 4.0-5.0 um 5. 5.0-10.5 um 6. 10.5-21 um |
| Incidence Angle: | 20 deg | Incidence Angle: | 20 deg (hemispherical, directional) 60 deg (directional) |

The procedure for using both instruments is identical and requires minimal setup. It is recommended to place the particles in an appropriate crucible-style holder. This holder is designed to support the aperture of the instrument during measurement, while allowing the incident beam to only contact the particle surface. The particle bed depth should be at least 5-10 particle diameters to ensure the base of the crucible does not impact the reflectance measurements. Two example particle holders are shown below in Figure 6. Two machined sample holders. Each holder is filled such that the measurement plane is flush with the aperture support surface.



(a) Round sample holder



(b) Square sample holder

Figure 6. Two machined sample holders. Each holder is filled such that the measurement plane is flush with the aperture support surface.

To conduct a measurement, perform the following steps:

1. Power on the instrument and follow the 410-Solar or ET100 on-screen calibration instructions
2. Prepare the particle sample
 - a. Ensure the particle samples are homogenously distributed. If necessary, invert the closed sample container several times to distribute.
 - b. Fill the sample holder with the particle sample such that it is level with the measurement plane of the holder, see filled sample holders in Figure 6 above
3. Take a measurement - Figure 7
 - a. Place the instrument aperture onto the holder such that the edges of the holder support the aperture gasket
 - b. Press the trigger to record a measurement
 - c. Assess on-screen readout for viability and errors



Figure 7. 410-Solar placed on a square sample holder for measurement

4. Repeat measurement process

- a. To collect a representative population, the particle holder should be emptied and refilled between each measurement.
- b. Repeat steps 2 and 3.

Files are saved on the instrument memory card in a flat-text format and are readily transferred to text or csv files. Data provided is raw only and must be weighted appropriately in post-processing.

6 References

- [1] ISO 9050:2003 Glass in building - Determination of light transmittance, solar direct transmittance, total solar energy transmittance, ultraviolet transmittance and related glazing factors
- [2] E. Nichelatti, "Complex refractive index of a slab from reflectance and transmittance: analytical solution" J. Opt. A: Pure Appl. Opt. 4 (2002) 400-403, stacks.iop.org/JoptA/4/400.
- [3] H. A- Macleod, "Thin-Film Optical Filters", 3rd edition, IOP editor, 2001.
- [4] L. Vriens, W. Rippens, "Optical constant of absorbing thin films on a substrate", Appl. Opt. 22 (1983) 4105-4110.
- [5] A. Fernández-García et.al, "Parameters and method to evaluate the reflectance properties of reflector materials for concentrating solar power technology under laboratory conditions" available online:
https://www.solarpaces.org/wp-content/uploads/202004_SolarPACES-Reflectance-Guidelines-V3.1.pdf
- [6] ASTM E903-82. Standard Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres, ASTM International, 2012.
- [7] ASTM G173-03 Standard Tables for Reference Solar Spectral Irradiances: Direct Normal and Hemispherical on 37° Tilted Surface, ASTM International, 2003.
- [8] F. Sutter, M. Montecchi, A. Morales Sabio, G. San Vicente, A. Fernández-García, J. Pernpeintner, T. Reche-Navarro, S. Caron, L. Martínez-Arcos, A. Calderón, M. Majó, I. Fernández, P. Davenport, T. Farrell, C. Ho: Round Robin Test of Absorptance and Emittance of Particles for CSP. To be published in the proceedings of the SolarPACES conference 2022.