EUROMET PROJECT
FINAL REPORT

1.	Ref. No.: 516	2. Subject Field: Thermometry	
3	Type of collaboration: Intercomparison of measurement standards		
4A.	Partners: (institutions)	BNM-LNE (Trappes - France) PTB (Braunschweig - Germany)	4B. CEC funded? No
5.	Participating countries: FR- DE		
6.	Title: Intercomparison of total hemispherical and normal spectral emissivity measurements		
7.	Progress:		

1. Measurement programme

The measuring conditions for the intercomparison are given in the following table. It has been decided to perform the measurements on the same samples in the two laboratories to reduce the discrepancies resulting from the sample preparation.

	Temperatures	Wavelength	Atmosphere
Total hemispherical emissivity	40 °C, 150 °C, 200 °C	all the spectrum	vacuum
Normal spectral emissivity	150 °C, 350 °C, 550 °C	2,5 µm to 13 µ	air

2. Selection and preparation of the samples

The selected materials were AISI 304 L stainless-steel for the total emissivity and Inconel 600 alloy for the normal spectral emissivity measurements. The samples were sandblasted and oxidised in air, the sample of AISI 304 L at 360 °C and that of Inconel 600 at 800 °C. During the preparation, the homogeneity of the sample surfaces was checked visually and the stability of the oxidation process was checked by weighing the samples before and after heat cycling.

For the spectral emissivity measurements, a disc 30 mm in diameter was required by BNM-LNE and a disc 100 mm in diameter by PTB. The "sample" for PTB, therefore, consisted of two parts: the disc suitable for BNM-LNE and a ring (int. diam. 30mm, ext. diam. 100 mm).

For total hemispherical emissivity measurements, two discs of the same material (62.2 mm in diameter) were required by BNM-LNE, while PTB needed a disc 100 mm in diameter. The "sample" for PTB, therefore, comprised one of the two discs (62.2 diam.) and a ring (int. diam. 62.2 mm, ext. diam. 100 mm). All the parts constituting one sample (same material) were, of course, prepared in the same way to achieve radiative characteristics as identical and homogeneous as possible.

3. Methods and procedures of measurement

3.1. Normal spectral emissivity

The two laboratories used the same direct technique based on the comparison of the spectral radiance of the sample with that of a blackbody. The techniques were essentially different regarding the technical components, the spectroradiometers in particular, and the procedures for the measurement of the sample's surface temperature.

3.1.1. BNM-LNE

For normal spectral emissivity measurements, BNM-LNE uses a home-made spectroradiometer with interferential filters for the spectral selection. The spectral emissivity is given by the ratio of radiometric signals from the sample and the blackbody, corrected by the ratio of the real spectral radiance of the blackbody to that of the sample. The radiances are calculated taking the temperatures measured in the blackbody and on the sample into account (the two temperatures may differ) [1]. The calculation of the emissivity takes in account the real spectral sensitivity of the spectroradiometer for each spectral band. The surface temperature of the sample is measured using the "gradient technique". Two type S thermocouples are radially inserted in the sample at known depths. The gradient in the sample is assumed to be constant and the surface temperature is linearly extrapolated from the two measured temperatures.

3.1.2. PTB

PTB uses a modified NICOLET FTIR spectroradiometer for the measurement of the radiative fluxes emitted by the sample and the blackbody [3]. The sample of known thermal conductivity is attached to an electrically heated sample holder and is surrounded by a thermostated black enclosure. The temperatures of sample holder and enclosure are measured using resistance thermometers. The directional spectral emissivity is defined as the ratio of radiometric signals from the sample and the blackbody, corrected by a factor appropriately constructed from Planck's functions for the temperatures of spectrometer detector, blackbody and sample surface. The latter is determined indirectly by equalisation of the heat flux between holder and sample surface and the heat loss from the surface due to radiation and air convection. The total hemispherical emissivity required to estimate the radiative loss is calculated from the directional spectral emissivity by integrating it over wavelength and angle. The evaluation is realised in an iterative approach, including alternative calculation of the surface temperature and the total hemispherical emissivity until a convergence is achieved. The spectral resolution power for radiative measurements is much higher for PTB (FTIR) than for BNM-LNE (interferential filters). To make a comparison of the results of the two laboratories possible, the spectral emissivities of PTB were corrected with the aid of the following relation, taking into account the real spectral sensitivity of the BNM-LNE spectroradiometer.

$$\varepsilon_s'(\lambda_f) = \left[\int_{\lambda_{min}}^{\lambda_{max}} \varepsilon_s'(\lambda).S_\lambda.L_\lambda^\circ(T_s).d\lambda\right] \Big/ \left[\int_{\lambda_{min}}^{\lambda_{max}} S_\lambda.L_\lambda^\circ(T_s).d\lambda\right]$$

S_λ	: spectral sensitivity of the LNE's spectroradiometer,
$\varepsilon_s'(\lambda)$	: spectral emissivity measured by PTB.
λ_f	: central wavelength of the LNE's interferential filter,
λ_{min} - λ_{max}	: spectral band of the LNE's interferential filter
T_s	: surface temperature of the sample,
L_λ°	: Planck's function.

3.2. Total hemispherical emissivity

3.2.1. BNM-LNE

A calorimetric technique is used by BNM-LNE.
Two samples (discs) made of the same material and attached to a sample holder are heated simultaneously in a vacuum chamber. The walls of the chamber, coated with black paint, are thermostated by liquid nitrogen boiling at atmospheric pressure (78 K). The samples, surrounded by guard-rings heated at the same temperature as the samples, radiate only by the circular surface facing the chamber walls. The electrical power required to ensure stable temperatures inside the samples and the surface temperatures of the samples are measured. For surface temperature measurements, two thermocouples are radially inserted at known depth in each sample and the surface temperature is linearly extrapolated. The total hemispherical emissivity can then be roughly calculated according to the following relation :

$\varepsilon = P / [S . \sigma . (T_s^4 - T_0^4)]$ with :

P : electrical power,
S : radiating surface of the samples,
σ : Stefan-Boltzmann constant,
T_S : sample surface temperature,
T_0 : temperature of the chamber walls (78 K).

In fact a more complicated relation is used to consider the reflections of radiative fluxes on the chamber walls.

3.2.2. PTB

PTB uses the radiometric technique [2], i.e. comparison of the total (all wavelengths) directional radiance of the sample (angles from 0° to 75°) with that of a blackbody. The sample holder, the blackbody and the radiometer are placed in a vacuum chamber. The radiometer can be rotated so that either the sample or the blackbody can be seen. The sample is surrounded by a black enclosure thermostated at the same temperature as the receiver of the radiometer (a Moll type thermopile). The temperatures of sample holder and of blackbody are measured. The total hemispherical emissivity is calculated from the directional emissivity by integration over the angle. As in the PTB's spectral emissivity technique, the temperature of the sample surface is calculated iteratively using the energy conservation principle at the surface.

4. Results

4.1. Normal spectral emissivity

The results are given in tables 2,3 and 4.
To check the stability of the sample, PTB performed spectral emissivity measurements on Inconel 600, at 150 °C, before and after the measurements at LNE. The differences of the two series of results are within ± 0.01 .

4.2. Total hemispherical emissivity

The results are given in table 1.
For BNM-LNE, the results are the averages of the emissivities of the two samples. For PTB, the result given for 40°C is that measured on sample 1. For 150°C and 200°C, the values are the averages of the emissivities of the two samples. The differences between the two samples, measured by PTB at 150°C and 200°C, are lower than or equal to 0.006. BNM-LNE performed measurements before and after the measurements at PTB. The differences between the two series of results are within ± 0.0008 for the three temperatures.

5. Discussion

For the normal spectral emissivity measurements, the agreement between the two laboratories is quite good. A significant discrepancy is only found at the temperature 350°C for the wavelength 2.52 µm . One must keep in mind that 2.5 µm is the lower spectral limit of the PTB's FTIR spectrometer and that atmospheric absorption occurs around that wavelength, so that radiative signals (by FTIR) are very noisy. For the three temperatures, the spectral emissivities measured by PTB are lower than those of BNM-LNE (see Figure 1), except for the wavelength of 6.4 µm. The atmospheric absorption by water vapour can explain the singularity at that wavelength. The spectral curves of the relative differences between PTB and BNM-LNE are not typical of errors in surface temperature measurements. For wavelengths over 3 µm, all the differences between the results of the two laboratories are smaller than the root-sum-squares of the expanded uncertainties (k=2) claimed by the laboratories and, therefore, can be explained by the measurement uncertainties. Their main sources are the inhomogeneity of the sample surface, the non-linearity of the spectroradiometers, the surface temperature determination and the diffuse radiation (two mirrors in the spectroradiometer of BNM-LNE are slightly "diffuse"). The results confirmed the validity of the uncertainty analysis by the laboratories.

For the total hemispherical emissivity measurements, the results agree within the combined uncertainties even with a coverage factor k=1. The uncertainties of BNM-LNE are much lower than those of PTB, probably because the method of measurement is more direct and requires fewer parameters to be measured or known. The uncertainties of the PTB measurements is probably overestimated, especially for higher temperatures.

6. Conclusions

The results of the intercomparison show good agreement for the measurements of the normal spectral emissivity and the total hemispherical emissivity at PTB and BNM-LNE.
The measurement of the spectral emissivity around 2.5 µm using a FTIR spectrometer appears to be critical because of a bad signal-to-noise ratio and atmospheric absorption.
Sandblasted oxidised stainless steel and sandblasted oxidised Inconel are materials suitable for emissivity intercomparisons. They can be regarded as potential reference materials for emissivity measurements at low and medium temperatures.

Sample température	BNM-LNE Emissivity	BNM-LNE Uncertainty	PTB Emissivity	PTB Uncertainty	Difference PTB - LNE
40 °C	0.399	0.0060	0.420	0.04	0.021
150 °C	0.427	0.0068	0.433	0.02	0.006
200 °C	0.441	0.0075	0.438	0.02	-0.003

Table 1 : Total hemispherical emissivity of oxidised AISI 304L stainless steel.

Wavelength	BNM-LNE		PTB		Difference
(µm)	value	Uncertainty	value	Uncertainty	(PTB – LNE)
2.52	0.817	0.060	0.808	0.50	-0.009
3.62	0.805	0.040	0.792	0.04	-0.013
3.97	0.794	0.036	0.785	0.04	-0.009
4.53	0.779	0.031	0.767	0.02	-0.012
5.26	0.747	0.028	0.739	0.02	-0.008
6.43	0.675	0.024	0.685	0.02	0.010
8.05	0.596	0.020	0.586	0.02	-0.010
8.60	0.569	0.018	0.560	0.02	-0.009
9.88	0.520	0.016	0.503	0.02	-0.017
10.40	0.483	0.014	0.476	0.02	-0.007
11.71	0.441	0.013	0.426	0.02	-0.015
12.70	0.404	0.012	0.400	0.02	-0.004

Table 2 : Normal spectral emissivity of oxidised Inconel 600 at 150 °C.

Wavelength	BNM-LNE		PTB		Difference
(µm)	value	Uncertainty	value	Uncertainty	(PTB – LNE)
2.52	0.825	0.033	0.720	0.040	-0.105
3.62	0.810	0.028	0.788	0.030	-0.022
3.97	0.803	0.027	0.786	0.030	-0.017
4.53	0.787	0.026	0.769	0.020	-0.018
5.26	0.755	0.024	0.741	0.020	-0.014
6.43	0.685	0.019	0.694	0.020	0.009
8.05	0.613	0.018	0.588	0.020	-0.025
8.60	0.585	0.016	0.562	0.020	-0.023
9.88	0.519	0.015	0.505	0.020	-0.014
10.40	0.493	0.014	0.481	0.020	-0.012
11.71	0.445	0.012	0.436	0.020	-0.009
12.70	0.418	0.012	0.410	0.020	-0.008

Table 3 : Normal spectral emissivity of oxidised Inconel 600 at 350 °C.

Wavelength	BNM-LNE		PTB		Difference
(µm)	value	Uncertainty	value	Uncertainty	(PTB – LNE)
2.52	0.836	0.025	0.702	0.250	-0.134
3.62	0.818	0.020	0.789	0.050	-0.029
3.97	0.810	0.020	0.789	0.050	-0.021
4.53	0.793	0.020	0.774	0.030	-0.019
5.26	0.763	0.019	0.746	0.030	-0.017
6.43	0.688	0.017	0.690	0.030	0.002
8.05	0.615	0.015	0.595	0.020	-0.020
8.6	0.587	0.015	0.569	0.020	-0.018
9.88	0.530	0.013	0.513	0.020	-0.017
10.4	0.504	0.013	0.490	0.020	-0.014
11.71	0.457	0.011	0.446	0.020	-0.011
12.7	0.431	0.011	0.420	0.020	-0.011

Table 4 : Normal spectral emissivity of oxidised Inconel 600 at 550 °C.

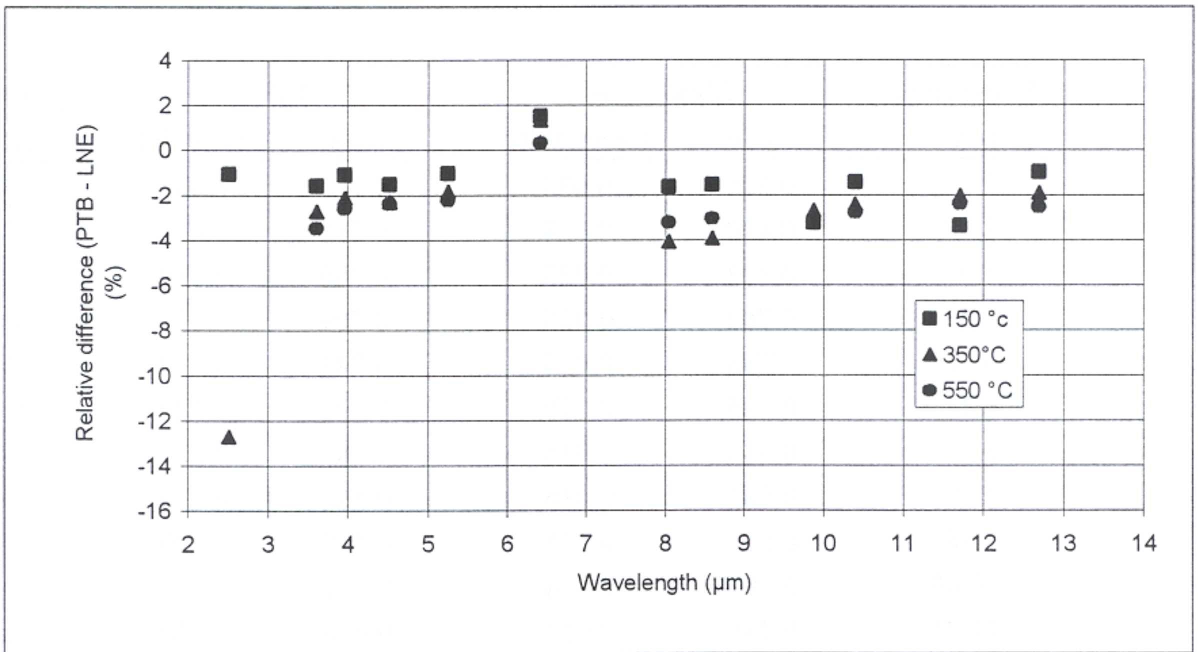

Figure 1 : Relative differences (in %) between normal spectral emissivities measured by PTB and BNM-LNE.

<u>**References**</u>

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9. Completion date: December 31st 1999	10. Coordinator´s signature:	11. Date: May 4th 2000