16NRM06 EMIRIM

Improvement of emissivity measurements on reflective insulation materials

Good-practice guide :

Calibration of integrating spheres setups and total hemispherical near-normal reflectometers used for measurement of total hemispherical emissivity of low emissivity foils.

Deliverable D5A

Main Author: Jacques HAMEURY

Co-authors: Jochen Manara, Maria Carla Arduini

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1. INTRODUCTION

In EU, the instruments mostly used for measurement of total hemispherical emissivity of low emissivity foils found at the surface of certain thermal insulation products are the emissometer TIR100-2 produced by INGLAS GmbH & Co.KG and infrared integrating spheres often used in association with a Fourier transform infrared spectrometer (FTIR).

The two techniques must be calibrated before measurements and along the phase of measurement to ensure the stability and trueness of response for all measurements.

Both types of instruments are reflectometers. They can be sensitive to the specularity of the sample and to the local angular distribution of the radiation reflected on the measurement spot.

This good practice guide gives the main recommendations for calibrating the TIR100-2 emissometers and the infrared integrating spheres with the objective to perform total near-normal emissvity measurements on reflective foils with the lowest possible uncertainties.

2. CALIBRATED STANDARDS REQUIRED FOR CALIBRATION

2.1. CALIBRATED STANDARDS FOR TIR100-2 EMISSOMETERS

For calibrations of TIR100-2 instruments, two types of standards are required, one with a very high emissivity and one with a very low emissivity. For measurements on low emissivity foils, the quality of the low emissivity standard and the uncertainty on its emissivity are most important. The standards should have an area large enough to cover the opening of the heating hemisphere (a diameter of 100 mm is appropriate).

Usually TIR100-2 instruments are calibrated with a low emissivity metal mirror surface and a black surface. A requirement for the two standards is small variations of spectral emissivities for wavelengths from 3.5 µm up to 35 µm.

The low emissivity standard can be a polished bare metal surface or a coating used for producing infrared mirrors applied onto a substrate. The reflective surface can be protected by a thin overcoating.

The high emissivity standard can be produced by coating a flat surface with a high emissivity paint. The paint NEXTEL 811-21 has been used for a long time for productions of surfaces with high emissivities and for comparing performances of high accuracy measurement techniques. One particular quality of NEXTEL 811-21 paint for the application is a very high near-normal spectral emissivity over the spectral range from 3.5 µm to 35 µm [1]. The thickness of the high emissivity paint should be limited in order to avoid excessive heating of the surface when calibrating TIR100-2 emissometers.

During the measurements for calibration, the surface of the standard is submitted to the hot air surrounding the hot hemisphere of the device and to the radiation emitted by the hemisphere. It is therefore recommended that the two standards have a high thermal capacity to reduce the heating when exposed to hot ait and to radiation. A thick plate of a metal with a high thermal conductivity is a way to get a high thermal capacity.

2.2. CALIBRATED STANDARDS FOR INTEGRATING SPHERES

It is recommended to have two calibrated standards for calibration of integrating spheres and for checking the sensitivity of the integrating sphere to the specularity of samples. One standard should have a very specular reflection (mirror) and the other standards should have a low emissivity very diffusing surface (like the Infragold® coating produced by Labsphere company USA).

If the measurement procedure with the integrating sphere does not allow the correction of the "standard/sample substitution error" it is recommended to have also a standard with a medium emissivity for improving the linearity of response of the integrating sphere.

2.3. SPECIFIC CONFIGURATION OF STANDARDS FOR CALIBRATION WITH A PRIMARY SETUP

If the standards must be calibrated with a primary technique, there are probably special specifications for geometry and thermal conductivity of the standard to fit the calibration setup. Specific housings for temperature sensors can be required also. The production of the body of the standard with appropriate geometrical specifications must be done before finishing the low or high emissivity surfaces.

2.4. HANDLING AND STORAGE OF STANDARDS

Pollution, mechanical deteriorations, oxidation of the surface can change significantly the emissivity of a standard particularly for low emissivity surfaces. The protection of the surfaces and appropriate procedures must be established for handling and storing the calibrated standards.

To always keep the availability of well-calibrated standards, it is recommended that the user has at least two sets of standard(s), a "primary calibrated standard(s) set" and a "working standard(s) set" for usual measurements. The "primary calibrated standard(s) set" is the one used for calibrating periodically the "working standard(s) set" and it is store in good conditions and used only for periodical calibrations of the "working standard(s) set" or for high precision measurements. The "primary calibrated standard(s) set" is the one calibrated to ensure traceability of the measurements perform by the user to the International System of Units (SI).

If specific mechanical supports facilitate the use and manipulations of the standards, it is recommended to build or acquire such supports.

3. CALIBRATION OF STANDARDS

For measurements of total near-normal emissivity of reflective foils with an uncertainty of 0.03 (k=2), the uncertainty on the total near-normal emissivity of the low emissivity standard should not be more than 0.02 (k=2).

The calibration of standards should be traceable to the international system of units (SI).

4. CALIBRATION OF TIR100-2 EMISSOMETERS

4.1. CALIBRATION OF TIR100-2 EMISSOMETERS FOR MEASUREMENT OF EMISSIVITY OF REFLECTIVE FOILS

The standards to be used are a specular (mirror like) low emissivity standard and a high emissivity standard.

Before calibration, the device must have been turn on for at least two hours to get temperature stabilization of the radiating hemisphere and of the internal elements. During the stabilization in temperature, the standard(s) used for calibration can be placed at a distance from the device to stabilize at the room temperature. The distance should not be

too short so that the temperatures of the standards are not influence by the temperature of the TIR100-2. The standards should neither be placed in view of the cavity side of the device. The heating, stabilization in temperature and calibration must be performed in calm air in a room at a steady temperature.
Once stabilized in temperature, the display shows the measured temperature of the hemisphere. The calibration should be done only when the displayed temperature is 100 ± 0.5 °C and when the temperature variates of less than 0.5 °C for 10 minutes.

For the calibration, it is recommended to follow the instructions from the manufacturer. The calibration starts with the high emissivity level.

When calibrating for each emissivity level, the emissivity value set in the device is displayed. This allows for controlling that the emissivity value set in the device is the correct one for the standard used. If the value is false, it must be changed in the configuration of the device (see manual).

For measurement with high accuracy, the TIR100-2 emissometer must be recalibrate each 10 minutes to correct for potential temperature drift.

4.2. MEASUREMENT OF THE SENSITIVITY OF THE INSTRUMENT TO THE ANGULAR DIFFUSION OF REFLECTION OF THE SAMPLE

It can be useful for users performing emissivity measurements on materials with various angular diffusions to have a quantitative evaluation of the sensitivity of the instrument to the angular diffusion of the surface tested.
A way to measured that sensitivity is to measure, using the TIR100-2 calibrated with a specular low emissivity standard, the total near-normal emissivity of a low emissivity very diffusing sample which total near-normal emissivity has been calibrated with a low uncertainty.

The difference between the measurement result from theTIR100-2 and the calibration value can be considered has an uncertainty related to the sensitivity of the TIR100-2 device to the angular diffusion of the surface tested. Measurements done in the EMIRIM project showed that, for Infragold®, the results of measurement from the TIR100-2 emissometer calibrated with a specular standard were higher than the calibration value with a difference below 0.015 in magnitude.

5. CALIBRATION OF INTEGRATING SPHERES

5.1. CALIBRATION OF INTEGRATING SPHERES FOR MEASUREMENTS OF EMISSIVITY OF REFLECTIVE FOILS

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

The calibration procedure depends on the optical configuration on the integrating sphere and can depend on the sensitivity of the integrating sphere response to the angular diffusion of the sample.

The techniques for controlling the optical configuration of an integrating sphere are presented in the good practice guide D5B "Procedures for measurement of total near emissivity of low emissivity foils enabling end-users to perform emissivity measurements on reflective foils with an uncertainty below 0.03 for emissivities below 0.1 by using integrating spheres setups or total hemispherical near-normal reflectometers".

For calibrating an integrating sphere and testing the sensitivity to angular diffusion of the sample it is recommended to have a calibrated specular standard (mirror) and a calibrated very diffusing standard (Infragold® type).

The calibrations presented in §5.1.1 and in §5.1.2 are sufficient if a procedure is applied to correct for the "*single-beam sample-absorption error*" [2,3].

5.1.1. Calibration of integrating spheres with a good optical configuration

The "good optical configuration" is illustrated in Figure 1.
It is defined by:

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

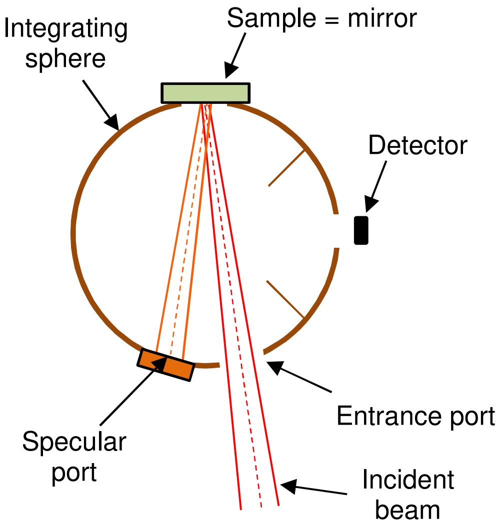

Figure 1: Good optical configuration for an infrared integrating sphere

For low emissivity reflective foils, it is preferable to use a specular calibrated standard for calibration.

It is highly recommended to test the sensitivity of the integrating sphere to the angular diffusion of reflection of the sample by comparison of the signal measured on a calibrated very reflective and very diffusing standard (Infragold® type) to the signal measured on the specular calibrated standard. If the ratios of the two signals are significantly different from the ratios of the near-normal spectral reflectances (known from primary calibrations) it means that the response of the integrating sphere depends on the angular diffusion of the sample.

If the sensitivity of the integrating sphere is not very dependent on the angular diffusion of the sample, the relative difference between the sensitivity obtained with a specular standard and the sensitivity obtained with a diffusing standard is then considered as a relative uncertainty on the measured reflectance.

If the sensitivity of the integrating sphere is dependent on the angular diffusion of the sample, and if the angular diffusion of the sample tested is not known it is recommended to use a mixed calibration using the specular standard and the very diffusing standard. Half the relative difference between the "specular" sensitivity and the "diffusing" sensitivity is then considered as a relative uncertainty on the measured reflectance due to the sensitivity to angular diffusion of the sample.

5.1.2. Calibration of integrating spheres with a poor optical configuration

If the optical defect of the optical configuration is "an area irradiated by the incident beam larger than the sample port" or "incident beam larger than the entrance port" (Figure 2) then a permanent radiation exists in the integrating sphere whatever the reflectance of the sample. This permanent radiation is detected by measuring the "zero level" without a sample or with a blackbody at the sample port.

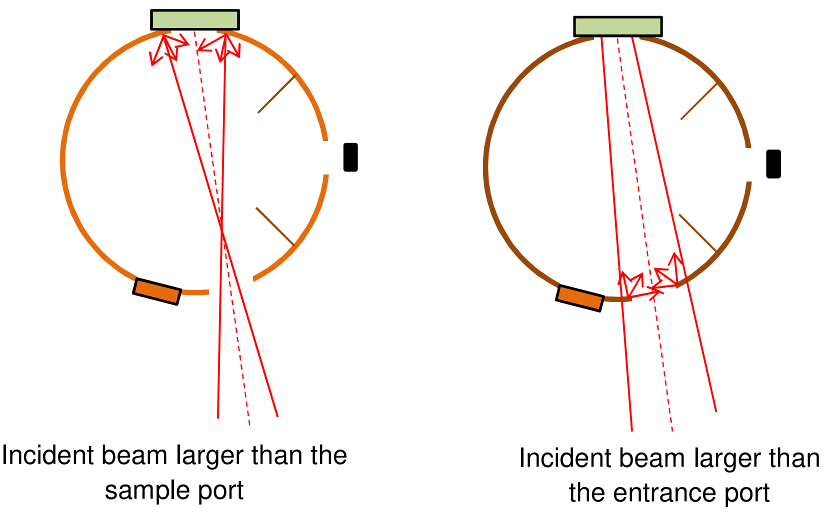

Figure 2: Optical configurations generating a permanent "zero signal"

In case of a high "zero signal", it is highly recommended to test the sensitivity of the integrating sphere response to the angular diffusion of the sample using indications in § 5.1.1. Indeed the sensitivity can be significantly affected by the "optical defects".

The decisions to use a specular standard, a diffusing standard or a mixed calibration should then be done with the same recommendations as in § 5.1.1.

5.1.3. Calibration of integrating spheres if the procedure for the correction of "single-beam sample-absorption error" is not applied

If a procedure for correcting for the "substitution error" is not applied, a systematic error exists when measuring the emissivity of a sample with an emissivity different from the one of the low emissivity standard. The emissivity result, without correction, is higher than the correct value if the emissivity of the sample is higher than the emissivity of standard.

A way to limit the error is to calibrate the integrating sphere at least two levels of emissivity, a low emissivity level (as usual) and a medium emissivity level. The sensitivity of the integrating sphere is then determined from the two measured signals and from the two emissivities of the standards. The difficulty can be to get a calibrated standard with a medium emissivity over a large spectral range. This is why, it is strongly recommended to try to have a procedure to correct the "substitution error".

5.1.4. Calibration of the spectral scale of Fourier Transform spectrometers (FTIRs)

The spectral scale generated by a Fourier Transform spectrometer can be controlled using a thin polystyrene foil calibrated for the spectral positions of some absorption peaks.

Usually, the uncertainty on the spectral scale has no effect on the uncertainty of the total near-normal emissivity measured on a reflective foil.

References

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