International Journal of Thermophysics, Vol. 26, No. 6, November 2005 (© 2005)
DOI: 10.1007/s10765-005-8609-0

Measurement of Infrared Spectral Directional Hemispherical Reflectance and Emissivity at BNM-LNE[1]

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An infrared reflectometer has been designed by BNM-LNE (Bureau National de Métrologie–Laboratoire National d'Essais) to measure the spectral directional hemispherical reflectance of solid materials at ambient temperature. For opaque materials, the spectral directional emissivity can be calculated from the measured reflectance. The reflectance can be measured from 0.8 to 14 μm in five directions with an angle of 12°, 24°, 36°, 48°, and 60° with respect to the normal to the surface of the sample. The optical arrangement to collect the reflected flux is based on the Coblentz arrangement (hemispherical mirror). In fact, four mirrors cut in an hemisphere are used to collect the flux reflected by the sample. This optical arrangement was chosen to limit the angle of incidence of rays on the detector (38° instead of 90° for the Coblentz arrangement). The final expanded uncertainty (level of confidence 95%) of the reflectance is estimated to be about ±0.03 for wavelengths between 0.8 and 10 μm and ±0.04 for wavelengths over 10 μm. The values of the spectral reflectance measured on a black paint and on a white ceramic tile are compared to those measured by the two laboratories PTB (Physikalisch Technische Bundesanstalt) and NIST (National Institute of Standards and Technology). The results validate the measurements performed at BNM-LNE.

KEY WORDS: solid materials; spectral directional emissivity; spectral directional hemispherical reflectance; uncertainty analysis.

[1] Paper presented at the Fifteenth Symposium on Thermophysical Properties, June 22–27, 2003, Boulder, Colorado, U.S.A.

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1. INTRODUCTION

For almost 20 years, BNM-LNE has developed and improved instruments to measure infrared radiative properties of materials. These properties are used for pyrometry, thermography, and heat balance calculation in many applications (research, industry processes, military applications). An infrared reflectometer was designed by BNM-LNE, some years ago, to measure the spectral directional hemispherical reflectance $\rho_\lambda(\theta, 2\pi)$ of solid materials at ambient temperature. For opaque materials, the spectral directional emissivity $\varepsilon_\lambda(\theta)$ can be calculated from the measured reflectance using the relation,

$$\varepsilon_\lambda(\theta) = 1 - \rho_\lambda(\theta, 2\pi). \tag{1}$$

The reflectometer, which is described, uses a novel optical arrangement (based on the Coblentz arrangement [1]) to collect the reflected radiation. Each source of uncertainty has been analyzed, and the overall uncertainty has been evaluated. The measurement results have been validated by comparison to other measurement techniques.

2. DESCRIPTION OF THE APPARATUS

Figure 1 shows a general diagram of the apparatus. Two sources can be used for the generation of the incident beam, a lamp for short wavelengths ($\lambda < 3\mu$m) and a blackbody for longer wavelengths. A monochromator with three gratings or a set of interference filters is used to select the spectral band (between 0.8 and 14μm). The radiation is modulated by a mechanical chopper preceding the monochromator or the filter. The incident beam is limited by a circular field stop and an aperture stop. The field stop determines the size of the spot on the specimen. A rotating flat mirror and a set of five flat stationary mirrors are used to focus the incident beam on the surface of the sample with one of the five possible angles of incidence ($12°, 24°, 36°, 48°, 60°$).

The principle used to collect the reflected flux, whatever the spatial distribution of the reflected radiation, is based on the Coblentz arrangement, but a set of four mirrors with spherical surfaces is used instead of a simple hemispherical mirror. The four mirrors were "cut" in an hemisphere as shown on Figs. 2 and 3. Figure 2 emphasizes the mirror with the entrance slots for the incident beam. Each of these four mirrors collects the radiation reflected in a quarter of the half space and focuses it at a point located in the same plane as the surface of the sample as illustrated on Figs. 4 and 5. The mirror M1 focuses the radiation on the point I1, the mirror M2 on the point I2, etc. Thus, the radiation reflected by

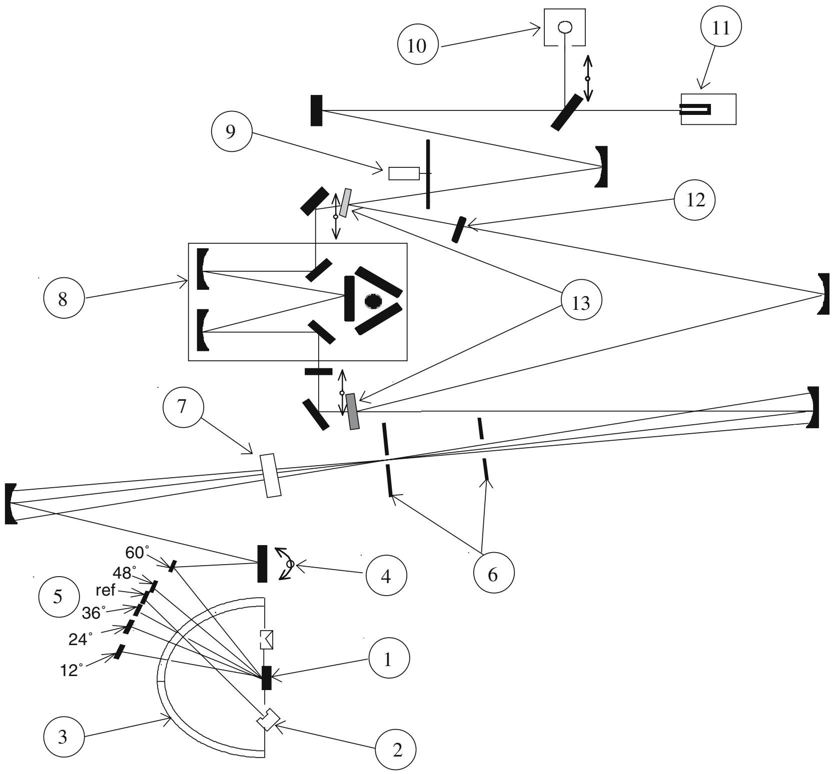

Fig. 1. Diagram of the apparatus: (1) sample; (2) detector; (3) set of four mirrors with spherical surface; (4) rotating plane mirror; (5) fixed plane mirrors for the selection of the angle of incidence; (6) field stop and aperture limiting stop; (7) polarizer; (8) grating monochromator; (9) mechanical chopper; (10) lamp source; (11) blackbody source; (12) interference filter; (13) flat mobile mirrors for the selection of the monochromator or the interference filters.

the specimen is focussed at four different points by the four mirrors. The pyroelectric detector (polyvinylidene fluoride film detector with a sensitive surface 10 mm in diameter) is successively placed at the four focal points. Thus, the sum of the four signals measured at the four focal points (the "sample" signal) is proportional to the total flux reflected by the specimen. When the detector is placed at one of the four focal points, three light traps are placed at the three other focal points to avoid interreflections. A mechanical rotating system carries the detector and the three light traps, and place them successively at the four positions. A "reference" signal is measured with the incident ray focussed directly on the detector.

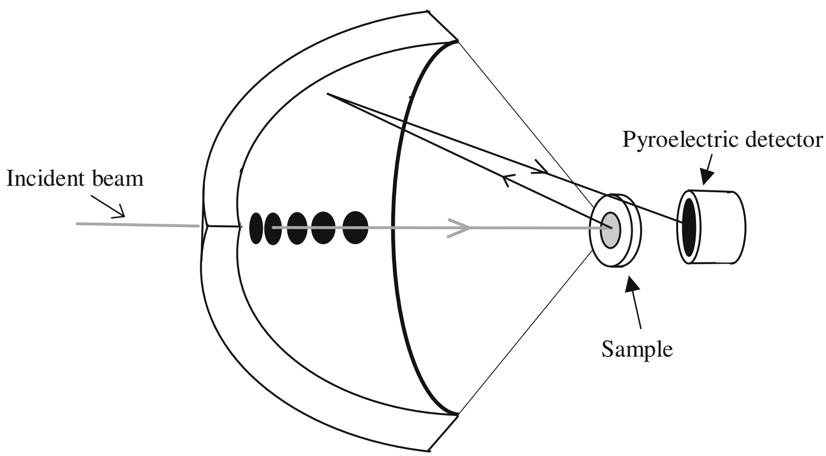

Fig. 2. Focussing of the reflected flux on the detector.

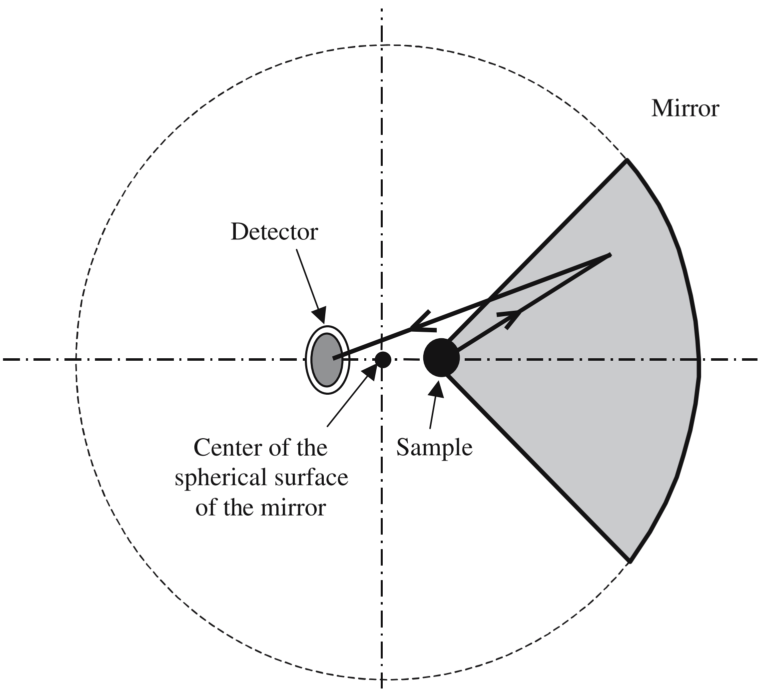

Fig. 3. Focussing of the reflected flux on the detector.

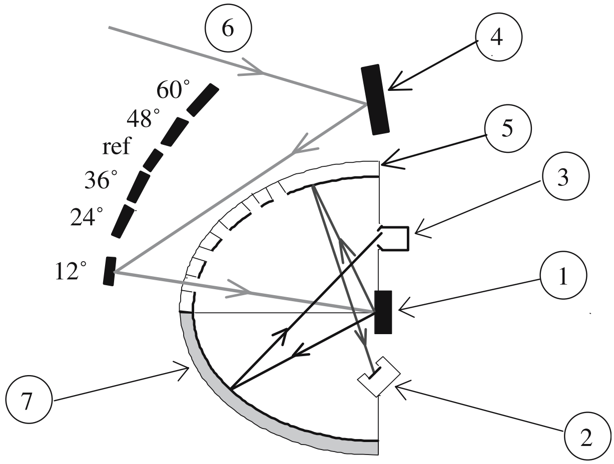

Fig. 4. Collection of the reflected radiation: (1) sample; (2) detector; (3) light trap; (4) rotating plane mirror; (5) collecting mirror M2; (6) incident beam; (7) collecting mirror M4.

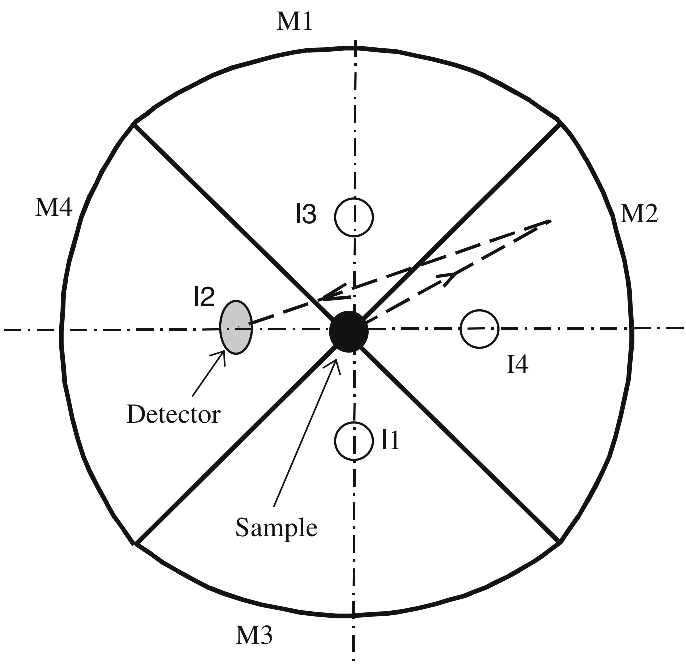

Fig. 5. Collection of the reflected radiation. Focussing of the reflected flux at four focal points.

The spectral directional hemispherical reflectance is then given by the relation:

$$\rho_\lambda(\theta, 2\pi) = \frac{1}{\rho_{\lambda\text{mirror}}} \frac{S_{\text{sample}}}{S_{\text{reference}}}, \tag{2}$$

where S_{sample} is the "sample" signal, $S_{\text{reference}}$ is the "reference" signal, $\rho_{\lambda\text{mirror}}$ is the spectral regular reflectance of the mirrors that collect the reflected radiation, λ is the wavelength, and θ is the angle that defines the direction of incidence. This optical arrangement was chosen to limit the angle of incidence on the detector; this angle is lower than 38°.

The reflectance of the four mirrors that collect the reflected flux has to be known to calculate the reflectance of the sample. Two plane mirrors were manufactured using the same process as for the spherical mirrors (same substrate, same treatment), and the specular reflectance of those mirrors was measured. For the measurement of specular reflectance, the two plane mirrors were, successively, placed as samples in the reflectometer. Assuming that the spectral reflectance of the plane mirrors is the same as the one of the collecting spherical mirrors, the ratio of the sample signal to the reference signal, measured in that configuration, equals the spectral reflectance of the mirrors squared.

3. EVALUATION OF UNCERTAINTY

The method used to calculate the uncertainty is the one recommended by the International Committee for Weights and Measures (CIPM) in the Guide to the Expression of Uncertainty in Measurement [2]. Uncertainties of all the parameters used in the calculation of the reflectance have been quantified. The uncertainty sources are: the uncertainty on the spectral reflectance factor of the collecting mirrors; the spatial non-uniformity of the sensitivity of the detector; the uncertainty on the linearity of the detection chain; the resolution of the detection chain; leakages of the reflected flux; the interreflection between the sample and the detector; the noise of the measured signals; the polarization of the incident radiation; the atmospheric absorption (difference of optical path between the "sample" measurement and the "reference" measurement); and the differences between the spectral regular reflectance of the mirrors that direct the incident beam on the sample ("incident" mirrors) and the "reference" mirror that directs the beam on the detector for the "reference" signal measurement.

The relative standard uncertainty of the spectral reflectance factor of the collecting mirrors is 1% for wavelengths between 0.8 and 1 μm and 0.5% for wavelengths above 1 μm.

The effect of the spatial non-uniformity of the sensitivity of the detector cannot be calculated rigorously as the real spatial distribution of radiation on the detector is not known. The spatial non-uniformity of the sensitivity has been measured, and the local sensitivity of the detector can change by about 10% over the sensitive area. The ratio of a signal measured with the ray focussed at the center of the sensitive area to a signal measured with almost all the sensitive area irradiated is equal to 1 ± 0.025. Thus, the relative standard uncertainty of the reflectance due to the spatial non-uniformity of the sensitivity is estimated to a level of 1.25%.

The linearity of the radiative measuring chain has been experimentally controlled using the flux addition method. The uncertainty due to linearity depends on the ratio "sample" signal to "reference" signal. The standard uncertainty due to linearity is 0.0025 for a ratio between 0.5 and 1; 0.005 for a ratio between 0.1 and 0.5; and 0.01 for a ratio between 0 and 0.1.

The uncertainty due to the resolution of the measurement is low compared to other sources of uncertainty, nevertheless, that uncertainty is taken into account.

The leakages of the reflected radiation arise mainly from the five slots drilled in one of the collecting mirrors. Light diffusion from the collecting mirrors, a tilt of the sample surface, and a bad position of the sample surface can also generate leakages. For a perfectly diffusing sample, the relative leakage through the five slots is equal to 0.62% of the reflected flux. For non-specular materials, a correction of 0.31% is made to take into account those leakages of radiation and the related relative standard uncertainty on the reflectance is 0.16%. The relative standard uncertainty evaluated for the leakages resulting from a tilt of the sample surface, the diffusion on the collecting mirrors, and a bad position of the sample is 0.2%. Thus, the overall relative standard uncertainty for the leakages is 0.26%.

The interreflections between the sample and the detector produce the same relative increase of the "sample" signal and "reference" signal. Therefore, in theory, the effect of the interreflections on the measured reflectance is null.

The optical path for the "sample" signal measurement is 0.2 m longer than the one for the "reference" signal measurement (twice the collecting mirrors curvature radius). Thus the "sample" signal is affected by the atmospheric absorption in the spectral bands 2.5–3 and 5–7.5 μm due to the water vapor absorption and in the spectral band 4–4.6 μm due to the CO_2 absorption. The optical system is located in a closed chamber, and a flow of dried air (dew point below $-20°C$) is maintained during the measurements. The resulting relative standard uncertainty due to the atmospheric absorption is 0.25% for the spectral bands 2.5–3 and 4–4.6 μm and is 0.5% for the spectral bands 5–7.5 μm.

The five plane mirrors that direct the incident beam on the sample ("incidence" mirrors) are supposed to have the same spectral reflectance as the plane mirror that directs the beam on the detector for the "reference" signal measurement ("reference" mirror). The set of six mirrors was supplied from the same manufacturer, so the substrate and the coating are the same. The differences of spectral reflectance arise mainly from the difference of ageing between the mirrors. The angles of incidence are almost the same (within 6°) for the six mirrors. The relative standard uncertainty of the reflectance coming from a possible difference of spectral reflectance between the "incidence" mirror and the "reference" mirror is evaluated to 0.5%. In the same way, the angle of incidence on the plane rotating mirror used to select the incidence is not the same for the "reference" signal measurement as for the "sample" signal measurement. The related relative standard uncertainty is 0.5%.

The noise on the measured radiative signals can be significant compared to the reference signal. Thus, for each radiative signal, the measurements are repeated several times and the mean value and the standard deviation are calculated. The uncertainty on the "sample" signal to "reference" signal ratio is then calculated by an adapted combination of the standard deviations of each radiative signal.

The polarization of the incident beam has been analyzed. When an interference filter is used for the spectral selection, the incident beam is almost unpolarized; the relative difference between the two polarization components is less than 2%. When the monochromator is used, the beam is significantly polarized, and the intensity ratio between the two polarization components can be larger than 2. Practically in most cases, the polarizer is not used because, when the incident beam is linearly polarized, the incident flux is almost divided by two and the signal-to-noise ratio is too low. Thus, when the incident beam is not linearly polarized, the relevant relative standard uncertainty component is evaluated to 0%, 1%, 1.5%, 2% and 3.5%, respectively, for the angles of incidence of 12°, 24°, 36°, 48°, and 60°.

The uncertainty related to the spectral bandwidth used for the measurement is not taken into account. The spectral bandwidth is just given as a measurement parameter.

In the evaluation of uncertainties, the character of reflection (diffuse, specular or mixed) is not analyzed specifically except for the leakages of the reflected radiation through the entrance slots. For the sample signal measurement, the character of the reflection influences directly the directional and spatial distribution of the radiation on the detector and the ratio of the sample signal to the reference signal. For the case of pure specular or mixed reflection, the uncertainty due to the spatial non-uniformity of the detector is considered equal to the one for a very diffusing sample, which is the worst situation. For the uncertainty due to the linearity defects of the radiometric chain, the real values of the ratios of the "sample" signal to the "reference" signal are considered in all cases.

4. VALIDATION OF MEASUREMENTS

Measurements were performed by LNE on a white ceramic tile (SRM 2019C) supplied and certified by the National Bureau of Standards (NBS) in 1983 for spectral directional hemispherical reflectance in the spectral range from 0.250 to 2.50 μm and at a 6° incidence angle; the results from NBS are given in Ref. 3. The results of LNE are given in Tables I and II for the spectral range of 0.8–14 μm and for the five directions. The results of NBS (6° incidence angle) and those of LNE (12° incidence angle) are compared in Table III for the spectral range 0.8–2.5 μm. The results of LNE (Table II) show that the spectral reflectances are almost the same for the two incidence angles 12° and 24°. Thus, we consider that the variations of spectral reflectance with the incidence angle are insignificant (noting the uncertainties) between 6° and 12°. The differences of spectral reflectances between BNM-LNE and NIST on the high reflecting white ceramic tile are very small compared to the uncertainties.

Measurements were also performed by BNM-LNE and by PTB (Ref. 4) on the black paint Nextel Velvet Coating 811-21 in the spectral

Table I. Results of Reflectance Measurements on the White Ceramic (SRM 2019c)

Wavelength (μm)	Reflectance incidence: 12°	Reflectance incidence: 24°	Reflectance incidence: 36°
0.8	0.855 ± 0.032	0.858 ± 0.036	0.863 ± 0.041
1.0	0.844 ± 0.027	0.850 ± 0.036	0.856 ± 0.041
1.5	0.862 ± 0.028	0.869 ± 0.033	0.873 ± 0.039
2.0	0.855 ± 0.028	0.866 ± 0.033	0.868 ± 0.038
2.5	0.826 ± 0.028	0.832 ± 0.033	0.829 ± 0.038
3.0	0.387 ± 0.015	0.413 ± 0.018	0.393 ± 0.020
4.0	0.322 ± 0.013	0.325 ± 0.015	0.332 ± 0.017
6.0	0.039 ± 0.015	0.035 ± 0.015	0.046 ± 0.015
8.03	0.043 ± 0.017	0.043 ± 0.017	0.055 ± 0.018
10.0	0.254 ± 0.019	0.250 ± 0.020	0.260 ± 0.022
14.0	0.131 ± 0.039	0.113 ± 0.039	0.155 ± 0.042

Table II. Results of Reflectance Measurements on the White Ceramic (SRM 2019c)

Wavelength (μm)	Reflectance incidence: 48°	Reflectance incidence: 60°
0.8	0.866 ± 0.047	0.881 ± 0.062
1.0	0.860 ± 0.047	0.875 ± 0.062
1.5	0.878 ± 0.045	0.896 ± 0.061
2.0	0.876 ± 0.045	0.887 ± 0.061
2.5	0.836 ± 0.044	0.855 ± 0.059
3.0	0.414 ± 0.024	0.422 ± 0.031
4.0	0.338 ± 0.020	0.366 ± 0.027
6.0	0.048 ± 0.015	0.083 ± 0.015
8.03	0.084 ± 0.020	0.120 ± 0.023
10.0	0.264 ± 0.024	0.306 ± 0.031
14.0	0.149 ± 0.042	0.180 ± 0.047

Table III. Comparison of the Results of BNM-LNE to those of NIST (NBS) on the White Ceramic (SRM 2019c)

Wavelength (μm)	BNM–LNE reflectance incidence: 12°	NIST (NBS) reflectance incidence: 6°	Difference LNE − NBS
0.8	0.855 ± 0.032	0.854 ± 0.01	0.001
1.0	0.844 ± 0.027	0.851 ± 0.01	−0.007
1.5	0.862 ± 0.028	0.859 ± 0.01	0.003
2.0	0.855 ± 0.028	0.863 ± 0.01	−0.008
2.5	0.826 ± 0.028	0.838 ± 0.01	−0.012

range from 4 to 14 μm; the results are given in Table IV. For the black paint, BNM-LNE measured the spectral directional hemispherical reflectance for the incidence angle 12° and then calculated the spectral directional emissivity while PTB measured directly the spectral normal emissivity by comparison of the spectral radiance of the sample to that of a blackbody. The spectral directional emissivity measured by LNE for the direction 12° is compared directly to the spectral normal emissivity measured by PTB. The results of spectral directional emissivity performed by PTB for different angles [4] show that the variation of spectral directional emissivity with direction is insignificant for incidence angles below 30°. The differences between the spectral emissivities measured by BNM-LNE and PTB on the black paint remain within the uncertainty estimates.

Table IV. Comparison of the Results of BNM-LNE to Those of PTB for the Black Paint (Nextel Velvet Coating 811-21)

Wavelength (μm)	BNM-LNE spectral emissivity incidence: 12°	PTB spectral emissivity incidence: 0°	Difference LNE − PTB
0.8	0.964 ± 0.01		
1.0	0.964 ± 0.01		
2.0	0.962 ± 0.01		
3.0	0.965 ± 0.015		
4.0	0.960 ± 0.02	0.975 ± 0.032	−0.015
4.5	0.962 ± 0.02	0.966 ± 0.011	−0.004
8.0	0.959 ± 0.025	0.974 ± 0.006	−0.015
10.0	0.950 ± 0.027	0.967	−0.017
12.0	0.940 ± 0.035	0.972	−0.032
14.0	0.940 ± 0.045	0.973	−0.033

5. CONCLUSION

The uncertainties have been analyzed in detail. For low reflecting samples, the main sources of uncertainty include the noise of the radiative signals. This can be explained by the low radiative detectivity of the pyroelectric detector and by the high sensitivity of this detector to mechanical and acoustical vibrations. For high reflecting materials, the main source of uncertainty is the non-uniformity of the sensitivity of the pyroelectric detector over the sensitive area.

The reflectometer developed by BNM-LNE can be used to perform reliable spectral directional hemispherical reflectance measurements on any solid material at ambient temperature with absolute uncertainties from 0.02 to 0.045 (level of confidence 95%). These levels of uncertainty are suitable for most industrial applications.

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