FINAL PUBLISHABLE REPORT

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1. LNE, France
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3. DTU , Denmark
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5. FhG, Germany
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7. IG, Italy
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9. ACTIS, France
10. INGLAS, Germany</td></tr>
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1. Overview

Reflective foil insulations are used in the aerospace, transportation, and power generation industries where protection from thermal radiation is required and in thermal insulation systems for buildings where the EU conformance is required to Directive 2010/31/EU. The European standard EN 16012 describes methods for determining the thermal performance of reflective insulation products for buildings which manufacturers must declare. However, the standard has limited scope due to difficulties in obtaining reliable results for very low emissivity foils from different measurement techniques. Poor technique selection by insulation manufacturers can lead to inaccurate declarations of product properties. This project has developed SI traceable insulation foil emissivity measurements with 0.03 uncertainty which have great potential for inclusion in a revision of EN16012. This will help end users to select appropriate measurement methods more easily during development and certification of new and more efficient reflective insultation products.

2. Need

Reflective foils are used as heat protection screens and materials in many other applications apart from building insulation, e.g. in aircrafts, land vehicles, boats, space crafts, nuclear power generation, packaging, satellites and for protection of people exposed to intense thermal radiations. The principle of reflective insulation relies on the high sensitivity of the radiant heat power exchange between two surfaces not in contact to emissivity. The lower the emissivity of the surfaces, the lower the exchanged radiant power will be. In order to conform to Directive 2010/31/EU on the energy performance of buildings, producers of reflective insulation products must declare values of total hemispherical emissivity for the product’s external surfaces. Declared emissivity values are used, in accordance with standards EN 16012 and ISO 6946, to calculate the thermal resistance of the insulation system under the condition of use. A comparison of measurement techniques organised by the standardisation group CEN/TC89/WG12, responsible for defining test methods and declaration rules for the thermal performance of reflective insulation products, showed high discrepancies with total hemispherical emissivity results ranging from 0.02 to 0.08 on the same reflective foil. These discrepancies can lead to the inadvertent under declaration of emissivity for reflective foils. The comparison included “integrating sphere” instruments and commercially available portable instruments (reflectometers). The sources of discrepancies were not explained, but likely sources were the geometrical, thermal and optical configurations of the measuring instruments and the type of reference sample used for calibration. Being unable to show that the measurement techniques evaluated were reliable when the emissivity is believed to be less than 0.05, CEN/TC89/WG12 set a limitation in EN 16012 that any 'measured' value of emissivity less than 0.05 has to be rounded up to 0.05. This limitation hampers new product development and market innovation since manufacturers cannot achieve the financial return from their investments in products with superior emissivity values below 0.05. To resolve this situation CEN/TC89/WG12 expressed a need for improvements to the accuracy of emissivity measurements of reflective foils used in insulation products. This project has addressed this by analysing carefully the sensitivities of the measurement techniques to the peculiarities of reflective foils, by defining the appropriate types of materials to be used for calibration, by improving reference techniques for calibration of the standards and by producing good practice guides for calibration of the end-users measuring instruments and for emissivity measurement on reflective foils. Recommendations were also given to CEN/TC89/WG12 for improvement of standard EN 16012.

3. Objectives

The overall aim of the project is to improve the measurement techniques recommended by the standards EN 16012 and EN 15976 and applied by end-users for the measurement of total hemispherical emissivity of reflective foils used in “reflective insulation” in order to obtain an uncertainty below 0.03 for emissivity below 0.1.

The specific objectives of the project were:

1. To analyse and test the different techniques and instruments used by end-users to characterise reflective insulation products. The sensitivity of these techniques in relation to the specificities of the reflective foils (specularity, angular diffusion, transparency, spectral properties, thermal inertia and non-flatness of surfaces) will be investigated to enable the definition of the most appropriate types of reference samples for ensuring traceability.
2. To improve and validate reference techniques based on different principles of measurement from at least two NMIs in Europe. The reference techniques will be able to measure total hemispherical emissivity

below 0.1 with an uncertainty below 0.02. They will be applicable to materials with different ratios of specular reflectance/hemispherical reflectance.

3. To build new competencies in Metrology Institutes in order to produce appropriate calibrated reference samples for characterising end-user instruments and for ensuring traceability of measurements, via calibration and measurement procedures developed in the project. Calibrated reference samples will also be produced for partners involved in the measurement techniques characterisation.
4. To establish calibration and measurement procedures enabling the end-users to perform emissivity measurements on reflective foils with an uncertainty below 0.03 for emissivities below 0.1.
5. To participate to the revision of EN 16012 and EN 15976, via the provision to CEN/TC 89/WG12 and CEN/TC 254/WG14 of amendments based on the technical results of the project. To communicate technical reports and guidelines on the calibration and use of end-users techniques to CEN/TC 89/WG12 and CEN/TC 254/WG14. To disseminate the technical results of the project to the wider scientific and industrial community.

1 Results

Objective 1: Analysis and test of the emissivity measurement techniques used by end-users to characterise reflective insulation products.

Introduction - Situation at the beginning of the project

The working group CEN/TC89/WG12 is a working group of CEN/TC 89 responsible for defining test methods and declaration rules for the thermal performance of products that rely upon their highly reflective (or low emissivity) surfaces to provide a portion of their claimed thermal performance. A comparison managed by CEN/TC89/WG12 in 2013 demonstrated high discrepancies in emissivity results which ranged from 0.02 to 0.08 between total hemispherical emissivity measurements obtained on 3 samples of the same reflective foil. The comparison involved “integrating spheres” instruments and commercially available portable emissometers (instruments measuring emissivity). The reasons for the discrepancies were not identified; suspected were the sensitivity of the measurement techniques to the angular distribution of the radiation reflected by the materials, problems with measurement traceability or with the measurement procedures. Most laboratories, certification institutes or manufacturers of insulation products in the EU use integrating sphere systems associated with Fourier transform spectrometers or TIR100-2 commercial emissometers for measuring emissivity of reflective foils. Those two techniques are recommended by standard EN 12898.

Characterisation of INGLAS TIR100-2 emissometers

The TIR100-2 emissometer is a commercial instrument allowing measurements of the total near-normal emissivity of opaque materials from very low to very high emissivity. It is manufactured and distributed by Inglas GmbH & Co.KG in Germany [1]. It measures the hemispherical near-normal reflectance of the sample. A hemispherical high emissivity hemisphere heated at 100°C produces the radiation incident on the sample surface. A radiometric detection system, constituted of an infrared lens and a thermopile detector, measure, the radiation reflected by the sample in a direction defined by an angle of 12° relative to the surface’s normal. The instrument is calibrated using an aluminium polished surface for the low emissivity level (high reflectance) and a black heavily corrugated surface for the high emissivity level (low reflectance). Between the two levels of calibration the response of the instrument is assumed to be linear with reflectance or emissivity. The principle of measurement is described in [1], [).

2] and [

3].

Two TIR100-2 emissometers were lent by INGLAS to LNE and PTB for detailed characterisations; the characterisations performed are described in detail in [).

2] and [

3]. PTB and LNE measured, using different techniques, the size of the measurement spot. PTB showed that 50% and 90% of the radiation collected by the radiation detection system came respectively from an area with a diameter of 10 mm and 20 mm. LNE measured an overall spot size of 24 mm. LNE also analysed the thermal stability of the radiating hemisphere, the uniformity of this temperature, the emissivity of the coating applied on the radiating hemisphere, the linearity of response of the radiometric detection system and the relative spectral sensitivity of the TIR100-2. The results allowed the establishment of an uncertainty budget. Table 1 shows two examples of uncertainty budgets for emissivity measurement on smooth low emissivity foils. The main source of measurement uncertainty, for low emissivity foils is the uncertainty on the total near-normal emissivity of the low emissivity standard used for calibration. The second uncertainty source is the non-uniformity in temperature of the hemisphere generating incident radiation. The evaluation of the uncertainty due to the non-homogeneity of hemisphere temperature is not obvious; it depends on the portions of the hemisphere "seen indirectly" by the radiometric detection system when the reference standard (mirror) is used and when the sample is tested. This means that this uncertainty depends on the flatness and on the angular diffusion at reflection of the sample tested and of the multi-reflections between the sample and the hemisphere. In order to validate the uncertainties assessed, a comparison between four TIR100-2 instruments and the reference radiometric technique from PTB was performed. TIR100-2 emissometers were operated by ACTIS, FIW, PTB and LNE and were calibrated using "Gold mirror" samples and Nextel samples with reference values from PTB. Results obtained on Infragold show that the TIR100-2 emissometer is not critically sensitive to the angular diffusion of the radiation reflected on the sample. Indeed, Infragold is very angular diffusing and Gold mirrors are specular. Measurements were also performed on five reflective foils, three of them being smooth and two having a heavily non-flat surface (the mesh reinforced foils). The results of total near-normal emissivity for the three smooth foils are in accordance with results from the reference technique of PTB. For one of the foils with non-flat surface ("Mesh reinforced foil – Gold color"), the results from three TIR100-2 are also in accordance with results from the reference technique of PTB. For the non-flat foil "Mesh reinforced foil – Alu color", the results from the four TIR100-2 instruments are well grouped but significantly lower than the ones from the reference technique of PTB; the difference is not explained.

Source of uncertainty	Uncertainty on the measured total near-normal emissivity (k=2)	
	Bare metal foil ε_{meas} = 0.023	Smooth polyethylene metalised foil ε_{meas} = 0.038
Non-stability of the hemisphere temperature	0.005	0.005
Non-uniformity of the hemisphere temperature	0.017	0.017
Multi-reflections between the sample and the hemisphere	0.0006	0.0014
Radiometric response linearity	0.003	0.003
Non-flat spectral response	0.0006	0.0062
Uncertainty on total near-normal emissivity of the low emissivity reference sample	0.022	0.022
Variation of the sample temperature (6 K)	0.0032	0.0056
Repeatability	0.004	0.006
Non-uniformity in emissivity	0.0044	0.010
Expanded combined uncertainty (k=2)	**0.028**	**0.032**

Table 1: Examples of uncertainty budget for measurement of total near-normal emissivity of reflective foils with TIR100-2 emissometers

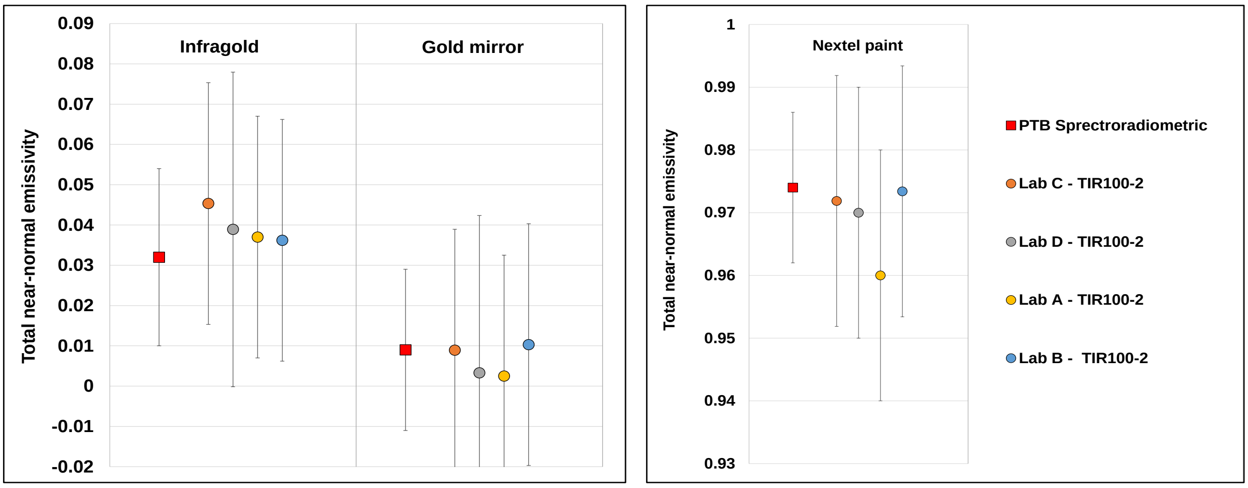


Figure 1: Comparison of total near-normal emissivity results obtained with TIR100-2 emissometers on Gold mirror, Infragold and Nextel paint

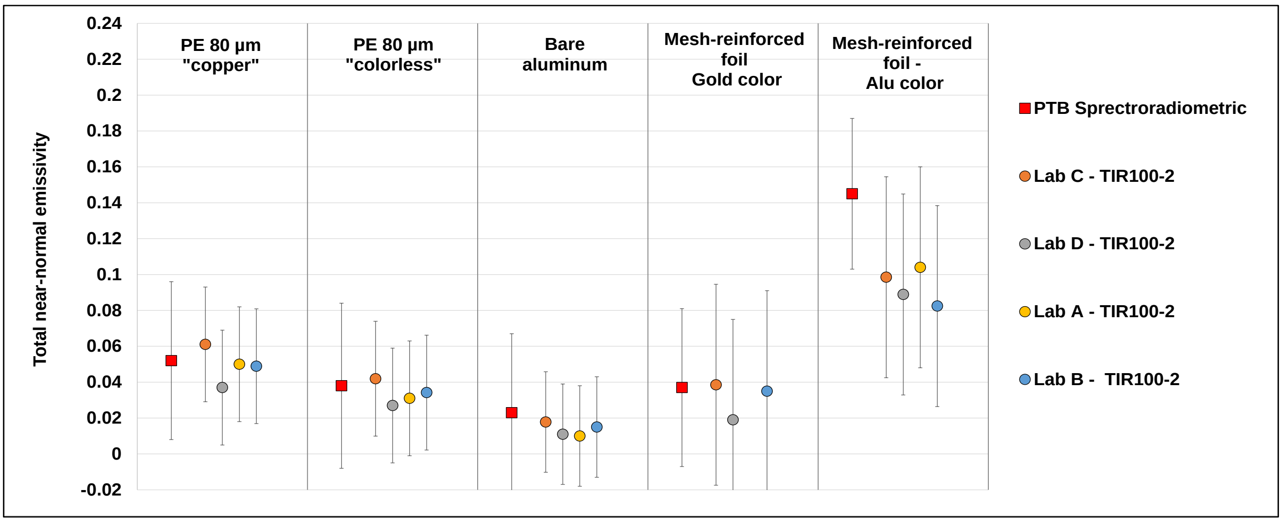


Figure 2: Comparison of total near-normal emissivity results obtained with TIR100-2 emissometers on reflective foils tested in EMIRIM

Conclusions: TIR100-2 emissometers calibrated with a specular reference (metal mirror) have limited sensitivity to the specularity of the sample tested and can measure total near-normal emissivity of reflective foils below 0.1 with an uncertainty below 0.3.

Characterisation of integrating spheres

Integrating spheres used in the infrared region are, most of the time, associated with infrared Fourier transform spectrometers (FTIRs). The parameter measured is the spectral near-normal hemispherical reflectance, and for opaque materials the spectral near-normal emissivity is calculated from the measured spectral reflectance. Integrating spheres must be calibrated for each measurement campaign typically with only one low emissivity standard either a mirror or a diffusing surface such as an Infragold® coating.

In the EMIRIM project, LNE characterised the angular variation of sensitivity of an integrating sphere (diameter 75 mm). The results showed that relative local variations of a few percent can exist around the specular direction; the integrating sphere tested has a "specular port". The source of variation of the local sensitivity is the space between the sphere and the specular port plug. Even if the space is narrow, its low reflectance is not negligible. The results obtained show clearly that an integrating sphere with a "specular port" can generate relative errors of a few percent on the reflectance measured when testing surfaces with some diffusion around specular direction. This error depends on the optical configuration of the integrating sphere (geometry of incident beam) and is specific to each setup. LNE showed that the relative sensitivity of their integrating sphere decreases linearly with the reflectance of the sample which generates an error if no correction is applied. This source of error called the "single-beam sample-absorption error", is already known but several users of integrating spheres are not aware of it [

4] or the "single beam substitution error" [

5]. It is possible to correct the error by applying a specific procedure of measurement (§ 0). Usually the spectral domain of measurement with an integrating sphere is limited for the longer wavelengths. The application of emissivity results for heat balance modelling in buildings would require spectral measurements up to 50 µm and most of time the spectral domain of measurement is limited at about 20 µm to 35 µm for the longer wavelength. Therefore, an assumption must be made for the spectral emissivities at high wavelengths. The assumption can be the stability of spectral emissivity at long wavelengths or a specific spectral curve defined by the operator. An uncertainty must be considered for the extrapolation of spectral emissivity at long wavelengths. LNE quantified the uncertainty related to extrapolation for the reflective foils tested in EMIRIM project. For low emissivity foils an uncertainty for extrapolation below 0.005 (k=2) is reasonable when spectral emissivity is measured at least up to 25 µm.

LNE established an uncertainty budget for the determination of total near-normal emissivity calculated from spectral near-normal emissivity measurements performed with an integrating sphere. An example uncertainty budget is given in Table 2. Data used for quantification of uncertainties were supplied by LNE, ZAE Bayern and IG. The main source of uncertainty is the uncertainty on the spectral near-normal emissivity of the calibrated standard used for calibration.

A comparison of measurements with four different integrating spheres from LNE, ZAE Bayern, IG and DTU was performed, the results are shown in Figure 3 and Figure 4; the setups were calibrated with gold mirror standards. For three integrating spheres the results for Infragold® showed a sensitivity to the angular dispersion of reflected radiation. The differences are about 0.02 from reference value from PTB for two integrating spheres and 0.044 for one integrating sphere. A detailed analysis of the optical configurations of the three integrating spheres showed that the optical configurations were not optimum. For the smooth foil "PE 80µm copper" one of the integrating spheres gave a result differing by 0.024 from the reference result from PTB. For the non-smooth mesh reinforced foils, the differences between the results from three integrating spheres are scattered and can be quite different from the reference results from PTB.

Uncertainty source	Uncertainty on measured total near-normal emissivity ($\varepsilon \leq 0.1$)
	Measurement spectral range: 5 µm to 25 µm
Uncertainty on measured spectral emissivity	0.023
Extrapolation of near-normal spectral emissivity up to 50 µm	0.0046
Short term stability of "integrating sphere and FTIR"setups	0.0030
Non-linearity of response due to multireflections (correction procedure is applied)	0
Non-uniformity in emissivity	0.004
Expanded combined uncertainty (k=2)	0.024

Table 2: Example of uncertainty budget for measurement of total near-normal emissivity on reflective foils with integrating spheres having correct optical configuration when a procedure for correction of non-linearity of response is applied

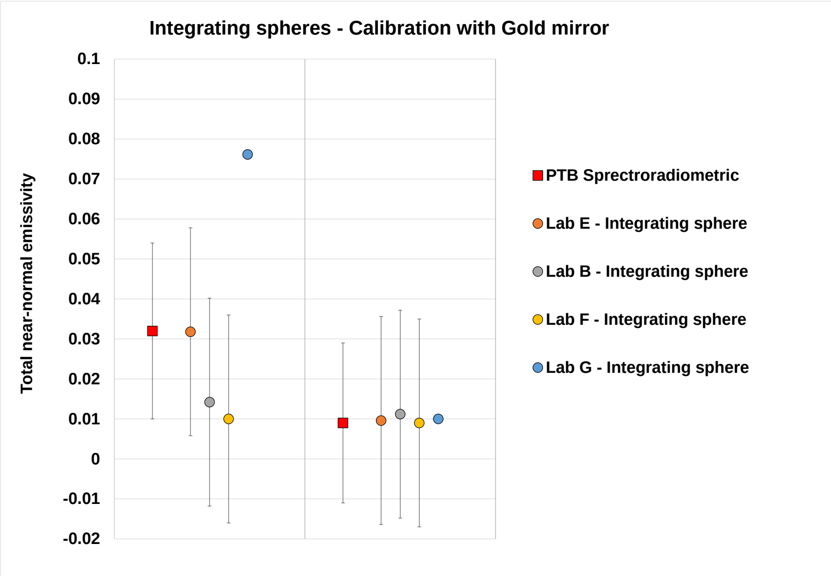


Figure 3: Comparison results on Infragold® and Gold mirror

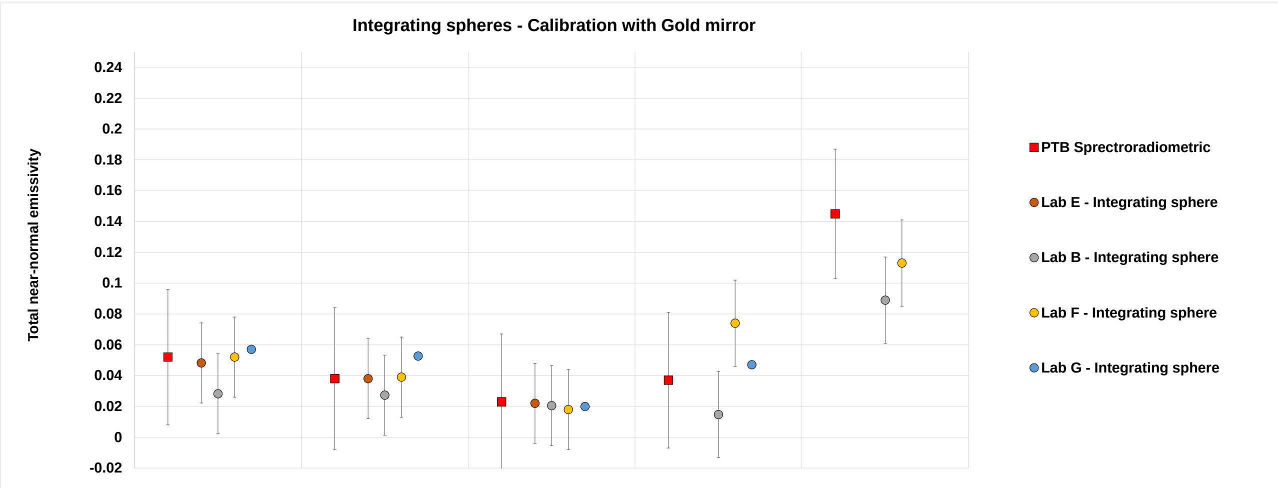


Figure 4: Comparison of total near-normal emissivity results obtained with integrating spheres on reflective foils tested in EMIRIM

Conclusions: For testing low emissivity foils, an integrating sphere with a poor optical configuration can be sensitive to the angular dispersion of the radiation reflected by the sample. As a consequence, using a specular or a diffusing standard can have a significant effect on the measurement result. The control of the optical configuration is important for ensuring the quality of measurements. For heavily non-smooth foils, the results can have a significant spread. This is due partly to the non-uniformity in emissivity of those foils but also to the quite small spot size and to a sensitivity to the local orientation of the area tested. With a correct optical configuration, the total near-normal emissivity of a smooth reflective foil can be measured with an uncertainty of 0.025 (k=2). The use of two calibrated standards one very specular and one very diffusing, allow the detection of a sensitivity to the angular dispersion of the radiation reflected by the sample.

Conclusions for TIR100-2 emissometers and infrared integrating spheres

TIR100-2 emissometers and integrating spheres associated to Fourier transform spectrometers can be used to measure total near-normal emissivity of smooth reflective foil with uncertainty below 0.03.

The parameter measured is the total near-normal emissivity and not the total hemispherical emissivity which is the parameter required for heat balance modelling of buildings.

When using integrating spheres for testing reflective foils, special attention must be paid to the optical configuration of the setup.

Objective 1 is fully met; the detailed analysis of end-user techniques (TIR100-2 emissometers and integrating spheres) showed that metal mirrors are suitable for calibration of both techniques. Special attention must be paid to the optical configurations of integrating spheres to perform reliable measurements on reflective foils.

Objective 2: Improvement and validation of reference techniques for measurement of total emissivities below 0.1 with an uncertainty below 0.02

Measurement techniques mostly used by end-users must be calibrated in total near–normal emissivity or in total hemispherical emissivity. The end-user techniques mostly used in the EU and analysed in the EMIRIM project are integrating spheres and the INGLAS TIR100-2 emissometer. The integrating spheres require, at least, a high-reflecting calibrated standard for traceably establishing the sensitivity in spectral near-normal hemispherical reflectance. TIR100-2 emissometers require a low emissivity and a high emissivity standard calibrated in total near-normal emissivity.

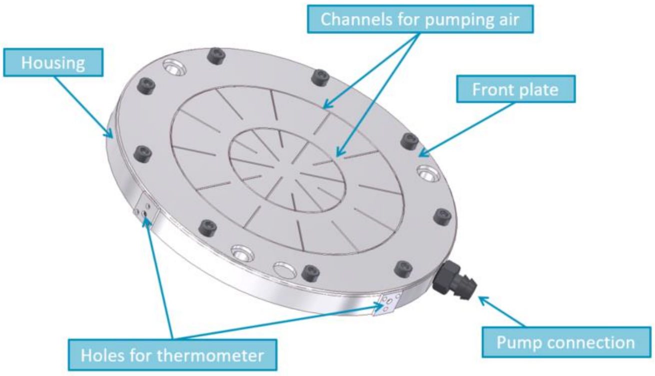
The main objective of the project being that end-users are able to measure total emissivities of low emissivity materials with an uncertainty of 0.03 (k=2), the objective set for the reference techniques was an uncertainty below 0.02 for materials with emissivity below 0.1 and with different ratios of specular reflectance to hemispherical reflectance. The improved setups should also allow the emissivity measurement of thin reflective foils for validation of end-user measurement techniques.

Reference technique at PTB

PTB routinely determines directional spectral and hemispherical total emissivities of solid materials with low uncertainties in air and under vacuum conditions. The PTB setup (EMAF) for directional spectral emissivity measurement in air is presented in detail in [6]. The measurement expanded uncertainty is below 0.015 for total emissivities measurements on solid surfaces with a high emissivity. The measurement technique is a direct technique based on comparison of the spectral directional radiance of the sample surface to the spectral radiance of a blackbody. The radiometric measurements are performed using a Fourier Transform Spectrometer (FTIR). 6The measurement of crumpled foils is challenging because of the limited thermal contact of such samples with the sample heater used for solid samples. Their poor thermal contact results in an uncertain and inhomogeneous sample surface temperature and this will dominate the overall measurement uncertainty. Therefore, the technical improvement made by PTB was the construction of a new sample holder, based on the principle of vacuum mounting to ensure a good and uniform thermal contact of the foil with the heater over all the heater area. This fixing technique allows also the free expansion and retraction of the sample. The new sample heater was designed to fit in the existing spherical enclosure surrounding the sample and to allow high-precision positioning of the sample for focusing of the FTIR-spectrometer. This is required for measurements at different angles and for applying the existing evaluation models for the calculation of the radiation budget and the consideration of multiple reflections between sample and the spherical enclosure which is very critical in the case of highly reflecting insulation materials.

The construction of the new sample holder and the sample holder in operation is shown in Figure 5. The new vacuum sample holder in operation can be seen in Figure 5. The hemispherical enclosure is presented here in opened position for illustration. The smooth surface of a foil in the foreground indicates excellent fixation of the sample on the vacuum sample holder.

PTB performed spectral directional emissivity measurements on a set of reflective foils presented in Figure 6. The results are given in Table 3.



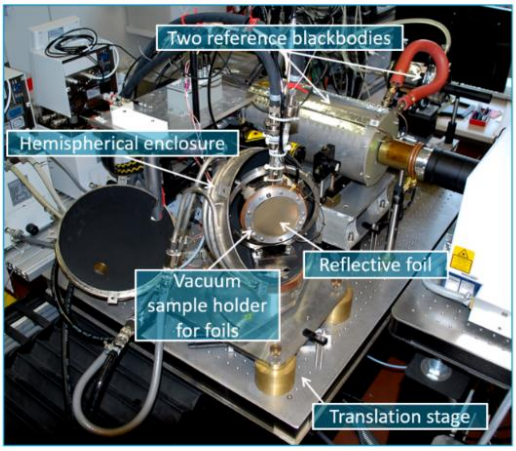


Figure 5: Left: New vacuum sample holder for emissivity measurements on thin films.
Right: Photograph of the setup for emissivity measurements in air (EMAF).

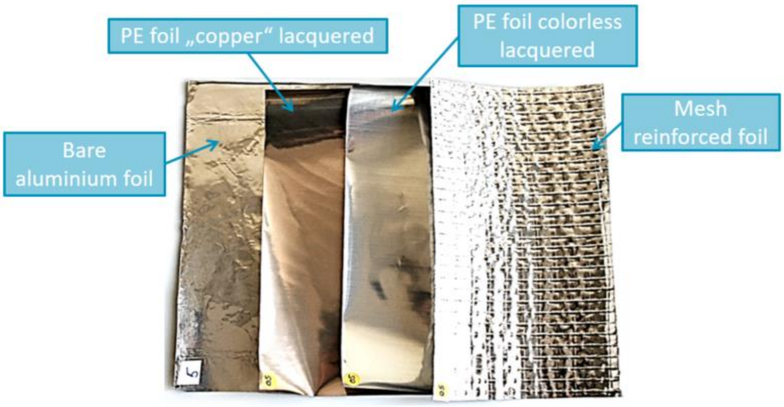


Figure 6: Photograph of the selected foils: Bare aluminum foil, PE foil „copper" lacquered, PE foil colorless lacquered and Mesh reinforced foil

Angle	Bare aluminum ε(25 °C)	$u(\varepsilon)$	PE foil colorless ε(25 °C)	$u(\varepsilon)$	PE foil "copper" ε(25 °C)	$u(\varepsilon)$	Mesh reinforced ε(25 °C)	$u(\varepsilon)$
20°	0.023	0.022	0.038	0.023	0.052	0.022	0.145	0.021
30°	0.017	0.022	0.040	0.023	0.051	0.023	0.145	0.021
40°	0.018	0.022	0.040	0.022	0.053	0.022	0.162	0.022
50°	0.043	0.022	0.071	0.022	0.088	0.022	0.167	0.021
60°	0.041	0.021	0.108	0.022	0.094	0.021	0.186	0.021
70°	0.053	0.022	0.046	0.024	0.126	0.021	0.213	0.020
ε **hem**	**0.039**	**0.019**	**0.070**	**0.022**	**0.085**	**0.022**	**0.179**	**0.020**

Table 3: Directional total and hemispherical total emissivities of the reflective foils in the wavelength range from 5 µm to 20 µm with their corresponding standard uncertainties

Conclusions: The new sample heater for spectral and total directional emissivity measurement of thin low emissivity foils allows measurements with standard uncertainties below 0.024 (k=1). This low uncertainty is allowed by a good thermal contact of the sample with the heater inducing a low uncertainty on the surface temperature of the sample. The EMAF setup from PTB was also used in the EMIRIM project for calibrating the solid reference samples with an expanded uncertainty below 0.022 (k=2), which were used to calibrate the end-users emissivity measurement instruments (integrating spheres and INGLAS TIR100-2 emissometers). Another interesting feature of the EMAF setup is the ability of the instrument to measure directional emissivity over a large angular range (10° to 70°). This gave valuable information useful for evaluating uncertainties related to the extrapolation of total hemispherical emissivity from total near-normal emissivity.

Reference technique at LNE

In the EMIRIM project, LNE improved a calorimetric technique for direct measurement of total hemispherical emissivity with an uncertainty below 0.02 on materials with an emissivity below 0.1 and with different ratios of specular reflectance to hemispherical reflectance. The principle of measurement is presented in [7]. The calorimetric technique of measurement is based on the heat balance of two samples, with disc shape, radiating in a very cold blackbody. The electrical power used to maintain the two samples at a steady temperature, the areas of the two circular surfaces radiating toward the blackbody, the surface temperature of the samples and the temperature of the blackbody cavity allow the calculation of the total hemispherical emissivity including all wavelengths and all directions.

At the beginning of the project, LNE had an old sample heater with poor metrological performance particularly regarding the sensitivity to the uncertainty on the balance between the sample’s temperature and the temperature of the thermal guard rings encircling the samples. Therefore, in the EMIRIM project, the technical objective was the construction of a new sample heater with metrological performance ensuring total hemispherical emissivity measurements with uncertainty below 0.02 for low emissivity surfaces.

LNE built a sample heater allowing the use of two samples (disc shape) with a diameter of 100 mm instead of 62.2 mm for the old heater. The radiating area is then multiplied by more than 2.5 reducing the relative influence of parasitical heat fluxes. The new heater was also designed to reduce significantly the heat transfer coefficient by thermal conduction between the samples and the thermal guard rings. A diagram of the heater is shown in Figure 7. For solid materials, the samples are discs with a diameter of 100 m and a thickness of 5 mm. A hole is bored radially in each sample for insertion of a thermocouple. The samples are pressed onto the sample heater using three clamps for each sample to get a uniform thermal contact with the heater. The thermal contact is also improved by using a small amount of thermal grease. For reflective foils, the two samples of the foil are bonded onto two metal discs made of aluminium alloy. The bonding is done by using thin double-sided tape evenly distributed over the entire surface of the discs. Figure 7 shows a sample of a foil mounted on the heating system.

The first tests on the foils showed that there was a risk of blistering between the film and the heater particularly under vacuum. The procedure used for avoiding blistering consists to puncture the foil and the tape with very small holes approximately each square centimetre of area. The overall area affected by the punctures remains small enough to not affect the mean emissivity of the sample.

A detailed uncertainty budget was established; an example of uncertainty budget is given in Table 4. For low emissivity materials the main sources of uncertainty are the parasitical heat fluxes between the samples section and the thermal guard section and the heat losses through the gas remaining in the chamber (pressure $< 10^{-2}$ Pa). For measurements on low emissivity foils with a total hemispherical emissivity below 0.1, the expanded uncertainty on the total hemispherical emissivity result is below 0.02. This uncertainty is valid only if the bonding of the foil on the metal substrates doesn't significantly change the surface of the foil tested.

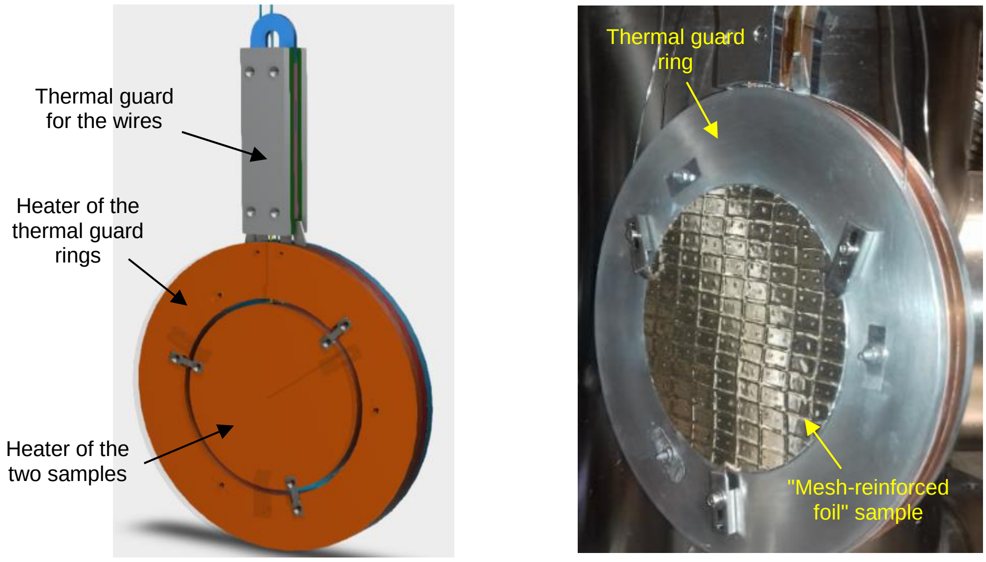


Figure 7: Left: View of the heater; Right: Photo of a sample (Mesh reinforced foil) mounted on the sample heater

Conclusions: LNE built a new thermal guarded sample heater for measuring directly total hemispherical emissivity of solid materials and of thin foils. The new sample heater makes it possible to test samples with radiating area more than twice as large as the area enabled by the previous heater. The new sample heater was designed in order to reduce the influence of temperature imbalance between the samples and the thermal guard rings. The expanded uncertainty estimated for the measured total hemispherical emissivity is 0.015 (k=2) for low emissivity solid materials and 0.020 (*k*=2) for low emissivity foils. The calorimetric technique is applicable to materials with any ratios of specular reflectance to hemispherical reflectance and gives directly the total hemispherical emissivity without any extrapolation process.

Parameter or model	**Value**	**Standard uncertainty (k = 1)**	**Induced expanded uncertainty on measured emissivity (k=2)**
Area of the samples	0.015708 m^2	3.14 . 10^{-5} m^2	0.00011
Area of the chamber	1.36014 m^2	0.06 m^2	3 . 10^{-7}
Area of the samples rings	0.03218 m^2	8.10^{-5} m^2	1.5 . 10^{-8}
Mean total hemispherical emissivity of the chamber walls	0.92	0.025	5.5 . 10^{-6}
Mean total hemispherical emissivity of the guard rings	0.05	0.02	1.8 . 10^{-6}
Measurement of electrical power	0.20732 W	2.6 10^{-4} W	1.2 . 10^{-5}
Mean surface temperature of the samples	30.00 °C	0.5 °C	2.7 . 10^{-4}
Mean temperature of the chamber walls	77.36 K	1 K	8.8 . 10^{-6}
Mean surface temperature of the rings	30.00 °C	1 °C	6 . 10^{-8}
Edge heat loss by radiation	0.01498 W	0.0038 W	0.001
Edge heat loss by conduction between the samples section and the thermal guard section	0.000 W	0.040 W	0.011
Heat loss by air remaining in the chamber	0.0426 W	0.022 W	0.0057
Model used for emissivity calculation	-	2.5 .10^{-5}	2 . 10^{-5}
Random variations of measured emissivities	-	0.001	0.002
Measured total hemispherical emissivity	**0.020**	**0.0075**	**0.013**

Table 4: Budget of uncertainty for measurement of total hemispherical emissivity of a solid material with an emissivity of 0.02

Reference technique at DTU

DTU developed a new setup for measuring the directional emissivity over a wide spectral band. Figure 8 shows a diagram of the setup. The radiation leaving the surface of the sample along the direction of measurement is measured using a wide band radiometer with a thermopile detector sensitive in the spectral band 0.6 to 40µm. The radiometer is calibrated as a wide band radiation thermometer using a reference blackbody. The sample to be tested in emissivity is attached onto a sample holder which temperature is controlled by a Peltier thermoelectric module. When testing foils, a sample of the foil sample is glued to an aluminium plate fixed on the sample holder (Figure 9). The sample holder is supported by a rotating mechanism in a chamber with walls very uniform in temperature and with steady temperature.

For directional total emissivity measurements, the sample is heated step by step in 10 °C increments, from room temperature up to about 60 °C and the apparent temperature of the sample surface is measured for different angles in function of temperature. During measurements the temperature of the chamber is maintained constant. The total directional emissivity of the sample is assumed to be constant over the sample temperature range covered for measurements. The emissivity value of the sample is then found by fitting the apparent temperature curve of the sample surface given by a model to the apparent temperature curve obtained experimentally. The model giving the apparent surface temperature integrates the measured surface sample temperature (extrapolated from the temperature measured in the plate supporting the foil) and the temperature of the chamber walls.

DTU estimated an expanded uncertainty (k=2) of 0.012 for total directional emissivity on a low emissivity uniform solid surface or on a low emissivity uniform smooth foil. DTU experimented thermal contact defects when testing structured (mesh reinforced) foils.

Conclusions: DTU built a new setup for measurement of "total" directional emissivity of solid materials and of reflective foils. The direct technique of measurement is used by comparison of the broad spectral band directional radiance of the sample to the radiance of a blackbody. The radiometric measurement is done with a wide-band thermopile detector. For the version of the setup built in EMIRIM project, the maximum angle of measurement is about 50° to the normal to the surface of the sample. This limit angle is too small to allow a good extrapolation of total hemispherical emissivity from measured total directional emissivity for various angles.

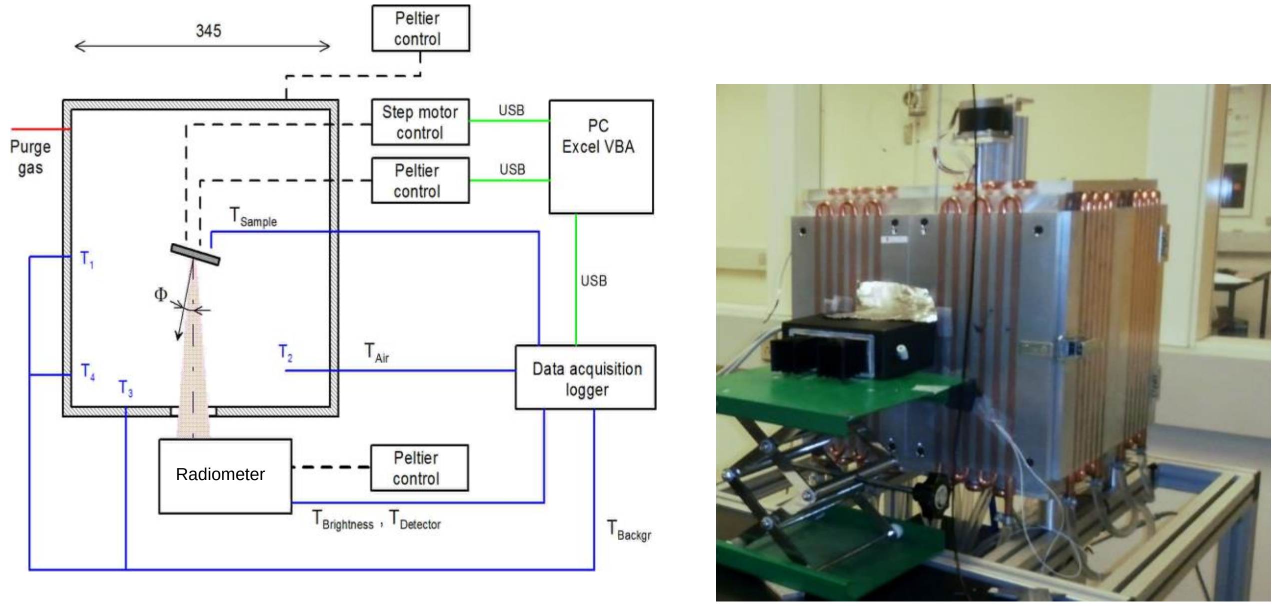
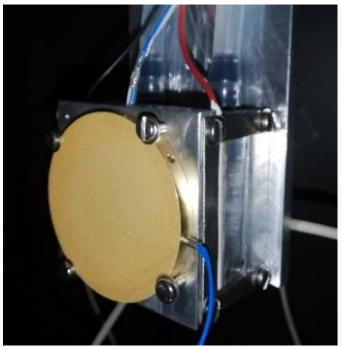


Figure 8: New DTU emissivity setup for fast automated measurements of directional emissivity



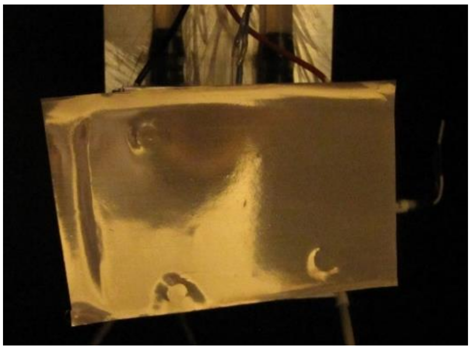


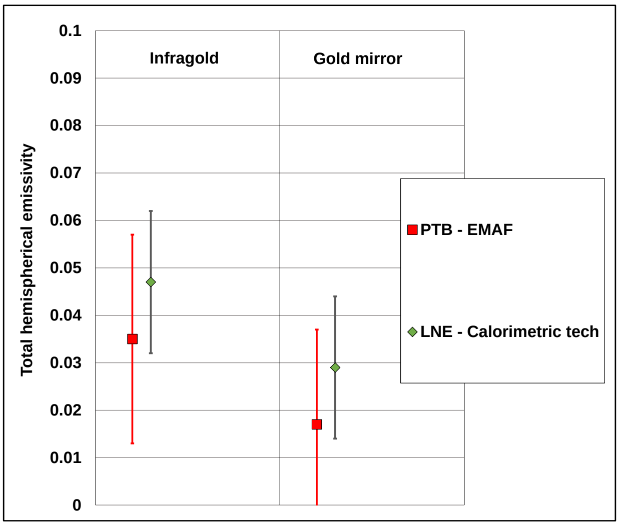
Figure 9: Left: solid Infragold sample attached to the sample holder;
right: foil sample attached to the sample holder

Comparison of the improved Reference techniques

PTB and LNE performed total hemispherical emissivity measurement on the solid low emissivity materials Infragold® and gold mirror and on the high emissivity Nextel Velvet black coating 811-21. The results are compared graphically in Figure 10.

PTB and DTU performed total near-normal emissivity measurement on the same materials. The results are compared graphically in Figure 11.

For total hemispherical emissivity measurements on low and high emissivity solid materials, the results from PTB and from LNE are in good agreement. For total near-normal emissivity and total directional emissivity measurements for angles from 10° to 50°, PTB and DTU obtained results in good agreement.



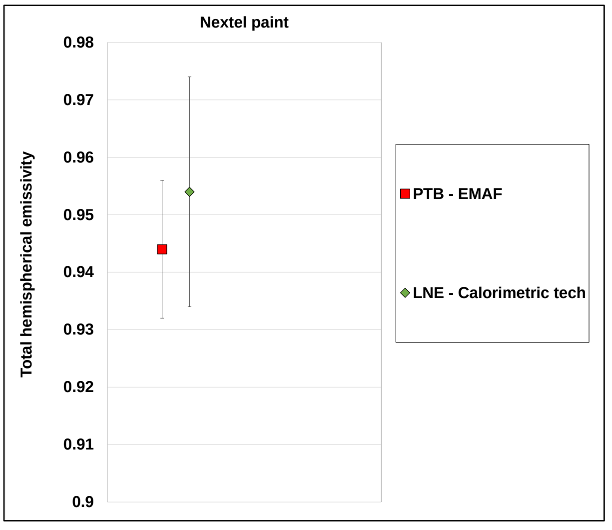


Figure 10: Comparison of total hemispherical emissivities measured on low emissivity and high emissivity solid reference materials

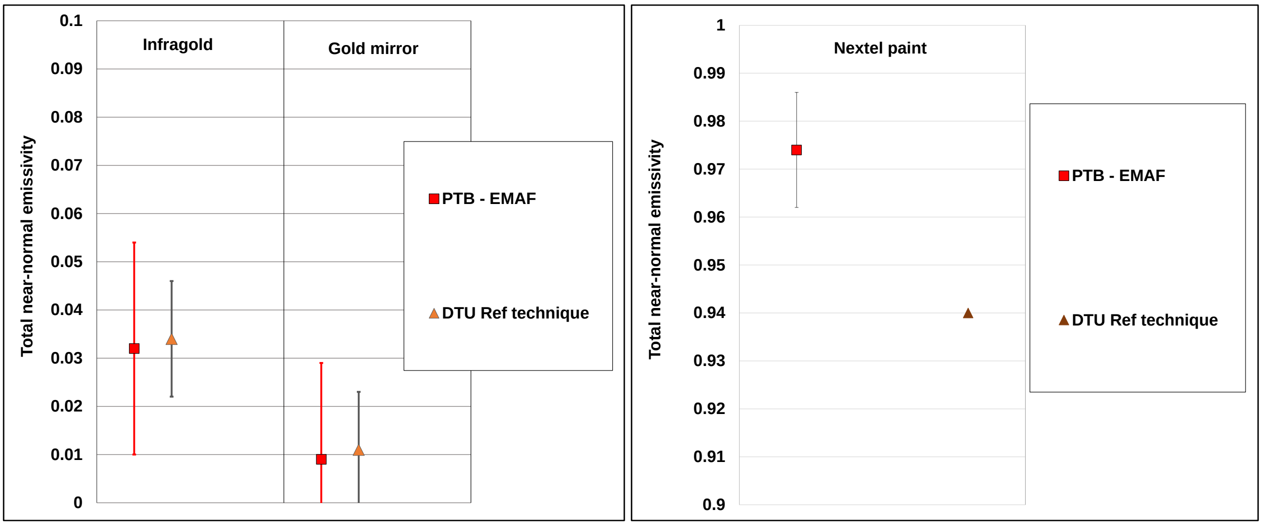


Figure 11: Comparison of total near-normal emissivities measured on low emissivity and high emissivity solid reference materials

Conclusions for the work performed in EMIRIM on the reference techniques for measurement of total emissivities

The objectives set for the EMIRIM project regarding the reference techniques have been met. At least two national metrology laboratories in the EU are able to perform total hemispherical emissivity measurements on low emissivity solid materials and on reflective foils with uncertainty approximate to 0.02 whatever the ratio of specular reflectance to hemispherical reflectance (more or less angular diffusing surfaces).

PTB is able to calibrate reference samples in spectral and total near-normal emissivity with expanded uncertainty around 0.02 (k=2). Therefore, users of integrating spheres or of INGLAS TIR100-2 emissometers have access to calibration services for the reference standards required by those techniques.

The capability of PTB to perform directional emissivity measurements over a wide angular range and the capability of LNE to perform total hemispherical emissivity measurements are valuable to start future work for evaluation of uncertainties related to the calculation of total hemispherical emissivity from the total near-normal emissivity which is the parameter provided by the integrating sphere techniques and by the INGLAS TIR100-2 emissometers.

Objective 3: Production of appropriate calibrated reference samples for characterising end-user instruments and for ensuring traceability of measurements

Emissivity measurement techniques mostly used by end-users in the EU for testing reflective foils are integrating spheres and the INGLAS TIR100-2 emissometers. These techniques measure the near-normal hemispherical reflectance of the sample and must be calibrated before each measurement session. Integrating spheres associated to Fourier transform spectrometers must be calibrated in spectral near-normal hemispherical reflectance and TIR100-2 emissometers must be calibrated in total near-normal hemispherical reflectance.

The reflective foils to be characterised in emissivity have innumerable surface structures and show visually very variable angular diffusion characteristics for reflected light depending on surface structure. At the beginning of the project, it was suspected that the emissivity measurement techniques were potentially sensitive to the angular distribution of the radiation reflected by the sample tested. In order to calibrate the measurement setups of the partners involved in emissivity measurements in the project and to analyse the sensitivities of the setups to the angular diffusions of the samples, different types of samples were produced and calibrated at the beginning of the project.

Production and calibration of solid reference samples for tests of performance of end-user emissivity measurement techniques and of improved reference techniques

PTB and LNE produced two types of very reflective solid samples with very different angular distributions of reflected radiation. The low emissivity specular (non-diffusing) samples were made of polished aluminium substrates coated with a pure gold layer and overcoated by a thin layer of Y_2O_3 for protection. The low emissivity diffusing samples were made of aluminium substrates coated with Infragold® by Labsphere (USA). Those materials (gold mirrors and Infragold®) are usually used for calibrating infrared reflectometers such as integrating spheres. High emissivity samples were also produced by PTB for calibrating TIR100-2 emissometers; these constisted of an aluminium substrate coated with the well-known high emissivity Nextel velvet 811-21 black paint [8]. PTB calibrated the samples in spectral directional emissivity using their EMAF setup for various angles up to 70° to the normal to the surface (Figure 12).

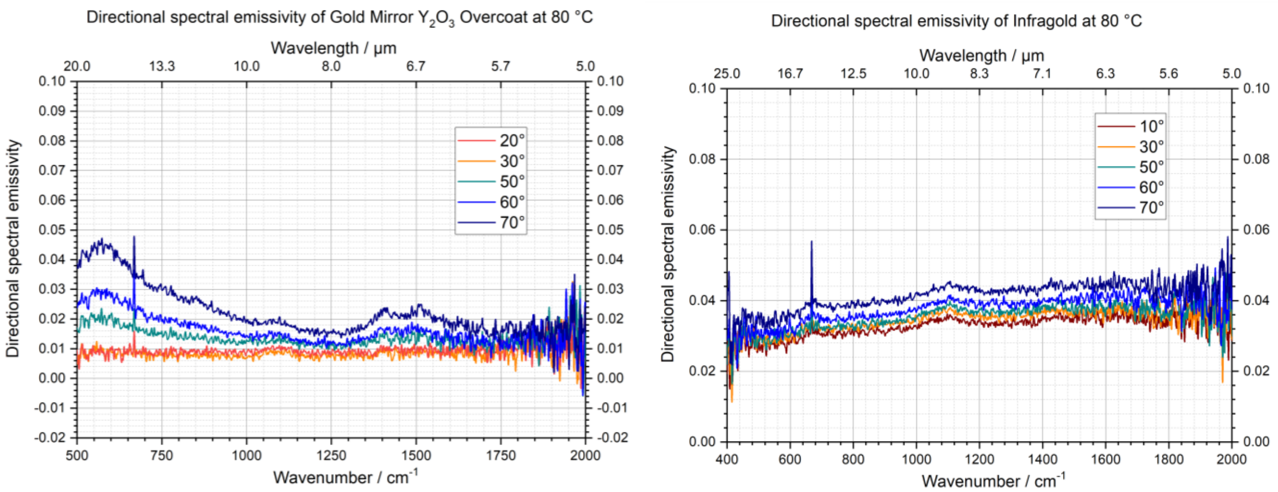


Figure 12: Left: Spectral directional emissivity curves obtained by PTB on a Gold mirror sample – Right: Spectral directional emissivity curves obtained by PTB on the Infragold® coating.

In order to test the linearity of response of end-user's emissivity measurement techniques, PTB also produced samples with "medium" emissivity. It is not easy to find materials having medium emissivity and quite flat spectral emissivity curves. This can be obtained by roughening or structuring the surface a low emissivity material (e.g. a metal). PTB structured the surface of aluminium substrates using a laser engraving technique. A photograph of the structure is shown in Figure 13; the 100-micrometer structure consists of aligned pyramids with an additional depression at the top and grooves between pyramids. After engraving, the surface was gold plated. PTB measured the spectral directional emissivity for several angles, the curves are shown in Figure

13. The emissivity of the gold-plated laser structured samples is about 0.2 and the spectral variations are limited in the spectral range 5 µm to 24 µm. However, reproducibility of emissivity measurements over several samples or over the surface of some samples showed that the emissivity is not reproducible enough for the application. This is probably due to the poor geometrical reproducibility of each element of the structure and to the poor reproducibility of the very local roughness of surface elements.

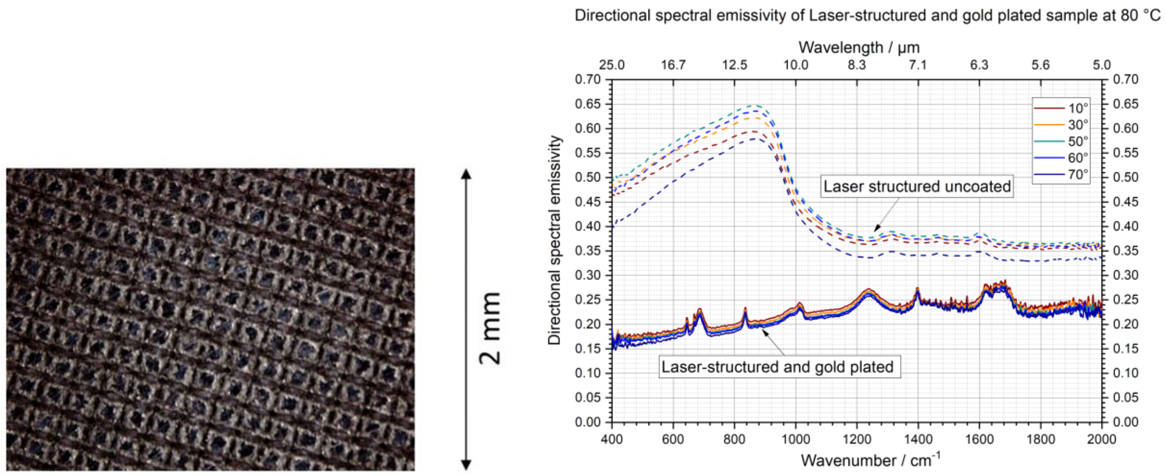


Figure 13: Left: Microscope image of the laser-structured aluminium substrate – Right: spectral directional emissivity curves of the gold plated laser structured samples.

In the EMIRIM project, PTB and LNE have produced 11 sets of reference samples. For calibrating measurement setups with integrating spheres disc shaped samples with a diameter of 50 mm were produced. For testing INGLAS TIR100-2 instruments, disc shaped samples with a diameter of 100 mm to cover the opening of cavity were produced. For the calorimetric reference technique of LNE, for each material two discs with a diameter of 100 mm were required.

Production and calibration of solid samples with specific angular distribution of reflected radiation

When compiling the technical protocol used in the project, it was thought that solid reference samples with specific surface structures could be required for calibrating end-user measurement techniques. Therefore work for producing specific solid samples with low emissivity and angular distribution of reflected radiation similar to the angular distribution of some existing reflective foils was carried out.

The work started by the experimental analysis of the angular distribution of the radiation reflected by some selected reflective foils. LNE measured the angular distribution of the radiation reflected by the foils tested in the project using an existing goniometer usually used to measure spectral reflectance of mirrors. The principle of the measurement is shown in Figure 14. The detection system, constituted of a small integrating sphere (∅ 75 mm) and of an infrared detector, is displaced by the goniometer around the centre of the sample. The variation of the measured signal with the angle depends on the angular distribution of the radiation reflected by the material tested.

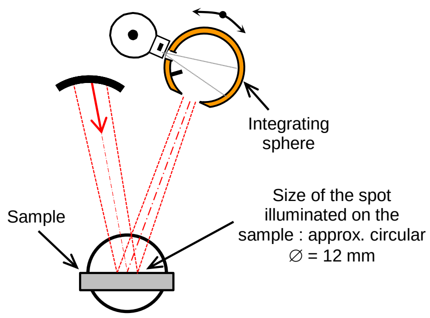


Figure 14: Principle of the goniometric system used to analyse the angular distribution of the radiation reflected by a sample in the plane of incidence.

Figure 15 shows examples of relative angular distribution curves obtained experimentally on a smooth foil and on a foil with a very structured surface. For the smooth foil, the angular distribution of reflected radiation is not dependent on wavelength and is confined within an angle of ± 2 to ± 3 degrees around the specular direction. For the very structured foil the reflected radiation is distributed over a much wider angular range (an angle of ± 40 degrees). These results show that the angular distribution depends highly on the macroscopic surface structure (the morphology) of the foil.

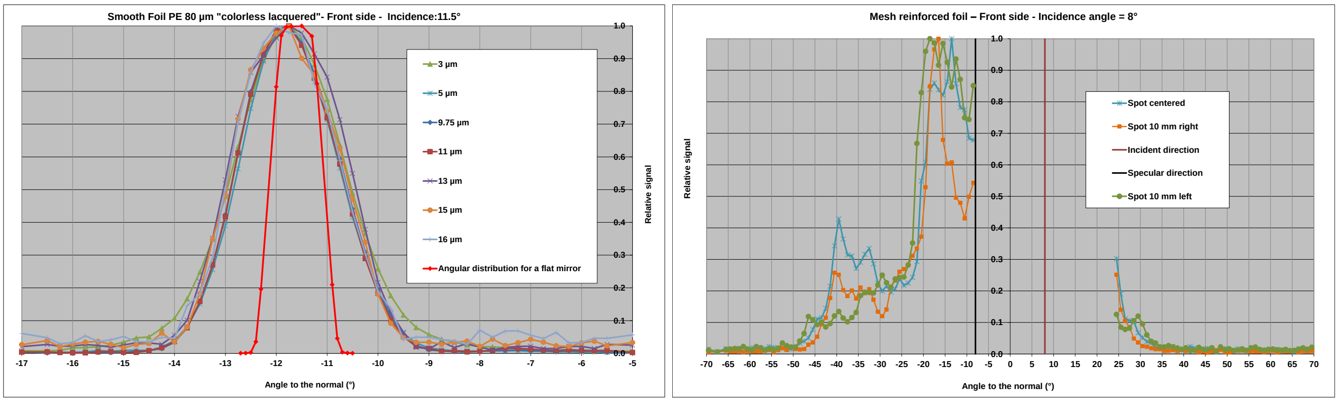


Figure 15: Left: Relative angular distribution curves of the radiometric signal obtained on a smooth reflective foil – Right: Relative angular distribution curves obtained on a foil with a very structured surface ("mesh reinforced foil" seen in Figure 7).

Once the angular distribution functions of reflected radiation were available, Aalto performed optical modelling in order to find the surface structure of a solid surface that would give the same angular diffusions as the foils selected. The modelling work of Aalto is presented in detail in [9].

For the smooth foils showing limited angular dispersion, Aalto found that a Torrance–Sparrow reflectance model describes well the reflectance. This model, based on geometrical optics, describes the surface as a number of microfacets oriented according to a specified distribution. But the characteristics found for the appropriate surface cannot be produced by a mechanical mean, indeed the mean profile amplitude of the appropriate surface is around Ra = 0.25 µm. For the very structured foil ("Mesh reinforced foil"), Aalto found that a wavy surface, locally specular, with a peak to peak amplitude of 500 µm and a wavelength about 7 mm would give an angular dispersion similar to the one obtained experimentally in the plane of incidence when this plane is perpendicular to the waves. But a wavy surface would give a very non-isotropic reflectance. In order to define a surface with a more isotropic behaviour, Aalto generated a theoretical surface structure with bell-shaped elements (Z-axis axisymmetric) arranged in a hexagonal configuration. Figure 16 shows a virtual view of such a surface and the shape of the bell-shaped "cavities".

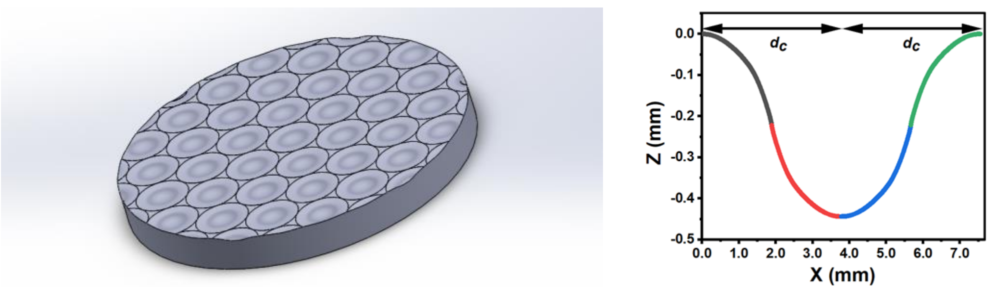


Figure 16: Left: Surface of a sample 50 mm in diameter with bell shape cavities arranged in hexagonal configuration – Right: Cross section view of a bell-shape "cavity" [9].

Production of solid sample with specific surfaces

Based on the surface morphologies defined by Aalto, Fraunhofer IPK produced two solid reference samples with structured surface: one with coarse structure and another one with fine structure with double density (Figure 17). A flat surface sample machined using the same process as the structured samples was also produced. The samples were made by precision milling of aluminium discs followed by a light polishing and are of round shape with a diameter of 50 mm and a thickness of 10 mm. Each sample has a radial hole for a temperature sensor.

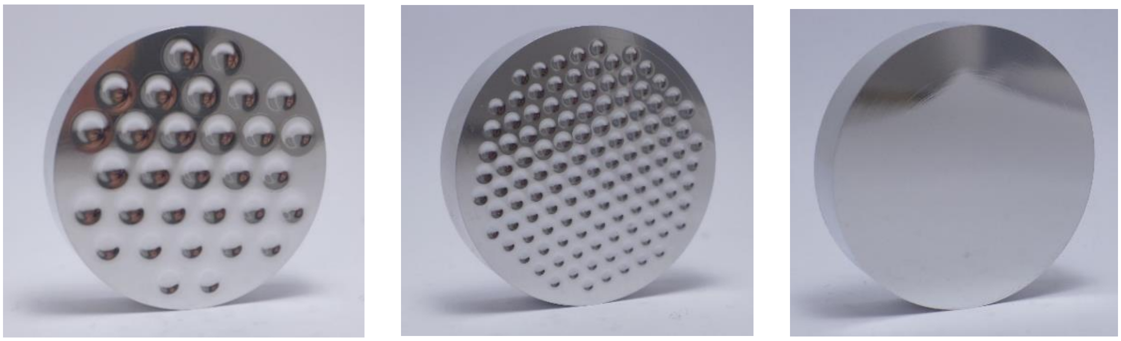


Figure 17: Left: Coarse structure – Middle: Small structure - Right: Sample with a flat surface.

Calibration of reference samples for the project

PTB calibrated the reference samples supplied to the partners of the project who performed emissivity measurements. The calibrations were done using the EMAF setup from PTB [6]. The results of total near-normal and total hemispherical emissivities are given in Table 5. Spectral directional emissivity was measured in the spectral range from 5 µm to 20 µm.

Angle	Nextel ε (79.7 °C)	$u(\varepsilon)$	Laser-structured gold plated ε (80.4 °C)	$u(\varepsilon)$	Gold Mirror Y_2O_3 overcoat ε (80.6 °C)	$u(\varepsilon)$	Infragold ε (80.6 °C)	$u(\varepsilon)$
10°	0.974	0.006	0.222	0.025	0.008	0.010	0.032	0.011
20°	0.973	0.006	0.219	0.025	0.009	0.010	0.032	0.011
30°	0.973	0.006	0.217	0.025	0.008	0.010	0.034	0.011
40°	0.972	0.006	0.214	0.025	0.009	0.010	0.034	0.011
50°	0.968	0.006	0.213	0.025	0.014	0.010	0.035	0.011
60°	0.961	0.005	0.212	0.025	0.018	0.010	0.037	0.011
70°	0.939	0.006	0.207	0.025	0.026	0.010	0.041	0.011
ε_{hem}	**0.944**	**0.005**	**0.211**	**0.025**	**0.017**	**0.010**	**0.035**	**0.011**

Table 5: Total directional and total hemispherical emissivities of Nextel 811-21, Laser-structured and gold plated, Gold Mirror Y2O3 overcoat and Infragold with standard uncertainties (k=1)

The results of total near-normal and total hemispherical emissivities for the solid samples with specific surfaces produced by Fraunhofer IPK are given in Table 6.

Angle	Sample 1 large structure ε (80 °C)	$u(\varepsilon)$	Sample 2 small structure ε (80 °C)	$u(\varepsilon)$	Sample 3 smooth surface ε (80 °C)	$u(\varepsilon)$
10°	0.045	0.009	0.041	0.010	0.039	0.010
20°	0.042	0.009	0.041	0.010	0.041	0.010
30°	0.042	0.009	0.041	0.010	0.041	0.010
40°	0.043	0.009	0.043	0.010	0.043	0.010
50°	0.048	0.009	0.050	0.010	0.051	0.010
60°	0.053	0.009	0.057	0.010	0.054	0.010
70°	0.064	0.009	0.067	0.010	0.062	0.010
ε_{hem}	**0.051**	**0.009**	**0.053**	**0.010**	**0.049**	**0.009**

Table 6: Total directional and total hemispherical emissivities of "Coarse structure sample", "Small structure sample" and "Flat sample" produced by Fraunhofer IPK with standard uncertainties (k=1)

Conclusions

PTB and LNE produced reference samples using reliable technical solutions already known for production of low emissivity samples and of high emissivity samples. For specular low emissivity samples a protected gold coating applied on polished aluminium was used. For high angular diffusing low emissivity samples, the Infragold® coating (from Labsphere company) was used. For the high emissivity samples, the Nextel velvet 811-21 black paint was used.

The attempt of PTB to produce reference samples with "medium" emissivity was not successful for the needs of the project with low uncertainty on the emissivity required. PTB produced by laser engravement a geometrically structured surface on aluminium substrate and the structured surface was gold plated. This gives a medium total emissivity around 0.2. The problem observed is the "poor" reproducibility of local emissivity or from sample to sample (spreading over more than 0.05). However, this work showed that structuring a metal surface and coating it with a low emissivity material is a way to produce surfaces with "medium" emissivities.

Aalto, LNE, PTB and Fraunhofer IPK collaborated for the definition, production and calibration of solid samples with specific structured surfaces giving angular distribution of reflected radiation similar to that of selected reflective foils. LNE measured, using a goniometer, the angular distribution of reflected radiation for several smooth foils and for a foil with a very structured surface (a "mesh reinforced foil"). Aalto was able to find by optical modelling surface structures giving angular diffusions similar to the ones obtained experimentally. The structure corresponding to the one of the "mesh reinforced foil" was the only one that can be produced by a mechanical process (machining or additive manufacturing). The structure corresponding to smooth foils is at the scale of roughness and is not easily producible. Aalto proposed surface structures with very shallow cavities arranged in a hexagonal configuration. Fraunhofer IPK produced using precision milling two samples with the defined structures and a flat surface sample. PTB measured the total directional and total hemispherical emissivities of the three samples. The achieved results showed that the measured angular distributions of the directional spectral emissivities is similar to that of a highly reflective insulation foil but also not very different to a plane sample. This leads to the conclusion that the developed and produced reference samples can be successfully used as calibration standards for emissivity measurements. This work showed that precision milling associated with a light polishing process is a way to produce solid samples with low emissivities and with surfaces having morphologies similar to morphologies of reflective foils with quite large surface structures.

Precision milling is also promising for producing solid surfaces with "medium" mean emissivities by machining tailored "cavities". The availability of solid samples with tailored medium emissivities would be useful for comparing emissivity measurement techniques and for producing calibrated standards for users of reflectometers not allowing easy correction of the "sample/standard substitution error".

The morphologies of surfaces of reflective foils used as external surface of building insulation products are innumerable. Nevertheless, even if the number of surfaces produced in EMIRIM project is limited, the project has demonstrated that the production of solid metal samples with tailored surfaces, obtained using precision machining, is a way to produce samples with low emissivities and with tailored angular diffusion of reflected radiation. The samples produced can be calibrated in total near-normal emissivity and in total hemispherical emissivity with an uncertainty of 0.02 using primary reference emissivity measurement techniques.

The objective of building new competencies in NMIs/Dis in order to produce appropriate calibrated reference samples for characterising end-user instruments and for ensuring traceability of measurements is met. Partners have demonstrated that metal mirrors, very diffusing low emissivity standards and high emissivity paints can be calibrated in total hemispherical emissivity with an uncertainty of 0.02 and are suitable for calibrating end-users measurement setups and for checking the sensitivity of the measurement techniques to the angular diffusion of the reflected radiation. The EMIRIM project has also demonstrated that it is possible to produce by precision machining and polishing low emissivity surfaces with specific angular diffusions.

Objective 4: Definition of calibration and measurement procedures enabling the end-users to perform emissivity measurements on reflective foils with an uncertainty below 0.03 for emissivities below 0.1

INGLAS TIR100-2 emissometers

The comparisons results (Figure 1, Figure 2) showed that TIR100-2 emissometers are not very sensitive to the specularity (angular dispersion of reflected radiation) of the sample. The total near-normal emissivity measured on Infragold by TIR100-2 instruments calibrated with gold mirrors remains within ± 0.015 from the reference value from PTB. In normal use TIR100-2 is calibrated using a mirror (low emissivity) and a high emissivity surface. The EMIRIM project confirmed that a specular standard is appropriate for calibration, it must be calibrated in total near-normal emissivity with traceability to SI.

The main recommendations for measuring total near-normal emissivity of reflective foils with a TIR100-2 emissometer are:

- maintain the samples and the calibrated standards at room temperature,
- use a sample holder with a high emissivity or a sample holder ensuring a good thermal contact between the sample and the holder,
- recalibrate the emissometer each 10 minutes to compensate for thermal drift,
- place the sample in contact with the spacers defining the plane of measurement,
- a sample should not be maintained in place more than a few seconds to avoid the perturbation of the hemisphere’s temperature and sample heating,
- for reflective foils a measurement should be performed in under 3 seconds to avoid sample heating,
- after a measurement or calibration, a pause of approximately 30 seconds should occur before placing a new sample in front ofthe hemisphere to avoid temperature perturbation of the hemisphere.

Calibration and Measurement procedure:

- After temperature stabilisation, calibrate the emissometer following the manufacturers manual.
- After switching on and temperature stabilisation, the device is in the "calibration configuration". Check that the emissivity values set in the device for the standards to be used are correct.The calibration configuration is accessible by pressing the "Calibr" touch button.
- Once in calibration configuration, place the high emissivity standard in the sample position and press "Calibrate High".
- After a beep, remove the standard and place the low emissivity standard in the sample position and press "Calibrate Low".
- After a beep remove the low emissivity standard and store the standards near the device but do not expose to the radiation of the hemisphere.
- Fix the foil sample onto the sample holder and place the sample in the sample position.
- 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 (temperature of the hemisphere) 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.

Integrating spheres

The quality of emissivity measurements depends on the optical configuration when using the integrating sphere. A typical optical configuration is shown in Figure 18.

Control of the optical configuration

A definition of a correct optical configuration is:

- 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 to 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 in good 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.

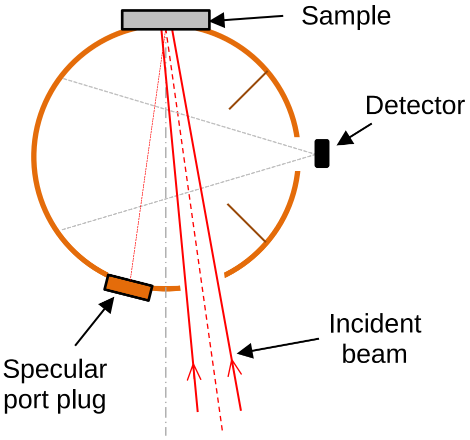


Figure 18: Typical configuration of an infrared integrating sphere

The main poor optical configurations found are shown in Figure 19. 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. When a specular sample is at the sample port, it is possible that the beam reflected specularly by the sample irradiates, after 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 walls 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.

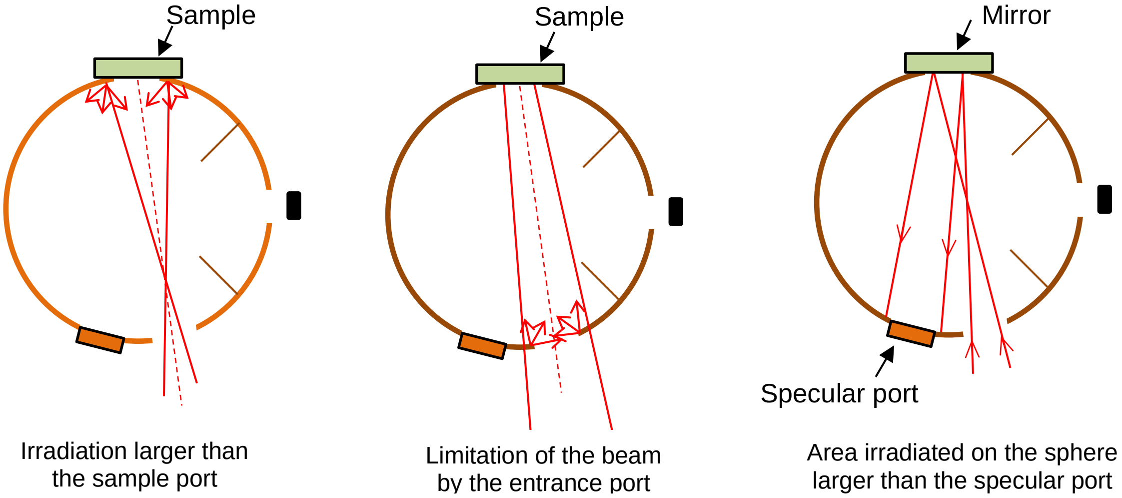


Figure 19: Bad optical configurations mostly found

Before using an integrating sphere for measurements, the optical configuration must be checked. If the setup allows the configuration of the spectrometer "in visible light" or the use of a visible light source, the optical control can be done visually. The points to be checked are the sizes of the incident beam at the level of the entrance port and at the level of the sample port. If working with visible light is not possible, the measurement of the signal without sample (or with a blackbody at the sample port) is a way to detect an incident beam larger than the sample port or larger than the entrance port. A way to detect if the area irradiated on the sphere is larger than the specular port when a mirror is placed at the sample port is to measure the reflectance of a mirror in "specular excluded mode" (without the specular port plug). If the optical configuration is correct, an almost zero signal must be obtained in "specular excluded mode".

A way to check that the integrating sphere is not too sensitive to the angular dispersion of the reflected radiation is to have two low emissivity calibrated standard, one very specular (a mirror) and one very diffusing (Infragold® like coating) and to compare the measurement result for the diffusing standard when the integrating sphere is calibrated with the specular standard to the calibrated emissivity value of the diffusing standard.

Measurement of signals

The measurements of signals must be performed once the response of the detector is steady. The spectrometer, the radiation source, the detector and the related electronic systems must be steady in temperature. If applied, the purging should have started enough time before the calibration.

In the recommended procedure, six signals are measured:

$S_{ref\ stand}$: signal measured on the calibrated reference standard (Ref. standard config.),

$S_{sphere\ ref\ stand}$: signal measured on the sphere wall with calibrated reference standard at the sample port (Sphere config. with ref standard),

S_{sample} : signal measured on the sample in test (Sample config.),

$S_{sphere\ sample}$: signal measured on the sphere wall with the sample in test at the sample port (Sphere config. with sample),

S_{zero} : signal measured whithout sample or with a blackbody at the sample port (Zero config).

$S_{sphere\ zero}$: signal measured without sample or with a blackbody at the sample port (Sphere config. no sample).

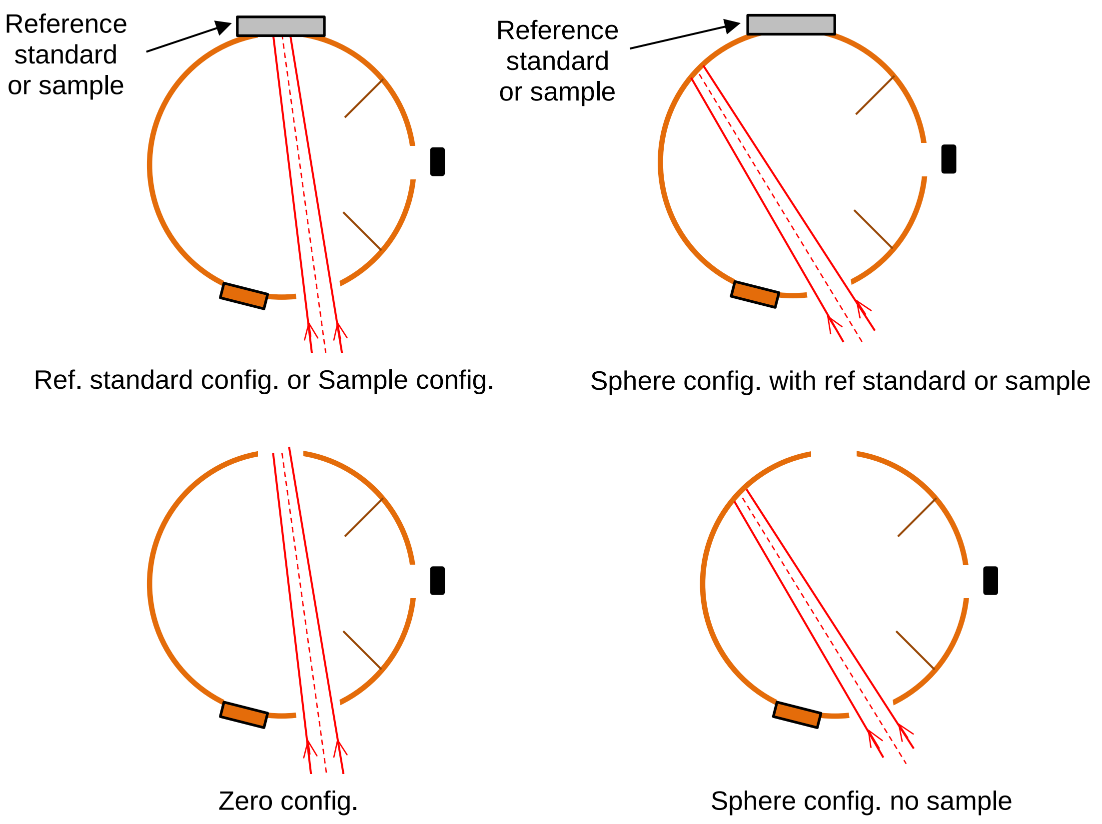


Figure 20: Configurations for measurement of signals

When the measurements on the foils are completed, a check of the calibration is done by a measurement on the reference standard as well as a measurement of the "zero signal". If the measurements of the foils take a long time, it is recommended to check the calibration periodically (each hour at least).

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

From the six signals measured, the spectral near normal emissivity is calculated using relation 1.

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

where $\rho_{ref\ \lambda}$ is the spectral near-normal hemispherical reflectance of the low emissivity calibrated reference sample.
The application of relation 1 requires that the sample is opaque.
If the zero signals are not measured for each measurement campaign, because the optical configuration is reliable to ensure a non-significant zero signal, then the ratio $\frac{S_{zero}}{S_{sphere\ zero}}$ is set at 0 in relation 1.

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. The quantification of the total hemispherical emissivity from the measured parameters is done through an intermediate step, the calculation of total near-normal 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). Therefore, the spectral range 5 µm to 50 µm is recommended by standard EN12898 for calculation of total emissivities.
The relation implicitly recommended by standard EN 12898 for calculation of total near-normal emissivity is

$$\varepsilon_{tot\ NN} = \frac{\int_{5\mu m}^{50\mu m} \varepsilon_{\lambda\, nn\, meas} \cdot l^{\circ}(T_{samp}, \lambda) \, .d\lambda}{\int_{5\mu m}^{50\mu m} l^{\circ}(T_{samp}, \lambda) \, .d\lambda} \qquad (2)$$

where ε_{λ} is the measured near-normal spectral emissivity of the sample, T_{samp} is the temperature of the material in use (usually the room temperature), λ is the wavelength and $l^{\circ}(T_{samp}, \lambda)$ is 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 emissivities measured at specific wavelengths, the temperature for calculation is 283 K). EN 12898:2019 specifies 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

$$\varepsilon_{tot\ NN} = \frac{\int_{5\mu m}^{\lambda max} \varepsilon_{\lambda\, nn\, meas} \, .l^{\circ}(T_{samp}, \lambda) \, .d\lambda}{\int_{5\mu m}^{50\mu m} l^{\circ}(T_{samp}, \lambda) \, .d\lambda} + \frac{\int_{\lambda max}^{50\mu m} \varepsilon_{\lambda} \, .l^{\circ}(T_{samp}, \lambda) \, .d\lambda}{\int_{5\mu m}^{50\mu m} l^{\circ}(T_{samp}, \lambda) \, .d\lambda} \qquad (3)$$

where λ_{max} is 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 that is to extrapolate the spectral emissivity from the spectral emissivity curve obtained experimentally. If the spectral curve in the spectral range of measurement is quite flat for longer wavelengths, then the spectral emissivity can be assumed to be constant from λ_{max} up to 50 µm. This assumption is quite realistic for low emissivity foils using metal foils or metal coatings. The relation becomes

$$\varepsilon_{tot\ NN} = \frac{\int_{5\mu m}^{\lambda max} \varepsilon_{\lambda\, nn\, meas} \, .l^{\circ}(T_{samp}, \lambda) \, .d\lambda}{\int_{5\mu m}^{50\mu m} l^{\circ}(T_{samp}, \lambda) \, .d\lambda} + \frac{\int_{\lambda max}^{50\mu m} \varepsilon_{\lambda max - 50\mu m} \, .l^{\circ}(T_{samp}, \lambda) \, .d\lambda}{\int_{5\mu m}^{50\mu m} l^{\circ}(T_{samp}, \lambda) \, .d\lambda} \qquad (4)$$

where $\varepsilon_{\lambda_{max}-50\mu m}$ is 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 by the operator in order to filter the noises on results at long wavelengths. Other models can be used for extrapolating spectral emissivity at long wavelengths. The selection of the model is the responsibility of the test manager and the related uncertainty must be included in the uncertainty budget.

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 localised (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 in then 180.

Extrapolation of total hemispherical emissivity from the total near-normal emissivity

The emissivity required 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".

$$\varepsilon_{tot\ hem} = 1.1887\,\varepsilon_{tot\ NN} - 0.4967\,\varepsilon_{tot\ NN}^{2} + 0.2452\,\varepsilon_{tot\ NN}^{3} \qquad (5)$$

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

Relation 6 was established for coated glass. However, the optic theory gives quite similar ratios between the hemispherical emissivity and the normal emissivity for smooth low emissivity surfaces.

The results from PTB, given in Table 3, showed that, for the polymer reflective foils tested, the total hemispherical emissivities calculated by PTB from the total directional emissivities measured are higher than the total hemispherical emissivities given by relation 5 applied using the total near-normal emissivity value measured by PTB at 20°. For two smooth foils tested the magnitude of the difference is about 0.025. It must be considered that these results were obtained on very few types of polymer low emissivity foils and that uncertainties on total directional emissivity results from PTB are quite large. However, as a precaution and at this stage of knowledge, the conclusion of the partners in EMIRIM project is that relation 5 can give under-evaluated values of the total hemispherical emissivity of polymer reflective foils with an under-evaluation of about 0.025 for very low emissivity polymer foils.

Nevertheless, not applying relation 5 would give emissivity values even more under-evaluated. So the recommendation of the partners in the EMIRIM project is to apply relation 5 to calculate the total hemispherical emissivity from the total near-normal emissivity measured with TIR100-2 emissometers or integrating spheres and to consider an expanded uncertainty about 0.025 (k=2) for the extrapolation process. This recommendation should be applied to any polymer low emissivity foil without more detailed knowledge for the different types of reflective foils.
The combination of the uncertainty related to the extrapolation and of the uncertainty on measurement of the total near-normal emissivity with TIR100-2 emissometers or integrating spheres (0.25) gives a combined expanded uncertainty of 0.36 on the total hemispherical emissivity value.

Conclusions

Calibration and measurement procedures were established for integrating spheres and TIR100-2 emissometers. These procedures allow the optimum use of both measurement techniques for testing low emissivity foils. The EMIRIM project showed that integrating spheres can be sensitive to the optical configuration of the incident beam and that significant errors can occur when testing reflective foils with a poor optical configuration.

Furthermore, the two techniques measure the total near-normal emissivity and not the total hemispherical emissivity which is the parameter required for reflective foils. The total hemispherical emissivity can be calculated from the total near-normal emissivity measurement result but, at the knowledge stage at the end of the EMIRIM project, an expanded uncertainty about 0.025 (k=2) must be considered for the calculation process. This leads to a combined global uncertainty of at least 0.036 on the total hemispherical emissivity result for a reflective foil.

The objective is only partially met as the final uncertainty on total hemispherical emissivity results is higher than 0.03. A significant part of the uncertainty is related to the model for calculating the total hemispherical emissivity.

The establishment of more appropriate model(s) would require substantial work for analysing accurately the angular variations of the total directional emissivity of the different types of reflective foils. This work was beyond the resources available in this project.

Objective 5: Revision of standard EN 16012

Partners involved in EMIRIM project have analysed in detail standard EN 16012 and proposed modifications considering the results and knowledge gained in the project. The main modifications proposed by partners in EMIRIM project and remarks for improvement of EN16012 are given hereafter.

Proposed modifications in section 5.9.2 of EN16012

Initial text:

5.9.2.1 Procedure

The emissivity shall be measured using the apparatus defined in Annex D (or other equipment giving at least the same level of accuracy and validated against the total hemispherical integrative sphere method, which is the fundamental physical reference procedure).

Text proposed:

5.9.2.1 Procedure

The emissivity shall be measured using the apparatus defined in Annex D (or other equipment giving at least the same level of accuracy). Infrared integrating spheres associated to Fourier transform spectrometers can be used for measurements of total near-normal emissivity with uncertainty level similar to the one of apparatus described in Annex D. However, some integrating spheres doesn't have the most appropriate optical configuration for tests on low emissivity foils and must be used very carefully after a characterisation to quantify the uncertainty when applied to test reflective foils.

Justification of the modification:

Most of instruments with integrating spheres cannot be considered as reference instruments. They must be calibrated for each campaign of measurement by using a high reflective calibrated standard. Furthermore, the EMIRIM project has demonstrated that some integrating spheres can be sensitive to the specularity of the sample which is a problem for reflective foils. With integrating spheres, the measurements spots are usually quite small which is not favourable for reflective foils with a pattern.

Proposed modifications in section D4

Initial text:

D.4 Calculation of the emissivity

The emissivity is determined from comparing the measured result for the specimen with the two calibration standards. With the sensor signals (U, UH and UL) and the known emissivity of calibration standards (εL and εH), the emissivity, ε, of the specimen shall be calculated by:

$\epsilon = \epsilon H - (\epsilon H - \epsilon L) \times (UH - U) / (UH - UL)$ *(D.1)*

NOTE The measurement range of the apparatus is limited to values between those of the two calibration standards used, hence within the emissivity range of 0.02–0.94. However, there are practical limits to the measurement of very low values of emissivity, irrespective of the method used. Errors increase significantly below emissivity 0.05.

Text proposed:

D.4 Calculation of the emissivity

The total near-normal emissivity is determined from comparing the measured result for the specimen with the two calibration standards calibrated in total near-normal emissivity. *With the sensor signals (U, UH and UL) and the known emissivity of calibration standards (εL and εH), the emissivity, ε, of the specimen shall be calculated by:*

$\epsilon = \epsilon H - (\epsilon H - \epsilon L) \times (UH - U) / (UH - UL)$ *(D.1)*

NOTE The measurement range of the apparatus is limited to values between those of the two calibration standards used, hence within the emissivity range of 0.01-0.98. *However, there are practical limits to the measurement of very low values of emissivity* due to uncertainties of measurements *irrespective of the method used*.

Important remarks:

The relation above gives only the total near-normal emissivity. With integrating spheres, it is also the near-normal emissivity which is measured.

Recommendations should be integrated in the standard for extrapolating the total hemispherical emissivity from the result of total near-normal emissivity. It would be preferable that all users measuring effectively the total near-normal emissivity use the same extrapolation relation.

The emissivities required for calculation of the thermal resistance of an air space are the total hemispherical emissivities of the surfaces limiting the air space. This is explicitly mentioned in Standard ISO 6946:2017(E), Annex D: "Thermal resistance of airspaces", Section D.2: "Unventilated airspaces with length and width both more than 10 times thickness". Relation D.4 for calculation of E (intersurface emittance) uses ε1, ε2 defines as "the hemispherical emissivities of the surfaces bounding the airspace".

Standard EN 12898 - March 2019: " Glass in building - Determination of the emissivity" gives, in Section 6.2 "Corrected emissivity", a relation for extrapolation of hemispherical emissivity from near-normal emissivity.

$\varepsilon = 1.1887\ \varepsilon_n - 0.4967\ (\varepsilon_n)^2 + 0.2452\ (\varepsilon_n)^3$ (5)

where ε_n is the near-normal emissivity.

This relation 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.

In the EMIRIM project, total hemispherical emissivity measurements were performed on some low emissivity foils by PTB and LNE, the results from both laboratories were in good agreement. PTB performed also near-normal emissivity measurements on the same foils. The ratios between the hemispherical emissivities and the normal emissivities were greater than the ratios given by the relation above.

At this stage of knowledge (from results in the EMIRIM project), an uncertainty of about 0.025 should be considered specifically for the process of extrapolation for low emissivity foils.

Furthermore, the results from EMIRIM for the reflective foils showed a systematic under-evaluation of the total hemispherical emissivity when the model from EN 12898 is used for calculating total hemispherical emissivity.

This uncertainty of extrapolation as well as the uncertainty on the measurement of the total near emissivity must be integrated in the evaluation of the global emissivity on the total hemispherical result (see remark for Section D7 below).

This conclusion is based on a limited number of emissivity results with quite large uncertainties.

Proposed modifications in section D7

Initial text:

D.7 Expression of results

The emissivity of the specimen shall be expressed to 2 decimal places. All single measurements resulting in an emissivity < *0,02* or > *0,94 (measurement range of the apparatus) should be set to 0,02 or 0,94 respectively. The emissivity mean value, all the single values per specimen and the standard deviation of the results from the tested product shall be included on the test report. The emissivity mean-value shall be rounded to two decimal places.*

The mean value (one test result) from any one sample shall be derived from a minimum of 3 specimens taken from the sample with five measurements being taken on each specimen. 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) calculated using the 90/90 fractile rules from EN ISO 10456. The manufacturer may use a higher number of test results (samples) in the calculation. A mean value below 0,05 is declared as 0,05."

Text proposed:

D.7 Expression of results

The emissivity of the specimen shall be expressed to 2 decimal places. All single measurements resulting in an emissivity < 0,02 or > 0,98 *(measurement range of the apparatus) should be set to 0,02 or* 0,98 *respectively. The emissivity mean value, all the single values per specimen and the standard deviation of the results from the tested product shall be included on the test report. The emissivity mean-value shall be rounded to two decimal places.*

The mean value (one test result) from any one sample shall be derived from a minimum of 3 specimens taken from the sample with five measurements being taken on each specimen. 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) calculated using the 90/90 fractile rules from EN ISO 10456. The manufacturer may use a higher number of *test results (samples) in the calculation. A mean value below 0,05 is declared as 0,05."*

Important remark:

Members of EMIRIM project don't propose the modification of the limit value at 0.05 for the lowest declarable emissivity.

Indeed, the combination of the uncertainty of measurement of total near-normal emissivity with the uncertainty of extrapolation of total hemispherical emissivity (when using model from EN 12898) gives an uncertainty around 0.04 on the final result of total hemispherical emissivity when the total hemispherical emissivity is determined by using TIR100-2 emissometers or integrating spheres.

Conclusions

The proposals of modifications and remarks for improvement of EN16012 were submitted to the convenor of CEN WG12 in November 2020.

The main recommendation is that the calculation of total hemispherical emissivity from the measured total near-normal emissivity should be addressed in the standard.

2 Impact

Initial results from the project were presented at the 20th Symposium on Thermophysical Properties (United States, June 2018) via an oral presentation and intermediate results at the Tempmeko & TempBeijing 2019 Symposium (China, June 2019) as an oral presentation. At the 19th International Congress of Metrology (France, 2019) the project gave an oral presentation as well a poster presentation to promote project outcomes.

Two published open access papers present the results of a detailed characterisation of the TIR100-2 emissometer performed by PTB and the collaborative work performed to define the shape of the surface for production of solid samples with tailored angular diffusions of reflected radiation. Another paper on the comprehensive characterisation of the TIR100-2 emissometer giving a detailed uncertainty budget for emissivity measurements on reflective foils has been submitted for publication.

A project workshop was held in November 2018 to deliver training on '*Improvement of Emissivity Measurements on Reflective Insulation Materials'* to end-users from the thermal insulation industry, researchers and members of certification bodies as well as consortium members. The general content of the project and intermediate results on characterisations of end-user measurement techniques were presented and discussed. A second workshop in November 2020 for end-users from the thermal insulation industry, scientists and researchers presented project results, uncertainty budgets and recommended procedures for calibration and use of end-user emissivity measurement techniques. The proposals for improvement of standard EN 16012 were also presented in detail and discussed as well as the procedure for calculation of the total hemispherical emissivity from the measured total near-normal emissivity.

Impact on industrial and other user communities

The producers or distributors of thermal insulation materials for building wanting the "low emissivity effect" of their products to be considered by designers must declare a total hemispherical emissivity value to comply with EU Directive 2010/31/EU. Therefore, the project results benefit the producers of low emissivity insulation products, producers of flexible sheets for waterproofing buildings and organisations testing and certifying these products. End-user good-practice guides developed by the project are available from the project website.

The improvement of uncertainties for the measurement of low values of total hemispherical emissivity will facilitate the development of new high-performance thermal insulation systems like multi-layer-insulations (MLI) based on a stack of low emitting screens. These have various industrial and research applications including those where space is limited and weight penalties high such as satellites, space vehicles, aircrafts or applications where stacks of low emitting screens under vacuum can be used such as cryogenics and storage of liquefied gases. The improved knowledge of the performance and limitations of measurement techniques used industrially and the availability of appropriate calibrated samples will help end-users in the automotive, aeronautical and the space industries where reflective foil insulation is critical for the protection of people and infrastructures from intense thermal radiation. In these industries reliable emissivity measurements are often performed using commercially available instruments, and measurements with validated uncertainties will lead to improve demonstrations of products matching specifications. Therefore, many industrial sectors will benefit from the progress made in low emissivity measurements on reflective building insulation materials.

Impact on the metrology and scientific communities

In addition to presentations at scientific conferences and publications in scientific journals, this project and particularly the improvements developed at the reference level in National Metrology Institutes were presented to the Annual conference of the AK Thermophysik – GEFTA (Germany - 2018), EURAMET TC for Thermometry - Thermophysical Quantities of Materials WG and BIPM - CCT Task Group for Thermophysical Quantities (CCT-TG-ThQ).

This project has enabled the extension and improvement of three NMI measurement service capabilities for the calibration of emissivity standards with low uncertainties, that will facilitate the reliable validation and use of commercial emissivity measurement instruments in research and industry. The improved capabilities of PTB and DTU to measure, with low uncertainties spectral or total directional emissivities, will allow calibrations and measurements of emissivity at variable angles. The capabilities of most research laboratories or end-users are limited to measurement of spectral or total directional emissivity for a single direction close to normal. Therefore, PTB and DTU will be able to provide end-users with precise results at variable angles for applications in scientific research where angular emissivity values are required for precise heat balance calculations or applications of pyrometry or thermography for non-contact temperature measurements at non-normal directions. The calorimetric technique improved at LNE is rare even among research laboratories worldwide. It has the capability to directly measure the total hemispherical emissivity (all wavelengths, all directions) with a low uncertainty particularly for low emissivities. The improved calorimetric technique will be particularly useful for testing non-specular surfaces with very low total hemispherical emissivities. Potential applications include satellite measurement instruments that must be protected from parasitic thermal radiation.

Impact on relevant standards

The main objective of the project was to support CEN/TC 89/WG 12 to improve EN 16012 for the measurement of total hemispherical emissivity of low emissivity foils. Remarks and improved versions of the technical sections of the standard related to emissivity measurements were written and sent to convenor of CEN/TC 89/WG12 for potential incorporation into a revision of EN 16012 which will form a new EN standard for insulation products. The project addressed a lack of precision in the definition of the emissivity measured with the measurement techniques recommended in the standard which created the risk of manufacturers declaring undervalued emissivities for low emissivity insultation foils. It also addressed the absence that the total hemispherical emissivity is the parameter to be declared in specification for a thermal insulation product. The integrating sphere is mentioned in EN 16012 as a reference measurement technique, but its use with poor optical configurations may produce errors especially for reflective foils. This may inadvertently lead end-users to select a measurement technique that is quite difficult to use to obtain reliable results. Therefore, a recommendation for explaining the technical risks related to the integrating sphere technique was also provided.

In summary, the knowledge transferred from EMIRIM project to CEN/TC 89/WG12 should facilitate greater accuracy in emissivity measurement made by users applying standard EN 16012 and a reduction in the risk of measurement errors.

Good-practice guides for calibration and for emissivity measurement with integrating spheres and TIR100-2 emissometers were communicated to the convenor of CEN/TC 89/WG 12 and were presented and discussed during a workshop held in November 2020 involving end-users from the thermal insulation industry and members of the standards working group. These good-practice guides are available from the project website.

Longer-term economic, social and environmental impacts

The improvement of emissivity measurements on insulation products will help to increase the efficiency of thermal insulation systems for buildings, industries and home appliances. More reliable emissivity data will allow accurate calculations of heat transfer coefficients. In direct consequence, the results of the project will help in the reduction of energy consumption. The project will help to enhance consumer protection through the availability of insulation products with more accurate and validated declared performance values. More reliable and traceable emissivity measurements using commercially available instruments will help reduce the cost of product development and increase their performance. Reflective low emissivity surfaces are also used to protect people or structures from intense radiation sources such as a very hot surfaces; the reflective surface is then used in association with an insulation material or alone as a radiation screen. Typical applications are in the steel industry, glassmaking or firefighter protection. Radiation screens are also used widely to protect structures from thermal radiation emitted by hot elements. For example, from hot exhaust parts in vehicles, or the protection of critical elements or cables from fire. A particular area where a low emissivity is required is solar thermal collection, the lower the emissivity the higher the efficiency due to the reduction of thermal losses. For high tech solar absorbers low uncertainty on total hemispherical emissivity (around 0.02) is required for validating absorbers over long periods of use (ageing effect on emissivity) and for making investment decisions.

The improvements made and knowledge gained in this project for performing emissivity measurements will have positive applications in various areas particularly those related to energy saving and renewable solar powered energy production.

3 List of publications

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This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

4 Contact details

The project website is: http://projects.lne.eu/jrp-emirim/

The contact person is:

- Jacques Hameury, LNE, Jacques.hameury@lne.fr

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