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Report on the CCT Supplementary Comparison S1 of Infrared Spectral Normal Emittance/Emissivity

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Abstract

The National Measurement Institutes (NMIs) of the United States, Germany, France, Italy and Japan, have joined in an inter-laboratory comparison of their infrared spectral emittance scales. This action is part of a series of supplementary inter-laboratory comparisons (including thermal conductivity and thermal diffusivity) sponsored by the Consultative Committee on Thermometry (CCT) Task Group on Thermophysical Quantities (TG-ThQ). The objective of this collaborative work is to strengthen the major operative National Measurement Institutes' infrared spectral emittance scales and consequently the consistency of radiative properties measurements carried out worldwide. The comparison has been performed over a spectral range of 2 μm to 14 μm , and a temperature range from 23 $^{\circ}\text{C}$ to 800 $^{\circ}\text{C}$. Artefacts included in the comparison are potential standards: oxidized inconel, boron nitride, and silicon carbide. The measurement instrumentation and techniques used for emittance scales are unique for each NMI, including the temperature ranges covered as well as the artefact sizes required. For example, all three common types of spectral instruments are represented: dispersive grating monochromator, Fourier transform and filter-based spectrometers. More than 2000 data points (combinations of material, wavelength and temperature) were compared. Ninety-eight percent (98%) of the data points were in agreement, with differences to weighted mean values less than the expanded uncertainties calculated from the individual NMI uncertainties and uncertainties related to the comparison process.

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Keywords

Emittance; Emissivity; Infrared; Standards; Comparison; Spectral

1 INTRODUCTION

Infrared emittance is an important property for a wide variety of both thermal and optical applications. These include non-contact temperature measurements for industrial materials processing, energy conservation, structural defect detection, infrared signature detection and reduction, etc. In addition, emittance data is critical for thermal modeling of instrumentation from cryogenic radiometers to spacecraft payloads. A number of the major National Measurement Institutes (NMIs) have developed measurement capabilities and standards to support industrial needs in these areas for accurate emittance measurements. In recent decades, several inter-laboratory comparisons of infrared spectral emittance capabilities of NMIs have been conducted, including the Laboratoire National de Métrologie et d'Essais (LNE) of France, National Physical Laboratory (NPL) of the United Kingdom, the CNR Istituto di Metrologia "G. Colonnelli" (IMGC) of Italy (now the Istituto Nazionale di Ricerca Metrologica (INRIM)), and Physikalisch-Technische Bundesanstalt (PTB) of Germany. All but one of these have been bilateral or involved only two NMIs.^{1,2,3} The previous work and comparisons have been important to evaluate the sources of error and develop comprehensive uncertainty budgets and have helped to form a basis upon which to pursue a more comprehensive comparison. The CCT – WG 9 was formed in 2003 to help identify critical thermophysical quantities for which there was a need for comparisons (i.e. in order to strengthen the major NMI's infrared spectral emittance scales in support of radiative properties measurements around the world. The specific quantity being compared is the normal directional spectral emittance^{i,ii}, defined by:

$$\varepsilon(\lambda, T, 0^\circ) = L_\lambda(\lambda, T, 0^\circ) / B_\lambda(\lambda, T, 0^\circ) \quad , \quad (1)$$

where λ is the wavelength, T is the artefact temperature, $L_\lambda(\lambda, T, 0^\circ)$ is the spectral radiance per unit wavelength of the artefact in the normal direction, and $B_\lambda(\lambda, T, 0^\circ)$ is the spectral radiance per unit wavelength of the ideal blackbody in the normal direction as given by Planck's Law.

Table 1 shows a list identifying all of the variables contained in this report, along with brief description and locations where they are used.

The comparison was designed to be comprehensive, with the ranges of wavelength (2 μm to 14 μm) and temperature (23 $^\circ\text{C}$ to 800 $^\circ\text{C}$) selected to span the NMIs' capabilities to the

ⁱNote that for materials the term "emissivity" is an equivalent term, used perhaps more often than "emittance". However, the pilot laboratory prefers consistency with the terminology of the larger family of properties: transmittance, reflectance and absorptance, reserving the term "emissivity" for use in the description of blackbody sources. Standards dictionaries have not exclusively selected one over the other. For internal consistency, other than in the title, this note, and references, when discussing samples, we use only one term, "emittance", throughout this report.

ⁱⁱFrom this point on, for simplicity we will assume but not specifically denote the normal direction (0°).

extent that there would be results from at least two NMIs for each data point. Three materials with a range of emittance values, and being considered as potential standards, were selected for inclusion in the comparison: oxidized Inconel (OxIn), boron nitride (BN), and silicon carbide (SiC).

The original set of participants of the inter-laboratory emittance comparison included the NPL, the National Metrology Institute of Japan (NMIJ), LNE, the National Institute of Standards and Technology (NIST) of the United States, INRIM, with NPL serving as the coordinating pilot laboratory. NPL selected and produced the sets of oxidized Inconel (OxIn) and boron nitride (BN) artefacts, used in the comparison. NPL also developed an initial measurement protocol. Before measurements were begun, however, due to an organizational restructuring, NPL was not able to continue their participation. NIST then took over as the pilot laboratory. NIST produced and distributed a third artefact set of silicon carbide (SiC) and also revised the measurement protocol. At approximately the same time as NPL's withdrawal, the PTB joined the comparison as the fifth participant.

2 DESCRIPTION OF THE COMPARISON

2.1 Participants Parameter Ranges

Due to the variety of instrument designs and methods employed by the participants and detailed in Section 2.3, their ideal artefact sizes are different and incompatible with each other, with the exception of NMIJ and PTB. This meant that different artefacts had to be produced for each participant. It also meant that no participant, including the pilot, would be able to measure all the artefacts at all temperatures. It was possible, however, for the pilot lab (NIST) to measure all artefacts at room temperature (RT), which was done both before and after all other measurements by the other participants. Since PTB joined the comparison after the OxIn and BN artefacts were produced, but before the SiC artefacts were made, a second 45 mm SiC artefact is shown in the photo and was used for PTB. The artefact size for each participant is shown in Table 2.

2.2 Standard Artefacts

As stated above, three sets of artefacts were selected and produced for the comparison: BN, OxIn, and SiC. All three materials are nominally stable under heating in air after an appropriate bakeout preparation regime and are under consideration as potential candidate standard reference artefacts. In the past, OxIn has been a NIST (formerly NBS) Standard Reference Material 1440,⁴ BN has been extensively studied at NPL,⁵ and more recently, SiC has been investigated at NIST.⁶

Production of the artefact sets took place over more than one year. Tests were performed on an initial set of BN and OxIn artefacts produced by NPL. Due to significant variation between samples and initial heat treatment difficulties found after testing, second replacement sets were produced and processed. Although not ideal (Section 2.5.1.1), improvements were deemed sufficient to proceed with the comparison. The SiC artefacts obtained and heat treated by NIST were found to be acceptably uniform and stable. Details

of the materials' source, composition, heat treatment, and characterizations are given in Table 3.

Photos of the artefact sets used in the comparison are shown in Figure 1: BN in (a), OxIn in (b), and SiC in (c). 10 mm diameter $\times$ 10 mm thick artefacts, produced for NPL are shown, but were not used in the comparison.

2.3 Comparison Process Schedule

The comparison was carried out in a “star” mode in which the samples are provided by the pilot laboratory and sent out to all the participants at approximately the same time. This was done primarily because of the incompatibility of the measurement systems to a single artefact size, as described above. Other factors, including the overall duration of the comparison and the risk of artefact loss or damage made the star mode preferable. At the start of the measurement campaign, NPL sent the sets of BN and OxIn artefacts to NIST for measurement at room temperature (23 °C) in middle of 2007. Subsequently the artefacts were distributed to the other participants in late 2007. After the change in pilot laboratory, NIST measured the SiC artefacts at room temperature and delivered to participants in early 2008.

Measurements by all the participants were completed, and the artefacts returned to NIST by the end of 2008. NIST completed the repeat room temperature measurements of all artefacts by early 2009. Additional details of the history and process are given in Appendix A, the Technical Protocol.

2.4 Instrument, Method and Uncertainties Descriptions

2.4.1 NMIJ—The NMIJ measurement system employs an FTIR spectrometer, and is designed for normal spectral emittance measurements of opaque solid materials near ambient temperatures.⁷ A schematic of the system is shown in Figure 2. A liquid nitrogen cooled photovoltaic mercury cadmium telluride (MCT) detector operated with a current mode preamplifier, which has a high sensitivity and a highly linear response, was used instead of the conventional photoconductive MCT detector operated with a voltage mode preamplifier. In order to reduce the air absorption effect, all optical components including the sample and the reference blackbodies are operated in vacuum. The spectrometer is calibrated against high quality reference blackbodies. An appropriate phase correction technique is used to cancel the phase error caused by the emission from the surroundings.⁸ The sample spectral radiance, $L_v(\nu)$ is obtained from

$$L_v(\nu, T) = \text{Re} \left[\frac{C_s(\nu) - C_0(\nu)}{C_h(\nu) - C_0(\nu)} \right] \cdot [B_v(\nu, T_h) - B_v(\nu, T_0)] + B_v(\nu, T_0) = |L_v| \exp(i\phi_L) \quad ,$$

(2)

where ν is the wavenumber, C are the complex spectra obtained from viewing the sample, s , the cold blackbody, 0, and the hot blackbody, h ; B are the ideal blackbody spectral radiances for the hot and cold blackbodies; and ϕ_L is the phase.

$$L_\nu(\nu, T) = \varepsilon(\nu, T_s) \cdot B_\nu(\nu, T_s) + [1 - \varepsilon(\nu, T_s)] \cdot B_\nu(\nu, T_{\text{ambient}}) \quad (3)$$

is then solved for $\varepsilon(\nu, T_s)$ to obtain the sample emittance. Refinements are also made that take into account variations in the different regions of the background, which are seen by the sample.

An example uncertainty analysis for the BN sample is shown in Table 4.⁹ The same uncertainty analysis was performed for all intercomparison measurements with the resultant expanded uncertainties (combined uncertainties times $k=2$) provided. The emittance values and expanded uncertainties, after the processing discussed in Section 2.4, are shown in the results Tables in Appendix A.

2.4.2 LNE—LNE uses a custom designed infrared reflectometer to measure the spectral directional hemispherical reflectance of solid materials at ambient temperature.¹⁰ For opaque materials, the spectral directional emittance can be calculated from the measured reflectance. The reflectance can be measured from 0.8 μm 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). Four mirrors cut in a 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 μm and 10 μm and 0.04 for wavelengths over 10 μm . The uncertainty budget for emittance measurement of a comparison artefact is shown below in Table 5.

An apparatus for the measurement, in the temperature range of 200 °C to 800 °C, of normal spectral emittance of opaque good thermal conductors is shown in Figures 4 and 5, in side and top views, respectively.¹¹ The direct method based on the comparison of the spectral radiance of the sample with the spectral radiance of a blackbody is used. The apparatus is designed to enable broad control of influencing parameters.

The sample and the reference blackbody are located in a vacuum chamber, which enables measurements either with air or inert atmosphere. The reference blackbody is a V-grooved and oxidized refractory steel cavity heated with an electrical furnace. The temperature is determined by a calibrated type S thermocouple placed at the level of the bottom of the cavity. The thermocouple is calibrated periodically and the spectral radiance of the blackbody is also calibrated periodically by comparison to a portable calibrated blackbody. A temperature-controlled shield at ambient temperature, located at the ceiling of the chamber, is used to control the background temperature. The shield is coated with a high-emittance black paint. A cold blackbody controlled at a temperature close to the ambient is

used for correction of the background radiation. The sample is clamped in a crucible heated by an electrical resistor. Two shielded type S thermocouples are embedded in the sample in two holes drilled parallel to the surface, for determination of the sample surface temperature.

For the emittance measurement process, radiative signals and temperatures of the reference blackbody, the cold blackbody and the specimen are measured in succession, by rotating the collection optics as shown in Figure 5. The spectral emittance is then calculated using the following formula:

$$\varepsilon(\lambda_c, T_s) = \frac{V_s - V_0}{V_r - V_0} \cdot \frac{L_{\lambda_c}(T_r) - L_{\lambda_c}(T_0)}{L_{\lambda_c}(T_s) - L_{\lambda_c}(T_0)} \quad (4)$$

where λ_c is the central wavelength of the filter, V_s , V_r , V_0 are respectively the radiometric signals measured of the sample, the reference blackbody and the cold blackbody, T_s is the surface temperature of the sample, T_r is the reference blackbody temperature, T_0 is the cold blackbody temperature.

The main assumptions of the model are: the linearity of the spectroradiometer response, the stability of the temperature of the chopper blade, the equality between the cold blackbody radiance and the background radiance in the chamber. A listing of the significant uncertainty components for emittance including those contributing to the temperatures used in the emittance calculation is shown in Table 6.

2.4.3 NIST—The NIST setup of equipment for infrared spectral transmittance and reflectance, including indirect measurement of emittance, is shown in Figure 6. The spectral source is a Digilab FTS-7000^{iv} commercial Fourier transform spectrophotometer (FTS). Measurements are performed in one of three configurations: an internal sample compartment for transmittance, an external Infrared Gonio-Reflectometer Transmissometer (IGRT), for angle dependent measurements of specular samples, and the Infrared Reference Integrating Sphere (IRIS) for near-normal (8° incidence) measurements of both specular and diffuse samples. The IRIS is a custom diffuse-gold coated integrating sphere which employs an absolute measurement method to determine the reflectance from which emittance is calculated using Kirchhof's law and energy conservation, and was used for all measurements up to 150 °C.^{12,13}

The interface optics consisting of the aperture plate, filter wheel, and polarizers are commonly available to the IRIS and IGRT along with beam steering flat mirrors mounted on translation stages, to select between the instruments. An aperture plate is used to select a spot size and vary the flux levels on the sample. For the comparison measurements, a 10 mm diameter spot size was selected. Using a sample heater with an inserted thermocouple, the comparison artefacts are measured at temperatures from 23 °C to 200 °C.

^{iv}Certain commercial equipment, instruments, or materials are identified in this paper to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

A second NIST facility, the Infrared Spectral Emittance System (ISES) is based on the direct measurement of spectral emittance of a material, comparing the spectral radiance of the sample to that of a blackbody at a similar temperature.^{14,15} The ISES facility consists of several major subsystems designed to accomplish these requirements. These are shown schematically in Figure 7. Radiation from blackbody reference sources (to the left) is compared with that from a sample (located opposite the blackbodies) via rotation of a selection mirror. The reference source system consists of water, cesium and sodium heat-pipe blackbody sources, with calibrated platinum resistance thermometers (PRTs) traceable to ITS-90, on a motorized translation stage. Looking down, the source selection mirror can also view an ambient temperature stabilized “cold” vertical blackbody source (not shown). The sample system consists of several interchangeable heater mounts and manipulation stages. As the primary means of sample temperature measurement, an integrating sphere reflectometer with a calibrated room temperature reference sample is used. The low scatter interface optics¹⁶ is not shown except for the rotatable mirrors, for source selection and for detector selection. The emitted radiation is measured with an FTS or filter radiometer, as selected by means of the mirror and a translation stage.

NIST employs the sample temperature measurement method developed by INRIM,¹⁷ in which the sample emittance at the desired temperature is obtained from a hemispherical-directional reflectance factor measurement at the wavelength of a narrow-band filter radiometer, and a subsequent relative radiance comparison of the sample and a reference blackbody.¹⁸ Both Si (905 nm) and InGaAs (1550 nm) radiometers are used to cover the temperature range of 150 °C to 1000 °C. Separate InGaAs and Si pyrometers traceable to the ITS-90 can be used to verify the PRT readings with radiance temperature measurements of the blackbodies.

NIST also employs the same measurement method and calculations for emittance as NMII, using Equations 2 and 3.

Example uncertainty budgets for sample measurements on both of NIST’s systems are shown in Tables 7 and 8. Table 7 shows the emittance uncertainty budget for the directional-hemispherical reflectance measurements using the IRIS sphere with sample heater. Table 8 shows the complete emittance determination for measurements using the ISES system, including the components for the sample temperature measurement process as well as the spectral radiance comparison. Each table shows the budget for one combination of artefact, temperature and wavelength.

2.4.4 INRIM—For room temperature emittance, INRIM employs a blackbody reflectometer to characterise reference standards for normal spectral emittance in the spectral range from 1 μm to 15 μm , shown schematically in Figure 8.¹⁹ As compared with previous reflectometers of the same type, this apparatus takes advantage of a heat-pipe device, which greatly improves the temperature uniformity in the blackbody cavity. The measurement method consists of comparing the radiance of a cooled sample, which is held in a heated blackbody cavity, and the radiance of the cavity itself. The ratio of these radiances yields the sample reflectance. The main sources of uncertainty in the measured reflectance originate from: (i) departures from blackbody conditions due to the presence of the sample, apertures and the

temperature gradients on the cavity wall; and (ii) emission of the sample due to heating which leads to an overestimate of reflectance.

The INRIM system used for sample temperatures above 250 °C is shown in Figures 9 and 10. The hybrid method developed by INRIM and used by NIST (Section 2.3.3), consists of two stages.^{17,20} First, a two step process to determine the sample surface temperature: a) measurement of the heated sample reflectance (1 - emittance) using an integrating sphere (Figure 9), and an appropriate narrow wavelength band, with a filter pyrometer; and b) a subsequent relative radiance measurement at the same wavelength, comparing to a reference blackbody (Figure 10). Second (also Figure 10), the relative spectral radiance measurement is made, using a set of filters or monochromator to obtain the sample spectral emittance.

The primary components of uncertainty for INRIM's two measurement systems are listed in Tables 9 and 10.

2.4.5 PTB—The experimental setup for the measurement of the directional spectral emittance in air consists of a Fourier-transform spectrometer, a sample holder with heater inside a spherical enclosure and a reference blackbody, all temperature-stabilized. The sample and the blackbody are alternately positioned in its detection area with the help of a translation stage (Figure 11). Since the emittance of the sample is less than unity, the sample also reflects part of the radiation emitted by the surroundings towards the spectrometer. An isothermal background at a known temperature and with a constant spectral emittance is provided here by the spherical enclosure around the sample. This enables the ambient radiation incident on the sample and, consequently, the emittance of the sample, to be determined from sample and blackbody relative spectral radiance and temperature measurements:²¹

$$Q = \tilde{L}_s(T_s) / \tilde{L}_{BB}(T_{BB}) \quad (5)$$

and

$$\varepsilon_s = \frac{Q \cdot (B(T_{BB}) - \varepsilon_D B(T_D)) + \varepsilon_D B(T_D) - \varepsilon_E B(T_E)}{B(T_s) - \varepsilon_E B(T_E)}, \quad (6)$$

where s denotes the sample, BB the reference blackbody source, D the detector, and E the environment. The significant sources of uncertainty, which form the uncertainty budget, are listed in Table 11.²² These include instrumentation factors, temperature measurements, and thermal properties affecting the sample and enclosure temperatures.

An example of the major uncertainty components and the resultant combined standard uncertainty is shown in Table 11. In addition the contributions of the sample surface temperature, the blackbody temperature, the detector temperature and the repeatability of the measured signal are shown.

The values of the uncertainty components are given with their absolute values and the respective units. This is because the sample surface temperature is not determined by an analytical expression but rather as a result of several nested iterative calculations, solving the equations describing the heat flow through the sample and the convective loss from the sample surface. Following the *Supplement 1 of the GUM*²³, the uncertainties of the sample surface temperatures were calculated by a Monte Carlo method. Briefly, all quantities that are relevant for the calculation of the sample surface temperature are varied independently in intervals with their respective uncertainties and according to their respective distribution functions. The solution for one specific set of input quantities gives one value for the sample surface temperature. The calculation is performed for a large number of sets of input quantities resulting in a distribution of the sample surface temperature. From that distribution a mean value and a standard deviation can be calculated. The latter serves as the standard uncertainty for the sample surface temperature. More details can be found in Reference 22.

2.5 Processing of the Comparison Data

2.5.1 Description of Differences to be Compensated/Corrected—In this comparison, several factors need to be taken into account and compensated for, in order to properly compare results from all participants. These are: 1) the artefact-to-artefact variation, 2) the artefacts' stability, 3) the differences in spectral resolution, and 4) the differences in the spectral data interval.

2.5.1.1 Artefact-to-Artifact Variation: All artefacts were measured at room temperature (RT) at NIST using the IRIS system prior to their distribution to the other participants. The results are shown in Figure 12(a) to (c). Both the BN and OxIn exhibited considerable variation across the set of 4 artefacts. This is likely due, in part, to separate sources of the raw material used for the 2 mm thick artefacts, and the others artefacts. The SiC artefacts exhibit excellent uniformity. The method used to take into account the artefact variation is to normalize the results for each artefact to the mean value of the room temperature emittance measured by NIST, using a multiplicative correction factor, $f_i(\lambda)$, providing a RT-normalized emittance as shown in Equations 7 and 8:

$$\varepsilon'_i(\lambda, T) = \varepsilon_i(\lambda, T) \cdot f_i(\lambda) \quad , \quad (7)$$

where

$$f_i(\lambda) = \frac{\sum_{i=1}^n \varepsilon_{i,3}(\lambda, RT)}{n \cdot \varepsilon_{i,3}(\lambda, RT)} \quad , \quad (8)$$

and i is the participant number, n is the total number of participants (5), the first element of a subscript pair refers to the participant artefact, and the second element refers to the measurement lab (NIST).

2.5.1.2 Artefact Stability: All artefacts were returned to NIST after the participant's measurements were completed. They were re-measured at room temperature to determine whether any change had occurred. Both the BN and OxIn artefacts exhibited some significant changes. All of the BN changes occur in three spectral bands. Supplemental measurements indicated that the changes were variable upon exposure to air, purge, vacuum and heating. This indicated likely absorption and desorption of moisture from the air. Hence, all results for the affected filters at 2.2 μm , 3.0 μm , 3.6 μm , 6.4 μm , 7.0 μm , and 7.5 μm have been excluded from the comparison.

The only artefacts showing a significant non-reversible spectral change were the OxIn artefacts. The change in the infrared spectral emittance measured at RT for OxIn is shown in Figure 13 for all artefacts. Three participants heated artefacts up to and above 700 °C (LNE, NIST, and INRIM). The stability of the emittance in the NIST artefact was effectively monitored at 1550 nm by means of the artefact temperature measurement process, which includes an integrating sphere reflectance measurement.⁶ An increasing deviation in emittance with temperature at 1550 nm from a linear trend was observed for temperatures above 500 °C, as shown in Figure 14. The time sequence of the measurements follows from the lowest temperature point in the lower left of the plot and continues up to 550 °C, followed by repeats at 350 °C and then 550 °C, with a very small change in emittance, followed by a gradual deviation from a linear increase in emittance with temperature up to the maximum measurement temperature of 800 °C. Finally, a room temperature measurement shows a permanent shift in emittance, upon repeated measurements. A linear fit is made to the lower temperature data (below 500 °C) and used to gauge how much change occurred at which temperature. The corrections, which sum to one, are expressed as percent of the total change measured at RT, and applied as corrections to the data points at 550 °C, 600 °C, 700 °C, and 800 °C.

Similar behaviour was also directly observed by LNE, which performed RT measurements on their artefact between the elevated temperature runs. Therefore their result for the absolute emittance change was used for the LNE sample, NIST before and after measurements were used for NIST and INRIM samples. The split of how much change occurred at which temperature was derived from the time resolved NIST data for all three cases. Corrections to the emittance results were then made to the results at 550 °C and higher by subtraction of a corrective factor g_i , before normalizing for artefact-to-artefact variations using Equation 9.

$$g_i(T) = \begin{cases} h_i(T) \cdot (\varepsilon'_{i,pre}(\lambda, RT) - \varepsilon'_{i,post}(\lambda, RT)) & , i=2, 3, 4 \\ 0, & i=1, 5 \end{cases}, \quad (9)$$

$$\text{where } h_i(T) = \begin{cases} \begin{cases} 0.18, T = 550^\circ\text{C} \\ 0.29, T = 600^\circ\text{C} \\ 0.53, T = 700^\circ\text{C} \end{cases}, i = 2 \\ \begin{cases} 0.07, T = 550^\circ\text{C} \\ 0.11, T = 600^\circ\text{C} \\ 0.20, T = 700^\circ\text{C} \\ 0.62, T = 800^\circ\text{C} \end{cases}, i = 3, 4 \end{cases}$$

The applied correction spectra are shown in Figure 15 for the INRIM, NIST and LNE results. The 45 mm artefact was initially measured by NMIJ up to 100 °C, shortly after receipt from NIST, and then after a long interval, shipped to and measured by PTB up to 250 °C. Subsequently, the 45 mm was returned to NIST and re-measured at RT. There is no evidence within the individual sets of results for NMIJ or PTB of a non-reversible change in emittance, and no change in the NIST RT measurement scale during the comparison. A plausible explanation for the change in the RT measurements for the 45 mm artefact, seen in Figure 13, considering the dates of the initial artefact production and when the NIST, NMIJ and PTB measurements took place, is that the changes are due to ageing of the material. Hence, for the comparison, we have used the NIST “pre” measurement for normalization of the NMIJ results and the NIST “post” measurement for the normalization of the PTB results described in Section 2.5.1.1.

2.5.1.3 Spectral Resolution: Another important factor, resulting from the variety of instrumentation used by the participant NMIs, which needs to be evaluated is the difference in spectral resolution. A summary of the instrument types and spectral resolutions are shown in Table 12. The NMIJ, NIST and PTB spectral instruments are FT-IRs with constant and relatively high resolution. The INRIM spectral instrument is a monochromator for all wavelengths but 1.6 µm, where a filter is used, with comparable to slightly lower resolution at the shorter wavelengths. The LNE spectral instrument employs a set of interference filters with the lowest resolution. Transmittance curves for all LNE filters used for the measurements are shown in Figure 16.

Since all three materials exhibit spectral structure with features on a scale less than the spectral structure of the LNE filter widths, but greater than the resolution of the other systems, modifications of the directly measured emittance values have to be made. Our approach is to apply corrections based on the measured transmittance spectra of the LNE filters and the spectral responsivity of the LNE detector used to obtain the comparison values to obtain an equivalent emittance for comparison, $\varepsilon_i''(\lambda_k, T)$, as given by:

$$\varepsilon_i''(\lambda_k, T) = \frac{\sum_{\lambda_{k1}}^{\lambda_{k2}} \left(\varepsilon_i'(\lambda, T) - g_i(\lambda, T) \right) \cdot \tau_k(\lambda) \cdot \mathfrak{R}(\lambda) \cdot B_\lambda(\lambda, T) \cdot \Delta\lambda}{\sum_{\lambda_{k1}}^{\lambda_{k2}} \tau_k(\lambda) \cdot \mathfrak{R}(\lambda) \cdot B_\lambda(\lambda, T) \cdot \Delta\lambda}, \quad (10)$$

where k is the filter number, λ_{k1} and λ_{k2} are the wavelength limits of the region of transmittance of the filters, $g_i(T)$ is the correction factor from Equation 9, only for OxIn; otherwise it is equal to 0, $\tau_k(\lambda)$ is the spectral transmittance of filter k , and, $\mathfrak{R}(\lambda)$ is the detector relative spectral responsivity.

Several of the filter spectra contain a minor amount of out-of-band transmittance. This has been included in the calculation. For a number of results at both the upper and lower limits of the spectral ranges, the participant's spectral coverage does not completely overlap the filter transmittance range. For the cases where the spectral overlap is at least 50 % of the filter transmittance range, Equation 10 is used, with either λ_{k1} or λ_{k2} replaced with the participant's spectral limit for the measurement. For cases with less than 50 % overlap, the data is excluded from the comparison.

For the processing of the LNE data, the same averaging process is applied to the correction factor, $f_2(\lambda)$, from Section 2.5.1.1, to obtain a factor, $f'_2(\lambda)$, which can be used to appropriately correct the LNE data to compare it to mean values:

$$f'_2(\lambda_k, T) = \frac{\sum_{\lambda_{k1}}^{\lambda_{k2}} f_2(\lambda, T) \cdot \tau_k(\lambda) \cdot \mathfrak{R}(\lambda) \cdot B_\lambda(\lambda, T) \cdot \Delta\lambda}{\sum_{\lambda_{k1}}^{\lambda_{k2}} \tau_k(\lambda) \cdot \mathfrak{R}(\lambda) \cdot B_\lambda(\lambda, T) \cdot \Delta\lambda} \quad (11)$$

2.5.1.4 Spectral Data Interval: For the FT-IR based systems, the spectral data intervals are half the spectral resolution. For these systems, the data intervals are sufficient to carry out the averaging process described in Section 2.5.1.3. However, for the monochromator system of INRIM, this is not the case. The INRIM data interval is approximately 0.5 μm over most of the wavelength range, which is insufficient to perform the calculation of Equation 10. The wavelengths at which the INRIM data is provided correspond closely to the nominal values of the LNE filters. And since the INRIM spectral resolution is comparable to that of the NIST data, we can directly compare the INRIM and NIST data. In order to obtain appropriate filter averaged values for INRIM, we perform a two step process: 1) we obtain a ratio of the INRIM emittance value to the NIST value at INRIM's wavelengths, and then 2) multiply the NIST filter averaged result by that ratio, as given by:

$$\varepsilon''_4(\lambda_k, T) = \varepsilon''_3(\lambda_k, T) \cdot \frac{\varepsilon'_4(\lambda_{4,j}, T) - g_4(T)}{\varepsilon'_3(\lambda_{4,j}, T) - g_3(T)} \quad (12)$$

where $\lambda_{4,j}$ are the wavelengths at which INRIM measured emittance, and λ_k are the nominal LNE filter wavelengths.

2.5.2 Comparison Uncertainty Components—Each of the factors and the associated data processing steps described in the subsections of Section 2.5.1 result in contributions to the comparison uncertainty. The normalization process at room temperature, as well as the

application of it to results at all temperatures, since we have to assume that the correction factor is independent of temperature, contribute uncertainty.

The normalization uncertainty component for the extrapolation increases with temperature. The only data available to quantify the uncertainty is the change in the spread of the participants' measured results with temperature. Data for OxIn is normalized by NIST room temperature data at the lowest temperature of each set (NMI) and shifted to the same starting point, e.g. at 100 °C. Data from each wavelength of all the participants start at the same point. The relative changes with temperature for all filter wavelengths, using this process, are shown in Figure 17. The uncertainty component associated with the extrapolation of the room temperature normalization to the data at all temperatures, is taken to be $0.5 \times$ the standard deviation of the spread for each individual wavelength as described.

An uncertainty contribution is added for the artifact stability corrections for OxIn. The uncertainties associated with the adjustment of the data for the LNE filter transmission and the detector response curve are included. And finally the effect of spectral resolution differences were evaluated but resulted in negligible contribution to the comparison uncertainty. The comparison uncertainty was calculated using a minimum-maximum approach. All data is used with and without added uncertainties. Then the equivalent emittance for the comparison is calculated twice, with and without added uncertainties, and the difference of the two is used as to obtain the comparison uncertainty values.

2.5.3 Data Processing Procedure—A summary of the processing steps to obtain spectral emittance values for each participant that can be compared, as detailed in Section 2.5.1, is given as follows:

1. Calculate the mean of NIST-measured RT spectra,
2. Calculate ratios to the mean to generate correction spectra; calculate correction values for LNE using filter transmittance data, detector responsivity data and the Planck function.
3. Interpolate the lower resolution NMIJ and PTB data sets to match the wavelengths of NIST spectra.
4. Calculate correction for permanent sample emittance change at elevated temperatures for OxIn artefacts.
5. Apply individual correction spectra to data at all temperatures.
6. Calculate data points to compare to the LNE filter measurements: apply filter transmittance, detector response, and Planck function to NIST, NMIJ & PTB data.
7. Ratio the INRIM spectral data points (0.5 μm interval) to the NIST spectra; multiply NIST filter corrected values by the ratio.

These steps result in corrected emittance values and uncertainties for each participant. The overall process is illustrated graphically in Section 3.1.

2.5.4 Comparison Reference Value and Degrees of Equivalence—After preparation of the data for each participant, the results are compared. We employ the methodology recommended by the Consultative Committee on Photometry and Radiometry (CCT) Working Group on Key Comparisons (WG KG) ^{24,25} using weighted mean averaging with cut-off to obtain a nominal comparison reference value (CRV) $\varepsilon_{\text{CRV}}(\lambda_k, T)$, for each filter wavelength, material and artefact temperature, is given by Equation 13. In the weighted mean calculation, each participant's values are weighted by their quoted standard uncertainties up to a cut-off point, which is given by the average of the standard uncertainties below the median of all the standard uncertainties, given by Equation 14.

$$\varepsilon_{\text{CRV}} = \frac{\sum_i \varepsilon_i'' \cdot u_{\text{adj},i}^{-2}}{\sum_i u_{\text{adj},i}^{-2}}, \quad (13)$$

where

$$\begin{aligned} u_{\text{adj},i}(\varepsilon_i'') &= u(\varepsilon_i'') \quad \text{for } u(\varepsilon_i'') \geq u_{\text{cut-off}} \\ u_{\text{adj},i}(\varepsilon_i'') &= u_{\text{cut-off}} \quad \text{for } u(\varepsilon_i'') < u_{\text{cut-off}} \\ u_{\text{cut-off}} &= \text{average} \{u(\varepsilon_i'')\} \quad \text{for } u(\varepsilon_i'') \leq \text{median} \{u(\varepsilon_i'')\} \end{aligned} \quad (14)$$

The uncertainty for the CRV is given by:

$$u(\varepsilon_{\text{CRV}}) = \frac{\sqrt{\sum_i \frac{u^2(\varepsilon_i'')}{u_{\text{adj},i}^4(\varepsilon_i'')}}}{\sum_i u_{\text{adj},i}^{-2}(\varepsilon_i'')} \quad (15)$$

The individual participant's results are then compared to the ε_{CRV} values. The relative deviations from the ε_{CRV} , $D_i(\lambda_k, T)$, and their associated uncertainties, $U_i(\lambda_k, T)$ are the unilateral Degrees of Equivalence (DoE), which are calculated according to Equations (16) and (17):

$$D_i = \frac{\varepsilon_i'' - \varepsilon_{\text{CRV}}}{\varepsilon_{\text{CRV}}}, \quad (16)$$

$$U_i = \varepsilon_{\text{CRV}}^{-1} \cdot k (=2) \cdot \sqrt{u^2(\varepsilon_i'') + u^2(\varepsilon_{\text{CRV}}) + u_{\text{add}}^2 - 2 \cdot \frac{\frac{u^2(\varepsilon_i'')}{u_{\text{adj},i}^2(\varepsilon_i'')}}{\sum_j u_{\text{adj},j}^{-2}(\varepsilon_j'')}} \quad (17)$$

where the additional uncertainty component (u_{add}) is used to account for the normalization process, the correction applied for the temperature drift of the OxIn samples, the application of the LNE filter spectral transmittance and detector spectral response curves, and the differences in spectral resolution.

The error function, defined as

$$E_i(\lambda, T) = \frac{U_i(\lambda, T)}{|D_i(\lambda, T)|} \quad (18)$$

is used to examine the results for outliers. If $E_i \geq 3$, the result is deemed an outlier, and the corresponding data is excluded and the DoE are recalculated.

3 RESULTS

In the text and tables that follow, we identify the participants by number, and in the figures by the colors and symbols as shown in Table 13. The complete results are shown in Figures 20 to 65, for BN, OxIn, and SiC, and for all temperatures measured. The purple and blue symbols for NIST represent reflectometer and radiance measurement systems, respectively.

In Figure 18, the process previously summarized in Section 2.5.3 is illustrated with the example of the results for OxIn at 250 °C. Figure 18(a) shows the original spectral emittance data as submitted by the participant NMIs. The dispersion of the data is similar to the NIST room temperature results shown in Figure 13(a). After applying steps 1 through 5, the normalized corrected data in Figure 18(b) are obtained. The final steps 6 and 7 result in the emittance values shown in Figure 18(c), which are used for the comparison analysis.

3.1 Boron Nitride

An overview of the temperature-dependent spectral emittance of the NIST BN artefact is shown in Figure 19. Curves of emittance for each temperature from 23 °C to 500 °C are shown in (a), and curves for the change in emittance from that at room temperature (RT) 23 °C are shown in (b). A comparison of RT spectra taken before and after heating shows changes in the regions of 1.5 µm to 2.5 µm, from 2.7 µm to 3.2 µm and from 6.5 µm to 7.5 µm. Subsequent examination of measurements after varying amounts of exposure to ambient air, as compared to “clean air” (air, with CO₂ and H₂O removed) revealed that the artefact was retaining varying amounts of OH causing considerable variability in the spectrum in the regions noted above. Hence, we decided to exclude data at 2.2 µm, 3 µm, 3.6 µm, 6.4 µm, 7 µm and 7.5 µm due to the instability.

The unilateral relative DoE: the relative deviations and their expanded uncertainties (as error bars) for the BN measurements are shown in Figure 20 through Figure 30. The results are also tabulated in Appendix A.

3.2 Oxidized Inconel

An overview of the temperature dependent spectral emittance of the NIST OxIn artefact is shown in Figure 31. Curves of emittance for each temperature from 23 °C to 800 °C are shown in (a), and curves for the change in emittance from that at 23 °C (RT) are shown in (b).

The unilateral relative DoE: the relative deviations and their expanded uncertainties (as error bars) for the OxIn measurements are shown in Figure 32 to Figure 46. The results are also tabulated in Appendix A.

3.3 Silicon Carbide

An overview of the temperature dependent spectral emittance of the NIST SiC artefact is shown in Figure 47. Curves of emittance for each temperature from 23 °C to 800 °C are shown in (a), and curves for the change in emittance from that at 23 °C are shown in (b).

The unilateral relative DoE: the relative deviations and their expanded uncertainties (as error bars) for the SiC measurements are shown in Figure 48 to Figure 62. The results are also tabulated in Appendix A.

3.4 Error Function Results

To analyse the DoE data shown in the plots and tables, we use the Error function given by Equation 18, which is a ratio of the expanded uncertainty of the relative deviations, $U(D_i)$ relative to the absolute value of the relative deviations D_i . For the case in which the relative deviation is equal to the uncertainty, $E = 1$. All of the results have been combined into 3 summary plots shown in Figure 63 to Figure 65 for BN, OxIn, and SiC, respectively.

We see that the maximum $E_i(\lambda, T)$ is slightly less than 1.0 for BN, 1.5 for OxIn, and slightly less than 2.0 for SiC. Since we only consider a result to be outliers for $E_i \geq 3.0$, none of the results are in this category.

The numbers of instances of the Error function, $E_i > 1$, for the entire set of results, are sorted by material and NMI in Table 14. The rate for the entire data set of 2041 points is 1.9 %. The overall rate for each NMI is less than 5 %, which indicates good overall agreement among the NMIs. Only for SiC does NMI #2 have a higher rate of 10.9 %. Several factors distinguish SiC from the other two materials and may account for the higher discrepancy rate. The SiC artefacts are polished and specular, which may interact differently to the background, as compared to the more diffuse artefacts of BN and OxIn. Secondly, the emittance nearly varies over the complete range of 0 to 1 in a short spectral range, from 10 μm to 14 μm . Hence errors in the spectral transmittance of the filters and detector responsivity will lead to offsets between NMI #2 and the rest.

4 SUMMARY AND CONCLUSIONS

This CCT supplementary comparison investigated the agreement and the variability in infrared spectral emittance measurements performed by five NMIs. Artefacts of three different candidate standard materials, boron nitride, oxidized Inconel, and silicon carbide,

were produced and measured by all laboratories. Due to the incompatible sample size requirements of their measurement instrumentation, sets of different sized specimens of the three materials were produced and measured. The pilot laboratory performed emittance measurements of all the artefacts, but only at room temperature (23 °C).

Spectral emittance measurements were performed over varying wavelength and temperature ranges by the NMIs, extending from 23 °C to 800 °C. Different portions of these ranges include results from two, three and four NMIs, respectively, and over which the comparisons were made. Several factors, including artefact-to-artefact variability, artefact stability, differing spectral resolutions, and differing data intervals were encountered and accommodated in the data processing. In planning any future comparisons, these factors warrant a high degree of consideration. It may be necessary to limit the comparison to smaller temperature and spectral ranges and/or to those participants with similar instrumental parameters such as spectral resolution and sample size accommodation. In any case, preliminary studies of the selected artefact materials are a crucial element, which can lessen the chance of unforeseen problems during the intercomparison process.

Analysis of the results indicated good agreement between the NMIs considering the relative deviations and their uncertainties. Although some of the subsets of the results contained data with variation greater than expected for $k = 2$ expanded uncertainties (i.e. rate of 5 % outside), the deviations were not sufficient to be considered outliers.

The results of this comparison form a foundation for the establishment of CMCs (Calibration and Measurement Capabilities) at the participant NMIs, as well as a basis for future Key and Supplementary Comparisons of infrared emittance.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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Figure 1. Photos of a) boron nitride, b) oxidized Inconel, and c) silicon carbide artefact sets produced for the comparison. Artefacts for NPL (1 cm dia. $\times$ 1 cm thick) are also shown but were not used.

Figure 2.
The NMIJ spectral emittance measurement system.

Figure 3.
The LNE spectral reflectance measurement system.

Figure 4.
The LNE spectral emittance measurement system, side view.

Figure 5.
The LNE spectral emittance measurement system, top view.

Figure 6. Schematic of the NIST system used for the artefact reflectance measurements from 23 °C to 200 °C.

Figure 7.
Schematic of the NIST Infrared Spectral Emittance Characterization Facility.

Figure 8.
The INRIM Hohlraum spectral reflectance measurement system.

Figure 9.
The INRIM spectral emittance measurement system.

Figure 10.

Optical layout of the INRIM spectral radiance measurements, where the sources 1 and 2 correspond to the heated sample, and reference blackbody. The filter wheel was replaced by a monochromator for the intercomparison measurements.

Figure 11.
The PTB spectral emittance measurement system.

Figure 12.

Plots of NIST-measured emittance of the a) OxIn, b) BN, and c) SiC sample sets. Curves representing the mean of each set of spectra are also shown in black. For silicon carbide the difference to the mean is also plotted against 2nd Y-axis.

Figure 13.
Change in the infrared spectral emittance measured at RT for OxIn after all elevated temperature measurements were completed.

Figure 14.

Emittance of the NIST OxIn artefact at 1550 nm, as measured with a filter radiometer and integrating sphere, used for calculating corrections of sample change during heating.

Figure 15. Corrections applied to OxIn spectral reflectance data to compensate for artefact change (drift) with exposure to heating: (a) INRIM, (b) NIST, and (c) LNE.

Figure 16.
LNE filter transmittance spectra: a) below 6 μm , and b) above 6 μm .

Figure 17.

Data for OxIn is showing the relative change (to room temperature) in emittance vs. temperature for all filter wavelengths and participants (individual curves). The color labeling corresponds to that designated in Figure 12.

Figure 18. Spectral emittance results at 3 stages of the processing: (a) initial data; (b) after step 5; and (c) at the end of the last step.

Figure 19.

NIST spectral emittance results for BN: a) spectral emittance, and b) change in emittance from that at room temperature.

Figure 20.
Unilateral DoE of BN results at 23 °C.

Figure 21.
Unilateral DoE of BN results at 50 °C.

Figure 22.
Unilateral DoE of BN results at 100 °C.

Figure 23.
Unilateral DoE of BN results at 150 °C.

Figure 24.
Unilateral DoE of BN results at 200 °C.

Figure 25.
Unilateral DoE of BN results at 250 °C.

Figure 26.
Unilateral DoE of BN results at 300 °C.

Figure 27.
Unilateral DoE of BN results at 350 °C.

Figure 28.
Unilateral DoE of BN results at 400 °C.

Figure 29.
Unilateral DoE of BN results at 450 °C.

Figure 30.
Unilateral DoE of BN results at 500 °C

Figure 31.

NIST spectral emittance results for oxidized Inconel: a) spectral emittance, and b) emittance difference from that at room temperature.

Figure 32.
Unilateral DoE of OxIn results at 23 °C.

Figure 33.
Unilateral DoE of OxIn results at 50 °C.

Figure 34.
Unilateral DoE of OxIn results at 100 °C.

Figure 35.
Unilateral DoE of OxIn results at 150 °C.

Figure 36.
Unilateral DoE of OxIn results at 200 °C.

Figure 37.
Unilateral DoE of OxIn results at 250 °C.

Figure 38.
Unilateral DoE of OxIn results at 300 °C.

Figure 39.
Unilateral DoE of OxIn results at 350 °C.

Figure 40.
Unilateral DoE of OxIn results at 400 °C.

Figure 41.
Unilateral DoE of OxIn results at 450 °C.

Figure 42.
Unilateral DoE of OxIn results at 500 °C.

Figure 43.
Unilateral DoE of OxIn results at 550 °C.

Figure 44.
Unilateral DoE of OxIn results at 600 °C.

Figure 45.
Unilateral DoE of OxIn results at 700 °C.

Figure 46.
Unilateral DoE of OxIn results at 800 °C.

Figure 47.

NIST spectral emittance results for SiC: a) spectral emittance, and b) emittance difference from that at room temperature.

Figure 48.
Unilateral DoE of SiC results at 23 °C.

Figure 49.
Unilateral DoE of SiC results at 50 °C.

Figure 50.
Unilateral DoE of SiC results at 100 °C.

Figure 51.
Unilateral DoE of SiC results at 150 °C.

Figure 52.
Unilateral DoE of SiC results at 200 °C.

Figure 53.
Unilateral DoE of SiC results at 250 °C.

Figure 54.
Unilateral DoE of SiC results at 300 °C.

Figure 55.
Unilateral DoE of SiC results at 350 °C.

Figure 56.
Unilateral DoE of SiC results at 400 °C.

Figure 57.
Unilateral DoE of SiC results at 450 °C.

Figure 58.
Unilateral DoE of SiC results at 500 °C.

Figure 59.
Unilateral DoE of SiC results at 550 °C.

Figure 60.
Unilateral DoE of SiC results at 600 °C.

Figure 61.
Unilateral DoE of SiC results at 700 °C.

Figure 62.
Unilateral DoE of SiC results at 800 °C.

Figure 63.
Summary plot of the Error function results for BN.

Figure 64.
Summary plot of the Error function results for OxIn.

Figure 65.
Summary plot of the Error function results for SiC.

Table 1

List of variables and subscripts

Variable	Description	In Eqn.
e	Normal directional spectral emittance	(1), (3), (4), (6), (7), (8), (13), (15), (16), (17)
λ	Wavelength	global
T	Temperature	global
L_{λ}	Spectral radiance of the artefact in the normal direction	(1), (4)
B	Spectral radiance of the ideal blackbody as given by Planck's Law	(1), (2), (3), (6), (10), (11)
ν	Wavenumber	global
L_{ν}	Spectral radiance	(2), (3)
C	Complex spectrum	(2)
φ_L	Phase	(2)
V	Radiometric signal	(4)
L	Spectral radiance	(5)
f	Correction factor	(7), (8), (11)
n	Number of participants	(8)
RT	Room temperature	(8)
g	Correction factor	(9), (10), (12)
h	Weighting factor	(9)
e'	RT-normalized emittance	(7), (9), (10), (12)
e''	Equivalent emittance for comparison	(10), (12), (13), (14), (15), (16), (17)
τ	Spectral transmittance of filter	(10), (11)
$\mathfrak{R}$	Relative spectral responsivity of the detector	(10), (11)
f'_i	Correction factor for LNE values ($i=2$)	(11)
u	Standard uncertainty	(13), (14), (15), (17)
D	Relative deviations from CRV	(16), (18)
U	Expanded uncertainty for D	(17), (18)
k	Coverage factor for expanded uncertainty	(17)
E	Error function	(18)

Subscript	Description
0	Cold
h	Hot
ambient	Surrounding (room)
s	Sample
r	Reference

Subscript	Description
c	Central
BB	Blackbody
D	Detector
E	Environment
i, j	Participant number
pre	Before measurements
post	After measurements
k	Filter number
λ_{k1}	Wavelength limit 1 of the region of transmittance of the filter
λ_{k2}	Wavelength limit 2 of the region of transmittance of the filter
CRV	Comparison reference value
adj	Adjusted to cut-off value if criteria is met
cut-off	Lower limit to which uncertainty is adjusted if criteria is met
add	Additional uncertainty components

Table 2Overview of NMI Emittance Measurement Capabilities.ⁱⁱⁱ

Identifier Number	NMI	Wavelength (µm)	Temperature Range (°C)	Spectrometer Type	Artefact Size dia. x thick. (mm)
1	NMIJ	5 to 12	−20 to 100	FTIR	45 × 2
2	LNE	1 to 14	23, 100 to 800	Optical Filters	25 × 10
3	NIST	1 to 20	23 to 900	FTIR	18.5 × 5
4	INRIM	1 to 15	23, 250 to 1000	Monochromator	16 × 2
5	PTB	4 to 40	100 to 250	FTIR	45 × 2

ⁱⁱⁱSpectral and temperature ranges are nominal. Exact spectral ranges are dependent on sample temperature and emittance.

Table 3

Description of the Artefact Materials.

Material	Boron Nitride	Oxidized Inconel				Silicon Carbide	
Material Details							
Vendor(s)	Goodfellow Ltd. (UK)	Goodfellow Ltd. (UK)				Trex Enterprises (US)	
		Vereinigte Deutsche Metallwerke AG (Germany)					
Material Type	Hot Pressed BN	Inconel alloy 600				CVD Process	
		Inconel alloy 600					
Material Form (from Vendor)	2 mm sheet ϕ25.4 mm rod	2 mm sheet				N.A. *	
		ϕ75 mm rod					
Material Composition (4 most abundant, wt%)	N.A. *	Ni	Cr	Fe	C	N.A. *	
		73.0	16.5	9.0	0.7		
		Ni	Cr	Fe	Mn		
		75.4	15.5	8.0	<1		
Sample Machining & Polishing	Agate Ltd. (UK)	NPL Engineering Services (UK) (+ sand blasted)				Trex Enterprises (US)	
Artefact Properties							
Density (g/cm ²) (Vendor)	N.A. *	N.A. *				3.18 (±0.04) [†]	
Surface Roughness ^{**} RMA (µm)	0.7 – 1.4 (NPL)	2.4 – 4.4 (NPL)				<0.1 (Trex Ent.)	
Specular/Diffuse Reflectance (NIST)	95% Diffuse at 2 µm to 80% Specular at 14 µm	Diffuse				Specular	
Heat Treatment							
Where	NIST	NPL				NIST	
Temperature (°C)	600	980				600 + 900	
Duration (h)	30	30				10 + 20	
Atmosphere	Air	Air				Air	

* N.A. – not available.

[†] Trex Enterprises^{**} After heat treatment.

Table 4

NMIJ example uncertainty budget for the emittance measurement of the boron nitride sample at 100 °C and 10 μm wavelength, where $\varepsilon = 0.83$. (Relative quantities are shown.)

BN Emittance	Values (%)	
Radiation measurement		
Reference blackbody		
Reference platinum resistance thermometer	0.01 K	0.01
Effective emittance of the high temperature reference	0.9996 (±0.0001) 0.02 K	0.06
Stability of the temperature control		0.02
Effective emittance of the liquid nitrogen cooled reference	0.9994	N.S.
Nonlinearity of the spectrometer response		0.28
Size-of-source effect		N.S.
Temperature of the sample surface		
Reference platinum resistance thermometer	0.01 K	0.01
Stability of the temperature control	0.02 K	0.02
Temperature drop (thickness of 1 mm, thermal conductivity of 30 W m ⁻¹ K ⁻¹)	0.02 K	0.06
Correction of radiance reflected at sample surface		0.31
Noise on the emittance spectra		0.05
Reproducibility (stdev, 3 days)		0.01
Relative combined standard uncertainty (ε/ε)		0.43
Relative expanded uncertainty (ε/ε) ($k=2$)		0.86

N.S.: negligibly small.

Table 5

LNE uncertainty budget components for the oxidized Inconel sample directional spectral emittance. (Relative quantities are shown.)

Emittance Uncertainty Components	Sub components	Type	Value (%)
<i>Room Temperature Reflectometer</i> (Measurement at 5 μm - Emissivity = 0.772)			
	Sample signal noise	A	0.12
	Reference signal noise	A	0.06
	Nonuniformity of the detector	B	0.6
	Nonlinearity of the detector	B	0.1
	Reflected radiation losses	B	0.09
	Atmospheric absorption	B	0.15
	Collecting mirror reflectance	B	0.6
	Varying reflectance of "incidence mirrors"	B	0.44
	Sample position	B	0.15
	Sample orientation (angle)	B	N.S.
	Effective wavelength defined by the filter	B	N.S.
	Polarization of the incident beam	B	N.S.
Combined type A ($k=1$)		A	0.13
Combined type B ($k=1$)		B	1.0
Relative combined standard uncertainty ($k=1$)			1.0
Relative expanded uncertainty ($k=2$)			2.1

N.S.: negligibly small.

Table 6

LNE uncertainty budget components for the oxidized Inconel sample directional spectral emittance. (Relative quantities are shown.)

Emittance Uncertainty Components	Sub components	Type	Value (%)
<i>Direct Radiance Technique</i> (Measurement at 350 °C and 3 μm - Emissivity = 0.813)			
	Reproducibility & repeatability of emissivity results	A	0.12
	Sample surface temperature	B	1.2
	Blackbody radiance temperature	B	0.9
	Mean background temperature	B	0.4
	Nonlinearity of the detector	B	0.1
	Variation of atmospheric absorption	B	N.S.
	Effective wavelength defined by the filter	B	N.S.
	Size-of-Source Effect of measurement system	B	0.1
	Sample orientation (angle)	B	N.S.
	Combined type A ($k=1$)	A	0.12
	Combined type B ($k=1$)	B	1.6
	Relative combined standard uncertainty ($k=1$)		1.6
	Relative expanded uncertainty ($k=2$)		3.2

N.S.: negligibly small.

Table 7

NIST example uncertainty budget for an OxIn artefact, using the IRIS spectral reflectance system at 150 °C and 11 µm wavelength. (Relative quantities are shown.)

Emittance uncertainty components	Type	Value (%)
Inter-reflections	B	0.01
Detector nonlinearity	B	0.02
Atmospheric absorption variation	B	0.01
Beam flip	B	0.01
Inequivalent sample/reference beam alignment	B	0.01
Retro-reflected light lost out sphere entrance port	B	0.10
Spatial variation of sphere throughput	B	0.31
Errors in sphere mapping	B	0.13
Sphere entrance port overfill	B	N.S.
Sphere sample port overfill	B	0.02
Beam geometry, polarization	B	0.01
Phase errors	B	0.01
Sample uniformity	B	0.10
Sample emitted radiance correction	B	0.10
Combined repeatability & reproducibility	A	0.10
Relative combined standard uncertainty		0.39
Relative expanded uncertainty ($k = 2$)		0.78

N.S.: negligibly small.

Table 8

NIST example uncertainty budget for sample spectral emittance of the SiC sample, at 600 °C using the ISES spectral radiance comparison system. (Relative quantities are shown.)

Uncertainty budget of sample spectral emittance		
	Type	Value (%)
<i>Sample temperature calibration</i>		
Repeatability of temperature comparison	A	0.05
Sample reflectance		
Repeatability of reflectance comparison	A	0.03
Sample		
Alignment	B	0.19
Temperature	B	N.S.
Reflectance reference		
Calibration	B	0.09
Alignment	B	0.19
Sphere reflectometer	B	0.20
Radiometer calibration		
Calibration at FP	B	0.01
Interpolation	B	0.01
Alignment	B	N.S.
SSE of interface optics	B	0.04
<i>Spectral radiance comparison using FTIR</i>		
Repeatability of spectral radiance comparison	A	0.30
Calibration of room-temperature reference BB		
Temperature	B	0.01
Spectral emissivity	B	0.03
Calibration of reference BB		
Temperature	B	0.01
Spectral emissivity	B	0.03
Nonlinearity of the FT detector	B	0.03
Relative combined standard uncertainty		0.47
Relative expanded uncertainty ($k = 2$)		0.94

N.S.: negligibly small.

Table 9

INRIM example uncertainty budget for the Blackbody Reflectometer measurement of the oxidized Inconel sample, directional spectral emittance. (Relative quantities are shown.)

Emittance uncertainty components	Sub components	Type	Value (%)
<i>Room Temperature Blackbody Reflectometer</i>			
(measurements at 3 μm)			
	Signal noise	A	0.50
	Signal repeatability	A	0.50
	Monochromator wavelength scale	B	0.05
	Blackbody reflectometer	B	0.20
	Sample position and rotation angle	B	1.00
Relative combined standard uncertainty			1.24
Relative expanded uncertainty ($k = 2$)			2.48

Table 10

INRIM example uncertainty budget for the direct radiance technique measurement of the oxidized Inconel sample directional spectral emittance at 350 °C and 3 μm wavelength. (Relative quantities are shown.)

Emittance uncertainty components	Sub components	Type	Value (%)
<i>Direct Radiance Technique</i>			
Sample Temperature via Integrating Sphere (at 350 °C and 1.6 μm)	Signal noise	A	0.1
	Signal repeatability	A	0.1
	Filter radiometer wavelength	B	0.1
	Sample position and rotation angle	B	0.6
	Reference sample reflectance	B	1.0
<i>Spectral Radiance Measurements (at 350 °C and 3 μm)</i>			
	Signal noise	A	0.70
	Signal repeatability	A	0.50
	Sample temperature (from above)	B	0.65
	Monochromator wavelength scale	B	0.12
	Sample position and rotation angle	B	1.00
	Reflected ambient background rad.	B	N.S.
Relative combined standard uncertainty			1.48
Relative expanded uncertainty ($k = 2$)			2.96

N.S.: negligibly small.

Table 11

The PTB uncertainty budget of the directional spectral emittance measurement of the Boron Nitride sample at 200 °C and 10 µm. (Absolute quantities are shown for the individual components.)

Uncertainty Components	Sub components	Type	Value
<i>Temperature of reference blackbody</i>		A	0.20 K
	Calibration of temperature sensor	A	
	Repeatability of resistance measurement	A	
	Uncertainty of resistance measurement	A	
<i>Temperature of sample enclosure</i>		A	0.20 K
	Calibration of temperature sensor	A	
	Repeatability of resistance measurement	A	
	Uncertainty of resistance measurement	A	
<i>Temperature of detector</i>		A	0.20 K
	Calibration of temperature sensor	A	
	Repeatability of resistance measurement	A	
	Uncertainty of resistance measurement	A	
<i>Emissivity of enclosure</i>		B	0.040
<i>Emissivity of detector</i>		B	0.040
<i>Measured signal of spectrometer</i>			0.00083
	Repeatability	A	0.00083
	Nonlinearity	B	N.S.
<i>Temperature of sample surface</i>			0.91K
	Thermal conductivity of sample substrate	B	4.0 W m ⁻¹ K ^{#x2212;1}
	Thermal conductivity of contact layer	B	0.033 W m ⁻¹ K ⁻¹
	Thickness of heating plate	B	0.50 mm
	Thickness of contact layer	B	0.10 mm
	Thickness of sample substrate	B	0.20 mm
	Temperature of heating plate	A	0.10 K
	Temperature of enclosure	A	0.10 K
	Hemispherical emittance of sample (iteratively determined)	A	0.030
	Diameter of sample holder	B	5.0 mm *
	Diameter of enclosure	B	5.0 mm *
	Distance sample / enclosure	B	5.0 mm *
Relative combined standard uncertainty (%)			0.70
Relative expanded uncertainty (<i>k</i> = 2) (%)			1.40

* Uncertainties of effective lengths in convection model

Table 12

Instruments and their spectral resolution.

Participant		Instrument Type	Spectral Resolution (cm^{-1})
1	NMIJ	FT-IR	4
2	LNE	Filter	600 to 100 (from 1.5 μm to 14 μm)
3	NIST	FT-IR	8
4	INRIM	Monochromator Filter	45 to 5 (from 2 μm to 12 μm) 300 nm at 1.6 μm
5	PTB	FT-IR	16

Table 13

Identification of data in results plots and tables.

NMI	Number	Symbol
NMIJ	1
LNE	2
NIST	3
INRIM	4
PTB	5

Table 14

Number of instances of data points with the Error function greater than 1.

Material	BN		OxIn		SiC			Totals	
	Total #	$E_i > 1$	Rate, %	Total #	$E_i > 1$	Rate, %	$E_i > 1.5$	Rate, %	Rate %
1	19	0	0	23	0	0	1	4.4	0
2	149	0	0	242	4	1.7	27	10.9	4.9
3	162	0	0	274	0	0	1	0.3	0.1
4	73	0	0	168	3	1.8	2	1.1	1.2
5	59	0	0	72	0	0	0	0.0	0
Totals	462	0	0	779	7	0.9	31	3.9	1.9