



16NRM06 EMIRIM

Improvement of emissivity measurements on reflective insulation materials

Good-practice guide :

Procedures for measurement of total near-normal emissivity of low emissivity foils enabling end-users to perform emissivity measurements on reflective foils with an uncertainty below 0.03 for emissivities below 0.1 by using integrating spheres setups or total hemispherical near-normal reflectometers.

Procedure for calculation of total hemispherical emissivity

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1. INTRODUCTION

The objective of this good practice guide is to give to end-users the recommendations and procedures for:

- performing total near-normal emissivity measurements on reflective foils (with emissivity < 0.1) with an uncertainty below 0.03 using the commercial TIR100-2 emissometer or an infrared integrating sphere associated to Fourier Transform spectrometer,
- determining the total hemispherical emissivity of reflective foils (with emissivity < 0.1) with an uncertainty below 0.05.

The devices mainly used in EU by end-users for measuring the emissivity of reflective foils are integrating spheres associates with FTIR spectrometers and the reflectometer TIR100-2 from INGLAS company.

The two techniques of measurement were studied in detail in EMIRIM project and their performances for determining the total hemispherical emissivities of low emissivity reflective foils were quantified.

2. MEASUREMENTS WITH INTEGRATING SPHERES

2.1. GENERAL PRINCIPLE OF THE INTEGRATING SPHERE

Measurement of the near-normal spectral emissivity of the sample:

The principle of measurement is presented in **Erreur ! Source du renvoi introuvable.** The incident radiation beam (spectrally modulated) is produced by the FTIR spectrometer. The integrating sphere is used to collect the radiation reflected by the sample surface whatever the angular diffusion of reflection. The incident beam is slightly incline by reference to the normal to the sample surface so that the specular component of reflection is always collected by the integrating sphere. Due to multiple reflections of the radiation reflected by the sample on the integrating sphere walls, the integrating sphere method is in principle not sensitive to the angular distribution of the reflection on the sample and the signal measured by the detector is proportional to the reflectance of the sample. The system must be calibrated using a sample calibrated in near-normal spectral reflectance; it is preferable that the reference sample is highly reflective so that the "reference" signal measured is high and less affected by noises.

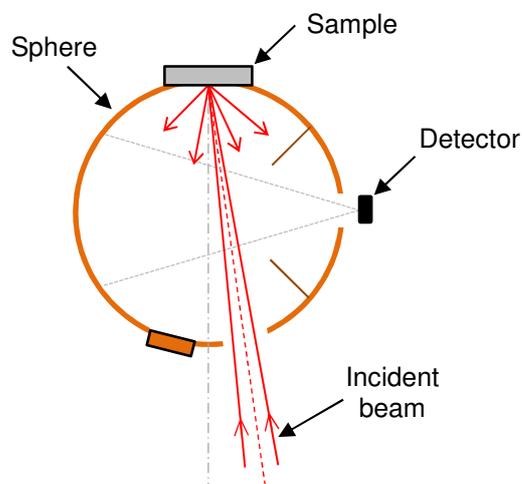


Figure 1: General principle of the integrating sphere

2.2. CALIBRATED STANDARD

For measurement of emissivity of low emissivity foils, the use of a specular low emissivity standard (a mirror) is recommended.

The calibration of the standard should be traceable to SI (International System of Units) and the expanded uncertainty on the total near-normal emissivity of the standard should be around 0.02 ($k=2$).

2.3. SAMPLES TESTED

If the reflective foils tested are transparent or have holes (e.g. for water vapour permeability), a high emissivity opaque surface should be placed in contact with the rear side of the foil during measurements. This configuration better represents the actual configuration of use of most of the reflective foils in thermal insulation systems with usually a high emissivity material behind the foil.

2.4. DESCRIPTION OF A SEQUENCE OF MEASUREMENT WITH CORRECTION OF THE "SINGLE BEAM SUBSTITUTION ERROR"

With an integrating sphere, when the high reflective calibrated reference sample is replaced by another sample, the mean reflectance of the cavity constituted of the integrating sphere walls, of the openings and of the sample surface visible from the inside of the sphere is changed. Thus, the radiometric sensitivity of the integrating sphere system is modified. This modification of radiometric sensitivity is usually significant and must be taken into account to calculate the spectral reflectance of the sample without error. The error generated by the variation of sensitivity with the reflectance (emissivity) of the sample is called "single-beam sample-absorption error" [1] or "single beam substitution error" [2].

Figure 2 shows the different configurations for reflectance measurements with systematic correction of the "single beam substitution error".

2.4.1. Measurement of signals

The measurements of signals must be performed once the response of the detector is steady. That means that the spectrometer, the radiation source and the detector must be steady in temperature as well as the related electronic systems. If the spectrometer and/or the sphere are purged with a gas flow, the purging should have started enough time before the calibration so that the response of the detector is steady.

In this procedure, six signals are measured:

- $S_{ref\ stand}$: signal measured when the beam is incident on the calibrated reference standard (Ref. standard config. in Figure 2.),
- $S_{sphere\ ref\ stand}$: signal measured when the beam is incident on the sphere wall with calibrated reference standard at the sample port (Sphere config. with ref standard in Figure 2.),
- S_{sample} : signal measured when the beam is incident on the sample in test (Sample config. in Figure 2.),
- $S_{sphere\ sample}$: signal measured when the beam is incident on the sphere wall with the sample in test at the sample port (Sphere config. with sample in Figure 2.),
- S_{zero} : signal measured without sample or with a blackbody at the sample port (Zero config in Figure 2.).
- $S_{sphere\ zero}$: signal measured without sample or with a blackbody at the sample port (Sphere config. no sample in Figure 2.).

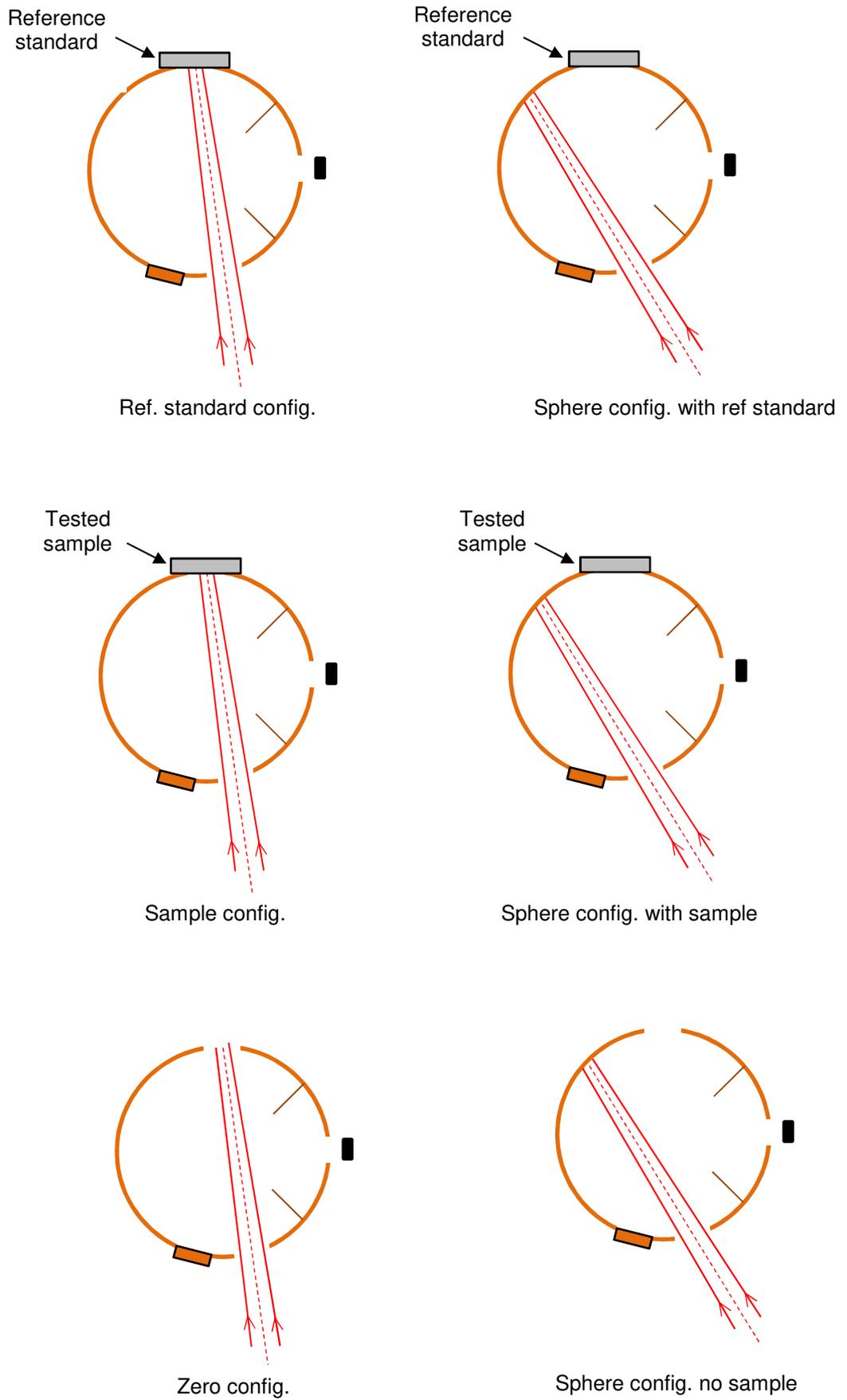


Figure 2: Configurations for measuring near-normal reflectance with an IR integrating sphere

For the configurations with the beam incident directly on the sphere wall, the beam must be incident always on the same area of the sphere for the three "sphere signals". For the six measurements, the power of the incident beam should not be affected by the configuration of measurement and the geometry of the beam should not be modified upstream of the entrance of the integrating sphere.

Remarks:

The signals above are spectral signals (signal for each wavelength). With Fourier transform spectrometers, a measurement gives a spectrum which is the set of spectral signals over the spectral range configured.

Users of integrating spheres associated to spectrophotometers for reflectance measurements in the UV, visible and near-infrared spectral ranges may be surprised by the need to perform six measurements to get the reflectance result. Indeed, with a spectrophotometer equipped with an integrating sphere, only two measurements are usually performed, the "reference measurement" and the "sample measurement". This is enabled thanks to the correct optical configurations of most commercial devices (zero signal = 0) and to the "double beam configuration" which corrects "automatically" the "single beam substitution error".

2.4.2. Calculation of near-normal spectral reflectance and of near-normal emissivity

From the six signals measured, the near normal spectral reflectance is calculation using the relation

$$\rho_{\lambda nn} = 1 - \frac{\left(\frac{S_{sample}}{S_{sphere sample}} - \frac{S_{zero}}{S_{sphere zero}} \right)}{\left(\frac{S_{ref stand}}{S_{sphere ref stand}} - \frac{S_{zero}}{S_{sphere zero}} \right)} \cdot \rho_{ref \lambda} \quad (1)$$

with

$\rho_{\lambda nn}$: the spectral near-normal hemispherical reflectance of the sample.

$\rho_{ref \lambda}$: the spectral near-normal hemispherical reflectance of the low emissivity calibrated reference sample.

The sample tested being opaque*, the near-normal emissivity of the sample is calculated using the relation

$$\varepsilon_{\lambda nn} = 1 - \rho_{\lambda nn} \quad (2)$$

* In fact, using a high emissivity opaque surface behind the sample ensures the opacity.

2.5. MEASUREMENT IF THE MEASUREMENTS FOR THE CORRECTION OF THE "SINGLE BEAM SUBSTITUTION ERROR" CAN'T BE DONE

If technically, it is not possible to perform the measurements with the beam incident directly on the sphere wall (or an equivalent configuration), it is difficult to perform measurements with small uncertainties due to the "single beam substitution error".

The possible solutions to correct for the "substitution" error can be:

- to establish a model of multireflections for the virtual cavity created by the sphere and the sample and calculate the relative variation of the sensitivity as a function of the reflectance of the sample,
- to use a set of calibrated standards with different level of reflectance to establish, from measurement results, the law of variation of the sensitivity as a function of the reflectance of the sample.

The solution of using a set of calibrated standards with different level of reflectance seems better but it is quite difficult to have surfaces with medium reflectances which are specular.

The uncertainties for these solutions could be quite large.

2.6. POTENTIAL DIFFICULTIES WHEN USING AN INTEGRATING SPHERE FOR REFLECTANCE MEASUREMENTS

The sequence of measurement with correction of the "single beam substitution error" presented in 2.4.1 is appropriate when the optical configuration of the integrating sphere is correct.

Users must be aware of the risk of poor or incorrect optical configuration when using "infrared" integrating spheres because the risk of occurrence is not low even with commercial integrating spheres.

2.6.1. Definition of a Good optical configuration

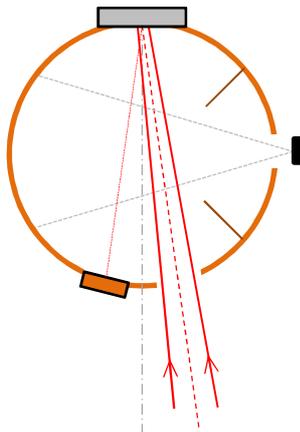


Figure 3: Good optical configuration when using an IR integrating sphere

A definition of a correct optical configuration can be:

- the incident beam is not limited by the entrance port of the sphere,
- the area irradiated on the sample is not larger than the sample port,
- the area irradiated on the sample is not too close from the edges of the sample port,
- when a specular sample is at the sample port, the area irradiated after the first reflection is entirely on the specular port plug,
- the sample surface is well in contact with the sphere (no gap),
- the detector cannot "see" directly the surface of the sample (baffle to screen the sample),
- the detector cannot "see" directly the specular port (presence of a baffle),
- the coating on the internal wall of the sphere is uniform in reflectance, including the specular port plug.

2.6.2. Bad optical configurations

The bad optical configurations that can occur are shown graphically in Figure 4, Figure 5 and Figure 6.

2.6.2.1. Configurations giving a "zero level" signals not equal to zero

The configurations giving a "zero level" signals not equal to zero are an incident beam limited by the entrance port of the sphere or/and the area irradiated by the incident beam is larger than the sample port.

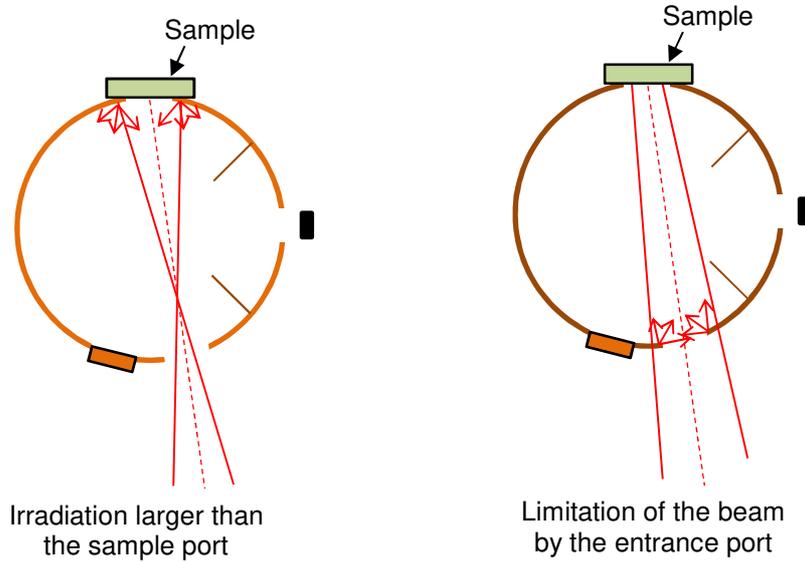


Figure 4: Configurations giving a "zero signal" different from zero

2.6.2.2. Configurations giving potentially errors on measured reflectance

a) Irradiation close to the sample port edges, gap between the sample and the sphere

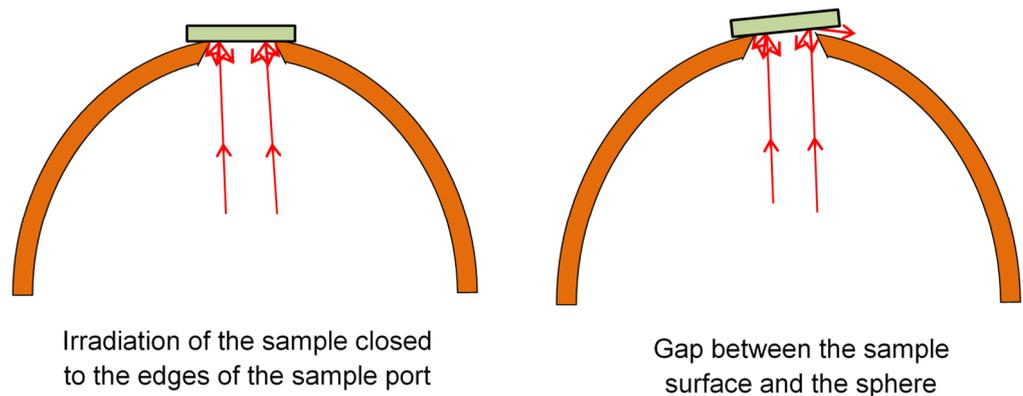


Figure 5: Configurations giving a response dependent on the specularity of the sample

When the field irradiated on the sample is close to the limits of the sample port, inter-reflections can occur between the sample surface and the edges of the sample port. It is highly probable that those inter-reflections depend on the angular diffusion of the radiation reflected by the sample producing different proportions of inter-reflections for the reference standard and for the sample tested.

When a gap exists between the sample surface and the sphere there is a risk of loss of the radiation reflected by the sample and therefore a risk of error on the measured reflectance.

b) Irradiation larger than the specular sample port when calibrating with a specular standard

When a specular sample is at the sample port, it is possible that the beam reflected specularly by the sample irradiates, after the first reflection on the sample, an area larger than the specular port. Even if a specular plug is at the specular port to avoid the loss of radiation reflected specularly, a narrow gap always exists between the specular plug and the edge or sphere at the specular port. This narrow gap is highly absorbing by comparison to the sphere coating and absorbs a part of the radiation reflected by a specular standard or sample. The proportion absorbed is not identical if the specularity (angle diffusion) of the sample tested is not the same as the specularity of the low emissivity standard. This produces an error on the measured reflectance.

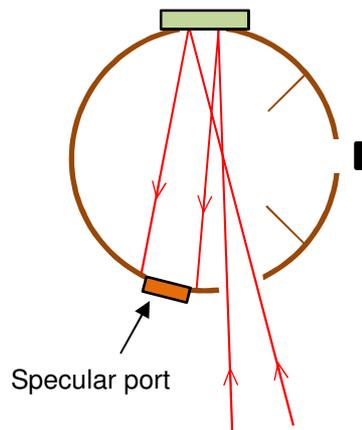


Figure 6: Configurations giving a response dependent on the specularity of the sample, specular irradiated area larger the specular port

c) Sensitivity of the integrating sphere sensitive to the angular distribution of the reflected radiation

In principle, the multi-reflections on the sphere wall make the radiance uniform over all the internal surface of the sphere. In reality, this effect is not perfect and the response can depend on the angular distribution of the radiation reflected by the sample, even if the optical configuration is correct.

2.7. QUALIFICATION OF AN INTEGRATING SPHERE FOR EMISSIVITY MEASUREMENTS ON LOW EMISSIVITY FOILS

Before using an integrating sphere, for measurement of emissivity of low emissivity reflective foils, it is recommended to check the optical configuration and the sensitivity to the angular distribution of the radiation reflected by the sample.

The results of those checkings can depend on the optical settings of the FTIR such as the dimension of the aperture defining the source size and the spectral range. The checkings must be performed for all the configurations likely to be used.

2.7.1. Checking of the optical configuration

a) Determination of the spectral reflectance equivalent to noise

It is useful for making decisions when checking the instrument to know the "spectral reflectance equivalent to the noise" of the detection system (detector, amplifier, analog-to-digital converter). The spectral reflectance equivalent to noise is the spectral reflectance measured without radiation entering in the integrating sphere.

First, the integrating sphere is calibrated using the normal procedure, then a sample (e.g. the standard) is placed at the sample port, the beam is totally blocked before the entrance of the sphere and a reflectance measurement is performed using the normal procedure. The beam can be blocked using a plate of an opaque material preferably with a low reflectance (to avoid radiation in the sample compartment or around the sphere).

The spectral reflectance obtained should be very low (below 0.005) in the spectral domain where the signal delivered by the detector is high with a high reflecting sample.

b) Zero reflectance signal measurement – Measurement of the spot size

After a calibration with a high reflective standard, a reflectance measurement is performed using the normal procedure without any sample or with a blackbody placed at the sample port. If the measurement is performed without any sample, the operator must ensure that no surface behind the sample port reflect a part of the radiation toward the sphere through the sample port.

The spectral reflectance curve obtained is compared to the "spectral curve equivalent to detection noise". If the spectral curves are similar in shape and level (within the noise), that means that the optical configuration is correct regarding the beam size at the entrance port and at the sample port. If the spectral curves obtained with a "zero reflecting sample" is significantly higher than the spectral reflectance curve equivalent to detection noise, it means that there is radiation inside the sphere even with a "zero reflecting sample" and it is likely that one or both of the two optical situations illustrated in Figure 4 exists.

If the spectrometer offers a short wavelength configuration with a lamp as radiation source, set the spectrometer in the short wavelength configuration and try to visualize the beam at the entrance port and at the sample port of the sphere (e.g. using a piece of white paper).

If it is not possible to visualize the beam:

- Change the diameter of the aperture defining the source size and recheck the "zero reflectance signal". It is possible that the source size aperture has an effect on the area irradiated in the sample port plane,
- To check if the beam is larger than the entrance port: Place an iris diaphragm close to the entrance port and measure the "zero reflectance signal" reducing by step the diameter of the iris diaphragm. If a reduction of signal is obtained with a diameter of the iris diaphragm smaller than the diameter of the entrance port, it means that the beam is smaller than the entrance port.
- To check if the beam is larger than the sample port: a quite simple solution is to use a sample of a reflecting material and having the sample crossing the sample port by step (Figure 7). The signal starts to increase when a part of the beam is incident on the sample and stabilizes when the beam is entirely reflected by the sample. The size of the spot irradiated by the incident beam is the distance from the point where the signal starts to increase to the point where the signal stabilizes. Here the "signal" can be the raw radiometric signal or the spectral reflectance at a wavelength where the detection system is very sensitive (low noise). It is preferable that the sample is flat, opaque and quite thin to avoid perturbations by reflections on the edge of the sample. The measurement can be done along two perpendicular directions.

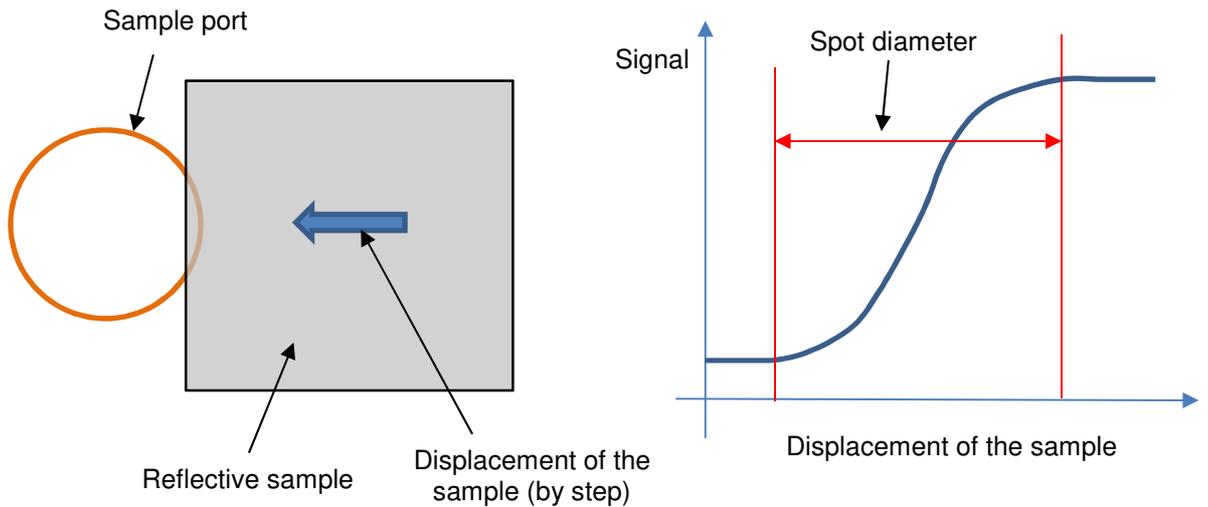


Figure 7: Measurement of the dimension of the measurement field at the sample port

- c) Checking that the area irradiated on the sphere after the first reflection on a specular sample is not larger than the specular port

To check that the beam reflected specularly by a mirror is fully incident on the specular port plug, a measurement with the "Specular excluded" configuration is performed (**Erreur ! Source du renvoi introuvable.**) with a mirror at the sample port. The specular plug is removed and if the configuration is correct the beam reflected by the mirror emerges entirely from the sphere. The spectral reflectance measured in "specular excluded" configuration should be close to the spectral reflectance equivalent to noise.

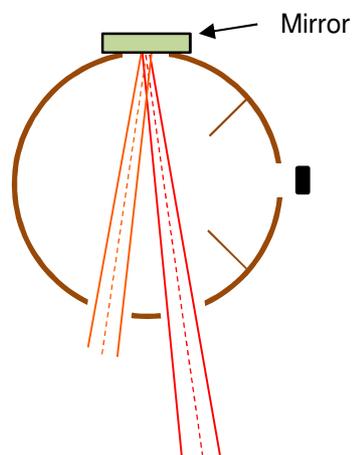


Figure 8: Specular excluded configuration

2.7.2. Checking the sensitivity of the integrating sphere response to the angular diffusion of the reflected radiation on the sample

For users, it is almost not possible to analyse in detail the angular sensitivity of their integrating spheres. Furthermore, the optical configuration of the beam produced by the FTIR spectrometer has an influence of the sensitivity to angular diffusion.

The procedure recommended for checking the sensitivity to angular diffusion of the sample, is to have two calibrated standards one very specular (mirror) and one very diffusing (Infragold® type

coating). Those two standards should be calibrated in near-normal spectral reflectance with an uncertainty as low as possible.

The instrument being calibrated with the specular standard, a spectral reflectance measurement is performed on the very diffusing standard (the complete measurement sequence described in 2.4.1 should be followed when possible).

The spectral reflectance curve obtained for the very diffusing standard is compared to the spectral reflectance curve from the calibration certificate. If the difference is significantly large by reference to the calibration uncertainties for the two standards a sensitivity to the angular diffusion of the sample should be suspected.

It can be preferable to compare the total near-normal emissivity or reflectance values in order to filter the spectral noises on the spectral reflectance curves. If the expanded uncertainties are 0.02 ($k=2$) for the calibration values of total near-normal reflectances (or emissivities) of the two standards then a difference higher than 0.025 should be considered as a suspicion of sensitivity to angular diffusion.

2.8. IMPROVEMENT OF AN INCORRECT OPTICAL CONFIGURATION

The defect such as the beam larger than the entrance can be partly corrected in terms of effect by placing a thin stop at the entrance port or before the entrance port. It is probably better that the sides of the stop have a very low reflectance to reduce parasitical reflected radiation. It is also probably better to avoid the presence of a window in front of the entrance port*.

The influence of a beam larger than the sample port can be reduced (may be) by placing a fixed thin stop at the sample port. This stop should have an opening a bit smaller than the sample port and its function would be to reduce the multiple-reflexions between the sample and the edges of the sample port*.

* Those potential solutions are given without any guaranty of efficiency.

2.9. PROCEDURE FOR MEASUREMENT OF THE TOTAL NEAR-NORMAL EMISSIVITY OF REFLECTIVE FOILS WITH AN INTEGRATING SPHERE

2.9.1. Preparation of the instrument

The measurements must be performed once the response of the detector is steady. That means that the spectrometer, the radiation source and the detector must be steady in temperature as well as the related electronic systems. If the spectrometer and/or the sphere are purged with a gas flow, the purging should have started enough time before the calibration so that the response of the detector is steady.

2.9.2. Calibration – Zero control

The integrating sphere is calibrated with a specular calibrated standard. The signals $S_{ref\ stand}$ and $S_{sphere\ ref\ stand}$ are measured as defined in 2.4.1.

The signals S_{zero} and $S_{sphere\ zero}$ are also measured as defined in 2.4.1. If after the measurement of S_{zero} it is demonstrated that S_{zero} is low enough to not apply a "zero correction" it is enough to measure only S_{zero} .

2.9.3. Measurement of signals on the foils

After calibration and zero control, the measurements are performed on the samples of the low emissivity foil tested.

The two signals S_{sample} and $S_{sphere\ sample}$, as defined in 2.4.1 are measured for each sample (position of the measurement spot).

2.9.3.1. Checking of the calibration

When the measurements on the samples of the foils are completed, a checking of the calibration is done by a measurement on the reference standard.

If the measurements of the foils take a long time, it is recommended to check the calibration periodically (each hour at least).

2.9.3.2. Calculation of the near-normal spectral reflectance

The near-normal hemispherical spectral reflectance is calculated using relation 1.

If the zero signals are nor not measured for each measurement campaign, because the optical configuration is known to ensure a non-significative zero signal, then the ratio $\frac{S_{zero}}{S_{sphere\ zero}}$ is set at 1 in relation 1.

The near-normal spectral emissivity is calculated over the spectral range of measurement using relation 2.

2.9.4. Calculation of the total near-normal emissivity

An "integrating sphere + FTIR spectrometer" setup allows measurement of the spectral near-normal emissivity over a limited spectral range. The parameter required for reflective foils used as external surface of thermal insulation products is the total hemispherical emissivity.

For applications in buildings insulation, the reflective foils are used around room temperature. The spectral range 5 μm to 50 μm represents more than 95.7% of the total thermal radiation emitted by a grey body (constant spectral emissivity).

The relation recommended by standard EN 12898 for calculation of total near-normal emissivity is

$$\varepsilon_{tot\ NN} = \frac{\int_{5\mu\text{m}}^{50\mu\text{m}} \varepsilon_{\lambda\ nn\ meas} \cdot l^{\circ}(T_{samp}, \lambda) \cdot d\lambda}{\int_{5\mu\text{m}}^{50\mu\text{m}} l^{\circ}(T_{samp}, \lambda) \cdot d\lambda} \quad (3)$$

with ε_{λ} the measured near-normal spectral emissivity of the sample, T_{samp} the temperature of the material in use (usually the room temperature), λ the wavelength and $l^{\circ}(T_{samp}, \lambda)$ the spectral radiance of a blackbody (Planck's law).

This relation is the one usually used as the definition of the total near-normal emissivity. The calculation of the integrated emissivity can be done using a spreadsheet, a software or the method suggested in Annex A of standard EN 12898:2019 (calculation of the mean value of a set of spectral reflectances measured on the foil at specific wavelengths, the temperature for calculation is 283 K).

EN 12898:2019 "Glass in building - Determination of the emissivity" specify that the emissivities of glasses should be calculated for the temperature 283 K which is a medium temperature between the indoor temperature and the outdoor temperature. Usually low emissivity foils have a quite constant spectral emissivity for all wavelengths. Therefore, the selection of the temperature from 280 K to 295 K should not have an important effect.

The "integrating sphere + FTIR spectrometer" setups are usually limited to less than 50 μm for the longer wavelengths. The limitation depends mainly on the instrumentation (FTIR instrument, diameter of the integrating sphere, geometrical arrangement of the optical system, type of radiation detector).

The previous relation can be written

$$\varepsilon_{tot NN} = \frac{\int_{5\mu m}^{\lambda_{max}} \varepsilon_{\lambda nn meas} \cdot I^{\circ}(T_{samp}, \lambda) \cdot d\lambda}{\int_{5\mu m}^{50\mu m} I^{\circ}(T_{samp}, \lambda) \cdot d\lambda} + \frac{\int_{\lambda_{max}}^{50\mu m} \varepsilon_{\lambda} \cdot I^{\circ}(T_{samp}, \lambda) \cdot d\lambda}{\int_{5\mu m}^{50\mu m} I^{\circ}(T_{samp}, \lambda) \cdot d\lambda} \quad (4)$$

with λ_{max} the high wavelength limit of the "integrating sphere + FTIR spectrometer" setup used for measurements.

Due to the spectral limitation of the instrument, the near-normal emissivity for the calculation of the second term of the relation is not measurable. A way to overcome the lack of knowledge of the spectral emissivity for longer wavelengths, is to extrapolate it from the spectral emissivity curve obtained experimentally. If the spectral curve in the spectral range of measurement is quite flat particularly for longer wavelengths then the spectral emissivity can be assumed to be constant in function of wavelengths up to 50 μm . This assumption is quite realistic for low emissivity foils using metal foils or metal coatings. With this assumption the relation becomes

$$\varepsilon_{tot NN} = \frac{\int_{5\mu m}^{\lambda_{max}} \varepsilon_{\lambda nn meas} \cdot I^{\circ}(T_{samp}, \lambda) \cdot d\lambda}{\int_{5\mu m}^{50\mu m} I^{\circ}(T_{samp}, \lambda) \cdot d\lambda} + \frac{\int_{\lambda_{max}}^{50\mu m} \varepsilon_{\lambda_{max-50\mu m}} \cdot I^{\circ}(T_{samp}, \lambda) \cdot d\lambda}{\int_{5\mu m}^{50\mu m} I^{\circ}(T_{samp}, \lambda) \cdot d\lambda} \quad (5)$$

with $\varepsilon_{\lambda_{max-50\mu m}}$ the mean near-normal spectral emissivity measured for the longer wavelengths of the spectral range covered by the instrument. The spectral range for calculating the mean value is selected in order to filter the noises on results at long wavelengths.

3. MEASUREMENTS WITH TIR100-2 EMISSOMETER

TIR100-2 is a standalone static instrument with no possibility to change configuration. The metrological performance is highly related to the design and configuration of the instrument.

It was demonstrated in EMIRIM project that TIR100-2 used with appropriate conditions is able to measure total near-normal emissivity on smooth reflective foils with an expanded uncertainty below 0.03 (k=2).

3.1. RECOMMENDATIONS FOR USE OF TIR100.2 WITH GOOD CONDITIONS – PREPARATION BEFORE MEASUREMENTS

The device should be used in calm air in a room with a steady temperature.

The TIR100-2 should be placed in a fixed position to guarantee no movement during measurement. It is recommended to avoid the presence of objects close and in front of the opening of the hot hemisphere materials to let the hemisphere dissipate heat freely and stabilize in temperature.

The TIR100 should be switched on two hours prior measurements to ensure that the temperature of the TIR100 black body is steadied at around 100°C. Once stabilized in temperature, the display shows the measured temperature of the hemisphere. The emissometer can be used when the displayed temperature is 100 ± 0.5 °C and when the temperature variates of less than 0.5 °C for 10 minutes.

3.2. PREPARATION OF THE SAMPLES AND OF THE REFERENCE STANDARDS

For reflective foils, it is recommended to use the sample holder for maintaining the foil during measurements and for avoiding a strong heating of the foil during measurements. Samples with dimension 200 mm x 120 mm are compatible with the sample holder supplied with the emissometer.

The samples and the reference standards should be at the same temperature for the accuracy of results. So the samples and the standards must be placed near the device (but not in front of the hemisphere) for a time long enough to stabilize in temperature.

3.2.1. Measurement of total near-normal emissivity on reflective foils

3.2.1.1. Recommendations for measurements on reflective foils

For reflective foils, it is recommended to use a sample holder with a high emissivity.

The TIR100 needs to be calibrated before measurements and recalibration is recommended each 10 minutes to compensate for thermal drift. So the calibration is integrated in the measurement procedure.

For accurate measurements, the standards surfaces and the sample surfaces must be vertical and in the same plane by reference to the hemisphere. Therefore, the standards and the samples must be placed in contact with the three rubber spacers defining the correct plane.

Even if the temperature of the hemisphere is steady before measurements, the presence, during measurement, of a sample almost closing the hemisphere changes the heat losses of the hemisphere and can change the temperatures of the elements of the radiation detection system. If a sample is maintained in front of the hemisphere for a long time or if a sample is placed in front of the hemisphere with a quite high frequency the hemisphere may be unsteady in temperature generating errors of measurement. Therefore a sample should not be maintained at the sample place by more than a few seconds and a duration of approximately 30 seconds should be respected before placing a new sample in front of the hemisphere.

3.2.1.2. Measurement procedure

After stabilization in temperature, the emissometer is calibrated following the manual.

After switching on and after stabilization in temperature, the device is in the "calibration configuration". Check that the emissivity values set in the device for the standards are the correct ones for the standards used.

In measurement configuration, the calibration configuration is accessible by pressing the "Calibr" touch.

Once in calibration configuration, place the high emissivity standard at the sample place and press "Calibrate High". After a beep, remove the standard and place the low emissivity standard at the sample place and press "CALIBRATE LOW". After a beep remove the low emissivity standard and store the standards near the device but not exposed to the radiation of the hemisphere.

Fix the foil sample onto the sample holder and place the sample at the sample place. Press "measure" on the display and remove the sample after the beep. Between the placement of the sample in contact with the TIR100-2 and the end of the measurement the time should not exceed 2 or 3 seconds to limit the heating of the foil. During the measurement, check that the temperature displayed is the same as when calibrating ± 0.3 K.

If the temperature has varied by more than 0.3 K from the temperature when calibrating, recalibrate the emissometer.

The emissometer must be recalibrated each 10 minutes.

4. SAMPLING – NUMBER OF MEASUREMENTS

At a minimum, the recommendations given in Standard EN 16012+A1 should be followed:

- For a batch of production, a minimum of three specimens should be taken from the sample to be representative of the length and width of the product to include a representative area of any printing or perforation where relevant.
- The emissivity shall be measured in five positions on each specimen.

This gives 15 measurements per batch (per side if sides are different).

Standard EN16012+A1 specifies also that the declared value for a product shall be based upon a minimum of 3 test results (wherever possible from at least 3 different production batches). Therefore, the declared value for a product should be based on a minimum of 45 individual measurements.

The recommendations given in Standard EN16012+A1, are appropriate for quite uniform reflective foils considering the reproducibility of the measurement techniques.

For heavily non-uniform (in terms of emissivity) foils, different strategies can be followed:

- taking numerous samples randomly to cover statistically all the non-homogeneities, a very large number of measurements could be required,
- if the non-homogeneities are specific and localized (e.g. printings), measurements can be performed on each area affected by a specificity and on the non-specific area and the mean emissivity is calculated by ponderation of each area. For each specific area enough measurements should be performed to get a representative result.

An organisation, certifying thermal insulation products, uses the following rule:

- 20 measurements per sample of foil,
- the emissivity is measured on three foils from the same batch,
- the declared value is based on results for three different batches of production

The overall number of measurements is then $3 \times 3 \times 20 = 180$ measurements.

5. CALCULATION OF THE TOTAL HEMISPHERICAL EMISSIVITY

The emissivity require for calculation of the resistance to heat transfers in an air space is the total hemispherical emissivity (Standard ISO 6946:2017(E)). The value obtained from measurement with an integrating sphere or with the TIR100-2 emissometer is the total near-normal emissivity.

For low emissivity foils, it is recommended to calculate the total hemispherical emissivity using the following relation given in standard EN 12898 - March 2019: " Glass in building - Determination of the emissivity" gives, in Section 6.2 "Corrected emissivity"

$$\varepsilon_{tot\ hem} = 1.1887 \varepsilon_{tot\ NN} - 0.4967 \varepsilon_{tot\ NN}^2 + 0.2452 \varepsilon_{tot\ NN}^3 \quad (6)$$

with $\varepsilon_{tot\ hem}$ the total hemispherical emissivity, and $\varepsilon_{tot\ NN}$ the total near-normal emissivity.

Relation 6 was established "For soda lime silicate glass and coated soda lime silicate glass".

However, the optic theory gives quite similar ratios between the hemispherical emissivity and the normal emissivity for smooth low emissivity surfaces.

The EMIRIM project has shown that relation 6 gave under-evaluated values of the total hemispherical emissivity of four reflective foils tested in this project. Nevertheless, at this stage of knowledge about the relation between the hemispherical emissivity and the near-normal emissivity for reflective foils, not applying relation 6 would give emissivity values even more under-evaluated. From results in EMIRIM project, the expanded uncertainty due to the extrapolation process is about 0.025 (k=2) for reflective foils and it should be combined with the uncertainty on the measured total near-normal emissivity to determine the global expanded uncertainty on the total hemispherical emissivity of the reflective foil.

References :

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