CMC review protocol for infrared spectral emissivity measurements

Part 1: normal (near-normal) emissivity (emittance)
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Scope
To provide a method of reviewing CMCs in the sub-field of thermal diffusivity for acceptance in Appendix C of the KCDB. The protocol partly covers service category number 6.1.2 “Thermal diffusivity” [m² s⁻¹] of the “CLASSIFICATION OF SERVICES IN THERMOMETRY” in the KCDB.

This protocol is applied to thermal diffusivity measurement in the thickness direction of a solid homogeneous isotropic sample with mm order thickness by the flash method (see Notes for details of the flash method).

The range of thermal diffusivity for this protocol is valid from 5.0 × 10⁻⁷ m² s⁻¹ to 2.0 ×10⁻⁴ m² s⁻¹.

Review criteria
I. KCDB registered international comparisons are available, such as an RMO supplementary comparison,
   I.1 Traceability of the normal emissivity measurement is established
      I.1.1 by oneself as a derived quantity
      or
      I.1.2 through a reference sample or reference samples supplied by the NMI or the NMIs which have a CMC for the relevant range of wavelength, temperature and emissivity, and the following condition is satisfied.

\[ U_{C_{MC}}(k=2) \geq U_{R_S}(k=2) \geq U_{R-C_{MC}}(k=2) \]
Here, $U_{CMC}(k=2)$ is the CMC in submission now. $U_{RS}(k=2)$ is the uncertainty of normal emissivity of the reference sample. $U_{R-CMC}(k=2)$ is the CMC of the NMI who determined normal emissivity of the reference sample.

I.2 The CMC is approved without scrutiny if the KCDB registered international comparison result satisfies the following conditions I.2.1, I.2.2 and I.2.3.

1.2.1

$$U_{CMC}(k = 2) \geq U_{NMI}(k = 2)$$

and

1.2.2

$$|E_n| = \left| \frac{x - X}{\sqrt{U^2_{CMC}(k=2) + U^2_{ref}(k=2) + U^2_{Comparison}(k=2)}} \right| \leq 1.0$$

for greater than or equal to 95 % of all the data values within the spectral and temperature ranges of the comparison,

and

1.2.3

$$U_{CMC}(k = 2) > \sqrt{U^2_{CMC}(k = 2) + U^2_{ref}(k = 2) + U^2_{Comparison}(k = 2)}/\sqrt{10}$$

for greater than or equal to 95 % of all the data values within the spectral and temperature ranges of the comparison.

Here, $U_{CMC}(k=2)$ is the CMC in submission and $U_{NMI}(k=2)$ is the expanded uncertainty for the KCDB registered international comparison reported by the NMI who is submitting $U_{CMC}(k=2)$. $x$ is the reported value from the NMI. $X$ is the reference value of the comparison. $U_{ref}(k=2)$ is the expanded uncertainty of $X$. $U_{Comparison}(k=2)$ is the expanded uncertainty of the international comparison.

I.3 When conditions I.2.1 and I.2.2 are satisfied but I.2.3 is not satisfied at some or all of the ranges in the comparison result, the inter-RMO scrutiny for these ranges based on technical evidence given in I.3.1 is required for the CMC approval.
I.3.1 Scrutiny items

• Report of the KCDB registered international comparisons
• Uncertainty budget and details of uncertainty evaluations
  • Uncertainty budget of the KCDB registered comparison
  • Uncertainty budget of \( U_{\text{CMC}} \)
• Typical emissivity spectra for the temperature and spectral range investigated, results of analysis and calculations to obtain emissivity as evidence of UCMC
• Other supporting technical documents as evidence, for example, peer review reports, published papers, and reports of the other international comparisons

II. When a KCDB registered international comparisons is not available, but an international comparison between NMIs is available,

II.1 It is required for the CMC approval that the international comparison result satisfies the following II.1.1, II.1.2, II.13 and II.1.4 or II.1.5.

II.1.1 \[ U_{\text{CMC}}(k = 2) \geq U_{\text{NMI}}(k = 2) \]

Here, \( U_{\text{CMC}}(k=2) \) is the CMC in submission. \( U_{\text{NMI}} (k=2) \) is the expanded uncertainty for the international comparison reported by the NMI who is submitting \( U_{\text{CMC}}(k=2) \).

and

II.1.2 Agreement of comparison results;
It is required that the result of the international comparison in the range is in agreement with uncertainty \((k = 2)\) among the participants.

and

II.1.3 Report of the international comparison;
Does the report of the international comparison fulfill the following?
• It is open to the public.
• It is reviewed by an expert in this field who does not participate in the comparison.
For example, papers published in journals and the open access reports in websites are available.
II.1.4  Requirements for uncertainty budget using the direct method (spectral radiance);
It is required that the following factors are included in the uncertainty budget.
The traceability of normal emissivity measurement is established by oneself as a derived quantity;
uncertainty components are:
• Sample surface temperature
• Spectral wavelength or wavenumber
• Radiance temperature of the reference source
or
• Temperature of the reference source
and
• Effective emissivity of the reference source
• Size-of-source effect
• Knowledge of the background temperature reflected by the sample
• Variance of the measurement
• Repeatability of the measurement
• Linearity of the detector
or
II.1.5  Requirements for uncertainty budget using the indirect method (reflectance or absorptance);
It is required that the following factors are included in the uncertainty budget, unless otherwise
demonstrated to be insignificant.
The traceability of normal emissivity measurement is established by oneself as a derived quantity;
uncertainty components are:
• Sample surface temperature
• Spectral wavelength or wavenumber
• Inter-reflections in the measurement beam path
• linearity of the detector
• Atmospheric absorption variation
• Inequivalent sample/reference beam alignment
• Retro-reflected light lost out the entrance port
• Directional variation of the throughput
• Entrance port overfill
• Sample overfill
• Sample temperature uniformity
• Sample emitted radiance correction
• Variance of the measurement
• Repeatability of the measurement

II.2 When conditions II.1.1, II.1.2, II.1.3, and II.1.4 (or II.1.5) are satisfied, the inter-RMO review based on technical evidence given in II.2.1 is required for the CMC approval.

II.2.1 Scrutiny items
• Report of the international comparisons
• Uncertainty budget and details of uncertainty evaluations
• Uncertainty budget of the international comparison
• Uncertainty budget of $U_{CMC}$
• Typical emissivity spectra for the temperature and spectral range investigated, results of analysis and calculations to obtain emissivity
• Other supporting technical documents as evidence, such as a peer review report or published papers

Notes
• Directional emissivity and spectral emissivity are defined in references [1] and [2], respectively. The CMC for normal emissivity shall be expressed as a relative expanded uncertainty. It is reasonable because it is a derived quantity of length, time and temperature.

• The term “emittance” is often used interchangeably with “emissivity” to describe the same quantity, as was done in the CCT-S1 report [3].

• The term “normal” in the case of emissivity is often used, as is the case here, to refer to any measurement geometry which is within a reasonably narrow range of direction angles about the normal (0°) to the surface, which is also referred to as “near-normal emissivity” and is typically within ± 10°. In fact, all emissivity measurements will include some range of incidence/viewing angles. Comparisons are feasible between systems that have various near normal geometries due to the fact that the variation in emissivity values about the normal, for many materials, is quite small and not a significant component of uncertainty. In cases where differences in measurement geometry may be significant, corrections for the differences should be made of included as an uncertainty component. In all cases it is important for any CMC to include the associated geometry of the normal emissivity submission.
Figure: The flow chart on the CMC review of spectral normal emissivity measurement
• A number of methods of directional emissivity measurement are available [4, 5]. The primary methods employed are 1) the “direct method”, a radiance comparison of a sample and a blackbody source, and 2) the “indirect method”, measurement of the hemispherical reflectance (and transmittance for non-opaque samples), from which the absorptance is calculated, and which, through Kirchhoff’s Law of Thermal Radiation, is equal to the emissivity. Often, the method selected is based on the relative spectral radiance available from the sample for the spectral range of interest, versus an external source and background radiation. Hence, the indirect method is often used at lower sample temperatures and the direct method for elevated temperatures.

• Indirect methods include both near-normal-hemispherical reflectance and hemispherical-near-normal reflectance. Devices used for these include both integrating spheres and hemispherical mirrors.

• Spectral selection can be accomplished in several ways. Common spectrally selective instrumentation includes 1) a set of narrow band filters, 2) a monochromator, and 3) a Fourier transform interferometer.

• Extensive descriptions of several measurement systems including employment of both primary measurement methods, and all three spectrometer types, as well as the corresponding example uncertainty budgets can be found in the KCDB registered Supplementary Comparison report CCT-S1 [3]. Comparisons were performed over a spectral range of 2 µm to 14 µm, a temperature range of 23 °C to 800 °C, and an emissivity range of 0.14 to 0.97. The emissivity range values are from average emissivity values over the individual filter bands employed by one of the participants, and on which basis all of the participants’ results were determined for the purposes of the comparison. The emissivity values of the higher spectral resolution data provided by the other participants ranged over 0.04 to 1.00, as can be seen in Figure 47 of the report.

• Due to the wide range of potential, emissivity values, materials, spectral range, temperature range, and measurement method, all of which will affect the associated uncertainties in different ways, it is not appropriate to select a single uncertainty value as a limit for assessing a CMC submission. Hence there is none included in Section II, and assessments should be made based on the other criteria described.
References


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on behalf of the CCT Task Group for Thermophysical Quantities