CMC review protocol for thermal diffusivity measurements

Part 1: solid materials by the flash method

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” [$m^2 \cdot s^{-1}$] of the “CLASSIFICATION OF SERVICES IN THERMOMETRY (July 2016)” 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 Note for details of the flash method.) The range of thermal diffusivity for this protocol is valid from 5.0 x 10^{-7} $m^2 \cdot s^{-1}$ to 2.0 x 10^{-4} $m^2 \cdot s^{-1}$.

This protocol is effective beginning on ** ** 2021 (date unknown).

Review criteria

I. KCDB registered international comparisons are available, such as an RMO supplementary comparison,

I.1 Traceability of the thermal diffusivity 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 CMC for the relevant range of thermal diffusivity,*
and the following condition is satisfied.

$$U_{\mathrm{CMC}}(k=2) \geq U_{\mathrm{RS}}(k=2) \geq U_{\mathrm{R\text{-}CMC}}(k=2)$$

Here, $U_{\mathrm{CMC}}(k=2)$ is the CMC in submission now. $U_{\mathrm{RS}}(k=2)$ is the uncertainty of thermal diffusivity of the reference sample. $U_{\mathrm{R\text{-}CMC}}(k=2)$ is the CMC of the NMI who determined thermal diffusivity of the reference sample.

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

I.2.1 $U_{\mathrm{CMC}}(k=2) \geq U_{\mathrm{NMI}}(k=2)$

and

I.2.2 $$\left|E_n\right| = \left| \frac{x - X}{\sqrt{U_{\mathrm{CMC}}^2(k=2) + U_{\mathrm{ref}}^2(k=2) + U_{\mathrm{Comparison}}^2(k=2)}} \right| \leq 1.0$$

and

I.2.3 $$U_{\mathrm{CMC}}(k=2) > \sqrt{U_{\mathrm{ref}}^2(k=2) + U_{\mathrm{Comparison}}^2(k=2)} \Big/ 3$$

Here, $U_{\mathrm{CMC}}(k=2)$ is the CMC in submission and U_{NMI} ($k=2$) is the relative expanded uncertainty for the KCDB registered international comparison reported by the NMI who is submitting $U_{\mathrm{CMC}}(k=2)$. x is the reported value from the NMI. X is the reference value of the comparison. U_{ref} ($k=2$) is the relative expanded uncertainty of X. $U_{\mathrm{Comparison}}$ ($k=2$) is the relative 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 of the comparison result, the inter-RMO scrutiny for these ranges based on technical evidences given in I.2.1 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_{CMC}
- Typical temperature versus time curve (thermogram) obtained by flash method for each temperature investigated, results of analysis and calculation to obtain thermal diffusivity as evidences of U_{CMC}
- Other supporting technical documents as evidence, for example, Peer Review report, published papers, and reports of the other international comparisons

II. An international comparison between NMIs is available,

II.1 The following condition is satisfied.

$$U_{\mathrm{CMC}}(k=2) \geq 7\%$$

II.2 It is required for the CMC approval that the international comparison result satisfies the following II.2.1, II.2.2, II.2.3 and II.2.4.

II.2.1 $$U_{\mathrm{CMC}}(k=2) \geq U_{\mathrm{NMI}}(k=2)$$

Here, $U_{\mathrm{CMC}}(k=2)$ is the CMC in submission. U_{NMI} ($k=2$) is the relative expanded

uncertainty for the international comparison reported by the NMI who is submitting $U_{CMC}(k=2)$.

and

II.2.2 Agreement of comparison results;

It is required that the result of the international comparison is in agreement in the range with uncertainty among participants.

and

II.2.3 Report of the international comparison;

Does the report of the international comparison fulfill the follows?

- It is open to the public.
- It is reviewed by an expert in this field who does not participate to the comparison.

For example, papers published in journals and the open access reports in Web site are available.

and

II.2.4 Requirements for uncertainty budget;

It is required that the following factors are included in the uncertainty budget.

The traceability of thermal diffusivity measurement is established

by oneself as the derived quantity

- Uncertainty of sample thickness
- Uncertainty of heat diffusion time
- Uncertainty of sample temperature

through the reference samples

- Uncertainty of thermal diffusivity value of the reference sample
- Variance of measurements
- Uncertainty of sample thickness

II.3 When conditions II.2.1, II.2.2 and II.2.3 are satisfied but II.2.4 is not satisfied, the inter-RMO review based on technical evidences given in II.2.1 is required for the CMC approval.

II.3.1 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 temperature versus time curve obtained by the flash method for each temperature investigated and results of analysis and calculation to obtain thermal diffusivity
- Other supporting technical documents as evidence, such as Peer Review report or papers

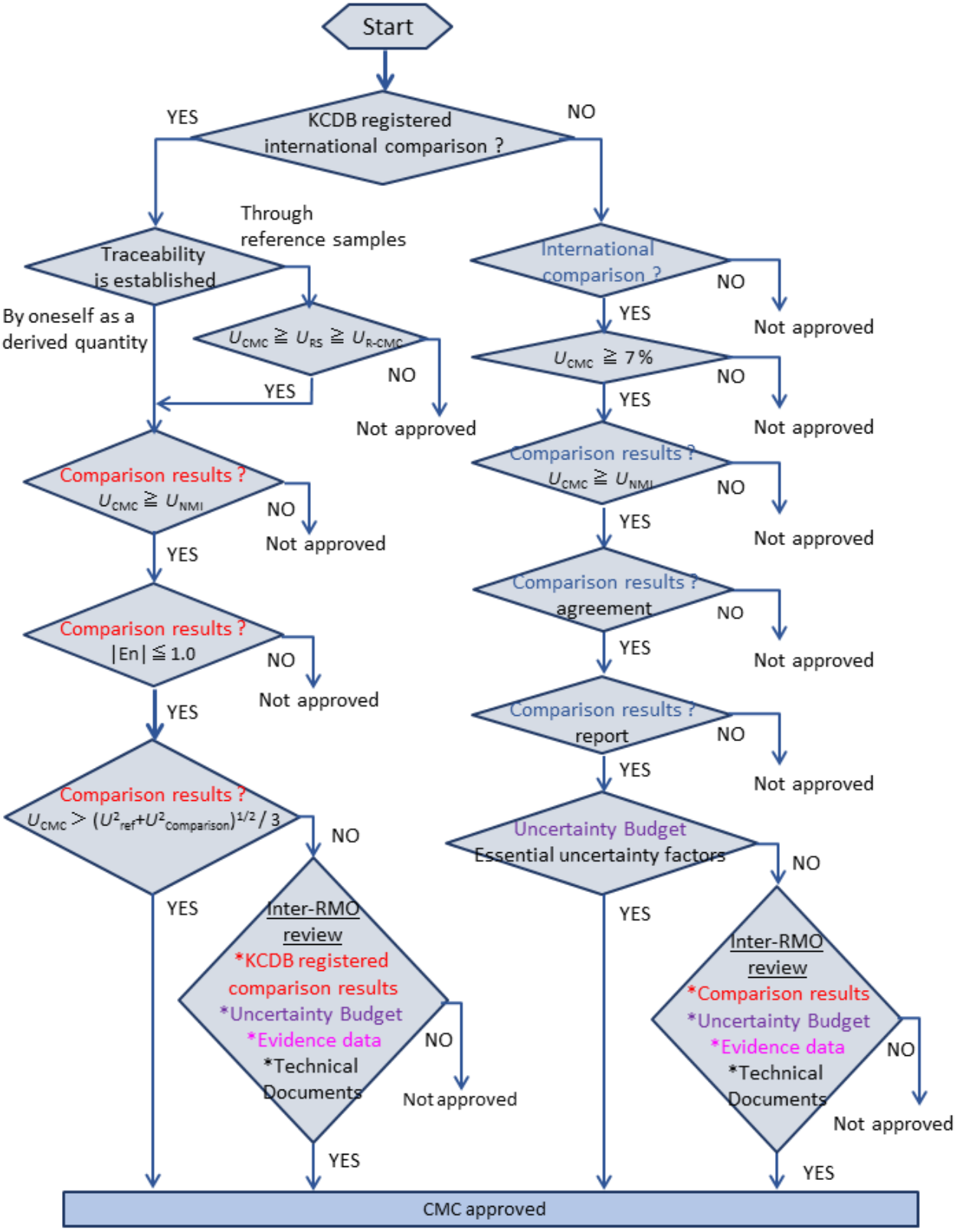

Figure: The flow chart on the CMC review of thermal diffusivity measurement

Notes

- Thermal diffusivity is defined in reference [1]. The CMC for thermal diffusivity measurements shall be expressed as a relative expanded uncertainty.

- The CMC for thermal diffusivity measurements is reviewed according to the rule of CIPM-MRA [2]. A quality system is necessary in addition to this review in order to register the CMC.

- The term “flash method” [3] includes the laser flash method and the xenon flash method.

- The thermal diffusivity in the range from 5.0 x 10^{-7} m^2s^{-1} to 2.0 x 10^{-4} m^2s^{-1} is usually measured by the flash method [4].

- Each value of thermal diffusivity must be associated with the temperature at which the measurement has been performed.

- A procedure to obtain intrinsic thermal diffusivity as physical property is effective [5].

References

[1] ISO 80000-5:2007 Quantities and units -- Part 5: Thermodynamics.
[2] *“Calibration and Measurement Capabilities in the context of the CIPM MRA (CIPM MRA-D-04.pdf)”*.
[3] W. J. Parker, R. J. Jenkins, C. P. Butler, G. L. Abbott: J. Appl. Phys. 32, 1679 (1961).
[4] Y. S. Touloukian, R. W. Powell, C. Y. Ho and M. C. Nicolaou, “Thermophysical Properties of matter – the TPRC data series volume 10 Thermal diffusivity”, PLENUM (1973).
[5] M. Akoshima, B. Hay, M. Neda, M. Grelard, Int. J. Thermophys. 34, 778 (2013)