World Academy of Science, Engineering and Technology
International Journal of Physical and Mathematical Sciences
Vol:14, No:2, 2020

Estimation of Uncertainty of Thermal Conductivity Measurement with Single Laboratory Validation Approach

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***Abstract*—**The thermal conductivity of thermal insulation materials are measured by Heat Flow Meter (HFM) apparatus. The components of uncertainty are complex and difficult on routine measurement by modelling approach. In this study, uncertainty of thermal conductivity measurement was estimated by single laboratory validation approach. The within-laboratory reproducibility was 1.1%. The standard uncertainty of method and laboratory bias by using SRM1453 expanded polystyrene board was dominant at 1.4%. However, it was assessed that there was no significant bias. For sample measurement, the sources of uncertainty were repeatability, density of sample and thermal conductivity resolution of HFM. From this approach to sample measurements, the combined uncertainty was calculated. In summary, the thermal conductivity of sample, polystyrene foam, was reported as 0.03367 W/m·K ± 3.5% (k = 2) at mean temperature 23.5 °C. The single laboratory validation approach is simple key of routine testing laboratory for estimation uncertainty of thermal conductivity measurement by using HFM, according to ISO/IEC 17025-2017 requirements. These are meaningful for laboratory competent improvement, quality control on products, and conformity assessment.

***Keywords*—**Single laboratory validation approach, within-laboratory reproducibility, method and laboratory bias, certified reference material.

I. Introduction

The thermal conductivity measurement is convenient technique by HFM apparatus. In general, the objectives of measurement result of thermal insulation are used for quality control, import and export, register for standard, research development, etc. The test results without measurement uncertainties are not fulfilling of some purposes because measurement results cannot be compared. To achieve these purposes, the measurement uncertainty could be estimated. When the need of testing laboratories are to be the ISO/IEC 17025-2017 accredited laboratories, the measurement uncertainty and traceability are required [1]. In the past several years, the uncertainty estimation by modelling approach [2] was recommended. However, it was quite complex and difficult on routine test measurement. Other approaches that fit for intended use are focused. By HFM measurement technique, the Certified Reference Material (CRM) is always used for accurate results. Because the certified value with associated uncertainty is provided by using metrological valid procedure [3], it is a simple way to establish metrology traceability of thermal conductivity measurement in accordance with ISO/IEC 17025-2017 requirements. In this study, the single laboratory validation approach [4] was used. The uncertainties of this approach [5] derived from two investigations. The precision investigation, the within-laboratory reproducibility, was defined by quality control material. The measured values were collected in normal operation for a period time. The use of SRM 1453 expanded polystyrene board [6] for bias assessment was essential activity in validation of measurement procedure. It was performed under appropriate within-laboratory reproducibility conditions which correspond to normal operation. The sources of error involved in measurement process were evaluated for uncertainties estimation of the method and laboratory bias. Hence, the uncertainty of the within-laboratory reproducibility and the method and laboratory bias can represent the accuracy of measurement process. The uncertainties of sample including the uncertainty from single laboratory validation were performed. Finally, the test report of thermal insulation showed thermal conductivity value with its uncertainty.

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II. Methodology

A. Investigate of Precision

Polystyrene specimen was used as a quality control sample in this study. It was conditioned in temperature and humidity chamber at 22 ºC and 50% relative humidity (RH) for 24 h before each measurement [7]. To measure the specimen, mean temperature at 24 ºC and temperature difference at 20 ºC were set on HFM. The thermal conductivity values were collected for 30 measurements. The mean value, upper/lower action limit and upper/lower warning limit were evaluated and plotted the control chart for further quality control in laboratory [8]. The standard uncertainty of within-laboratory reproducibility (u_{rw}) [5], standard deviation of this specimen was calculated as:

$$s_{meas} = \sqrt{\sum_{i=1}^{n} (x_i - \bar{x})^2/(n-1)} \quad (1)$$

where $\bar{x} = \sum_{i=1}^{n} x_i/n$; x_i is the measurement value at $i = 1\ to\ n$; n is the number of measurements; s_{meas} is the standard deviation of measurement

B. Assessment of Method and Laboratory Bias [5]

The CRM for bias checking, SRM 1453 expanded polystyrene specimen was conditioned in temperature and humidity chamber at 22 ºC and 50% R.H. for 24 h before measurement [7]. The dimension and mass of this specimen were measured by using steel ruler, digital caliper and balance. The density of specimen was basically calculated from its width, length, thickness and mass [9]. To measure thermal conductivity of the specimen, mean temperature at 24 ºC and temperature difference at 20 ºC were set on HFM. The thermal conductivity values were collected for 6 measurements.

1. The bias, the difference of mean value of measurement and certified value, was calculated as:

$$Bias = \bar{x}_{meas} - x_{crm} \quad (2)$$

where $\bar{x}_{meas}$ is the mean value of measurement; x_{crm} is the certified value of CRM. However, the certified value of SRM 1453 was used in this study calculated from equation assigned in certificate [6] as:

$$x_{crm} = 0.000111 - 0.0000424\rho + 0.000115T \quad (3)$$

where ρ is the bulk density (kg ·m^3); T is the mean specimen temperature (K)

2. The bias was assessed as:

$$|\bar{x}_{meas} - x_{crm}| > 2\sqrt{(s_{meas}^2/n) + u_{crm}^2} \quad (4)$$

where s_{meas} is the standard deviation of CRM measurement; n is the number of measurements; u_{crm} is the standard uncertainty of CRM.

If the magnitude of the mean value of measurement ($\bar{x}_{meas}$) from the certified value (x_{crm}) was larger than twice the standard uncertainty of this deviation (4), the deviation was considered to be significant bias. The observed bias was proceeded the correction [5]. Otherwise the deviation was insignificant. Insignificant bias was not corrected but accounted for in the uncertainty. However, u_{crm} is composed of the standard uncertainty from certificate, density, temperature, thickness of measurement and resolution as:

$$u_{crm} = \sqrt{u_{cer}^2 + u_{d(crm)}^2 + u_t^2 + u_{th}^2 + u_r^2} \quad (5)$$

- The u_{cer} is the standard uncertainty from certificate of CRM [2] calculated from its uncertainty value divided by coverage factor (k). In this certificate [6], the k value is equal 2.
- The $u_{d(crm)}$ is standard uncertainty of density of CRM calculated by (6):

$$u_d = \sqrt{u_w^2 + u_l^2 + u_h^2 + u_m^2} \quad (6)$$

where u_w is the standard uncertainty of width specimen; u_l is the standard uncertainty of length specimen; u_h is the standard uncertainty of thickness specimen; u_m = the standard uncertainty of mass specimen. These standard uncertainties derived from repeatability, resolution, calibration certificate of each measuring equipment.

- The standard uncertainty of temperature of HFM (u_t) in (5) was determined to check the temperature of plates by temperature recorder with type K thermocouple. The u_t was calculated as:

$$u_t = \sqrt{u_{at}^2 + u_{cert}^2 + u_{rt}^2 + u_{ut}^2} \quad (7)$$

where u_{at} is the standard uncertainty of repeatability; u_{cert} is the standard uncertainty of temperature device certificate; u_{rt} is the standard uncertainty of resolution of temperature reading; u_{dt} is the standard uncertainty of temperature uniformity of the plate

- The standard uncertainty of thickness (u_{th}) in (8) was evaluated for the plate separation measurement of HFM by using the 25 mm and 75 mm of the jigs. The thickness measurements of each jig were recorded on HFM for 5 measurements. The u_{th} was calculated as:

$$u_{th} = \sqrt{u_{ath}^2 + u_{certh}^2 + u_{rth}^2} \quad (8)$$

where u_{ath} is the standard uncertainty of repeatability; u_{certh} is the standard uncertainty of the jig's certificate; u_{rth} is the standard uncertainty of resolution of thickness reading on HFM

- The u_r is the standard uncertainty of thermal conductivity resolution of HFM. It was calculated as:

$$u_r = resolution/2\sqrt{3} \quad (9)$$

3. The standard uncertainty of the method and laboratory bias (u_{bias}) was calculated as:

$$u_{bias} = \sqrt{bias^2 + \left(\frac{s_{meas}}{\sqrt{n}}\right)^2 + u_{crm}^2} \quad (10)$$

C. Procedure for Sample Measurement

The sample specimen, polystyrene foam, was conditioned in temperature and humidity chamber at 22 ºC and 50% R.H. for 24 h before measurement [7]. The dimension and mass of sample were measured. The density of sample was determined [9]. The standard uncertainty of density was calculated as (6). To measure thermal conductivity of the sample, mean temperature at 24 ºC and temperature difference at 20 ºC were set on HFM. The replicate measurements were made and the mean value of thermal conductivity was reported. The standard uncertainties of measurement [2], [5] were calculated as:

1. Standard uncertainty from repeatability of sample (u_a)

$$u_a = s_{meas}/\sqrt{n} \quad (11)$$

2. Standard uncertainty of thermal conductivity resolution of HFM ($u_{r(x)}$) of sample reading was calculated as (9):
3. Standard uncertainty of density of sample, $u_{d(x)}$

The standard uncertainty of width (u_w), length (u_l) and thickness (u_h) and mass (u_m) derived from repeatability, resolution, calibration certificate of measuring these equipment. The standard uncertainty of density of sample ($u_{d(x)}$) was calculated as (6).

4. The combined uncertainty (u_c) was determined from uncertainty of within-laboratory reproducibility (u_{rw}), uncertainty of the method and laboratory bias (u_{bias}), uncertainty of thermal conductivity resolution of HFM ($u_{r(x)}$), uncertainty of density $u_{d(x)}$ and repeatability of sample (u_a) as:

$$u_c = \sqrt{u_{rw}^2 + u_{bias}^2 + u_{r(x)}^2 + u_{d(x)}^2 + u_a^2} \qquad (12)$$

5. The measurement result with expanded uncertainty (U) was calculated as [4]:

$$U = 2 \times u_c \qquad (13)$$

The estimation of uncertainty of thermal conductivity measurement with single laboratory validation approach is displayed in Fig. 1.

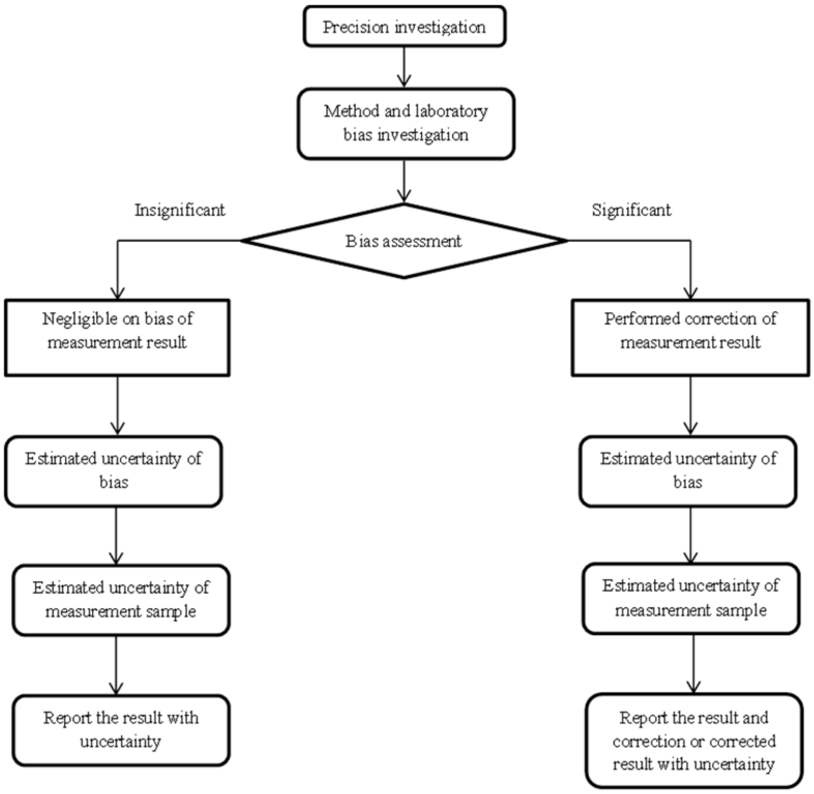

Fig. 1 The chart of uncertainty estimation of measurement by single laboratory validation approach

III. Results and Discussion

For investigation of the within-laboratory reproducibility, its standard uncertainty (u_{rw}), was 1.1%. The mean value of control sample was 0.03333 W/m·K. The standard deviation (1) was 0.00035 W/m·K. Thermal conductivity value, upper warning limit (UWL), lower warning limit (LWL), upper action limit (UAL) and lower action limit (LAL) were evaluated and plotted in Fig. 2 for monitoring precision in laboratory.

For investigation of bias, the bias value (2) was -0.00014 W/m·K. The repeatability and uncertainty of CRM was calculated then the assessment of bias was done.

For standard uncertainty of CRM (u_{crm}), it was affected by several sources uncertainties when was measured on HFM.

- The standard uncertainty from SRM1453 certificate (u_{cer}) was 0.8%.
- The standard uncertainty of the density of CRM ($u_{d(crm)}$) was 0.6% which was calculated as (6). By its dimension and mass measurements, the uncertainty of each value was derived from repeatability, the certificate of calibration and resolution of its measuring equipment. The uncertainty of width, length, thickness and mass were 0.05%, 0.05%, 0.6% and 0.1%, respectively.
- The uncertainty of temperature (u_t) from HFM was 0.7% which was calculated as (7). The standard uncertainty of repeatability (u_{at}) was 0.01%. The standard uncertainty of temperature device certificate (u_{cert}) was 0.67%. The standard uncertainty of resolution of temperature reading (u_{rt}) was 0.01%. The standard uncertainty of temperature uniformity (u_{ut}) was 0.07%.
- The standard uncertainty of the 25 mm and 75 mm of jigs was calculated as (8). The maximum uncertainty was representative of the thickness measurement. The uncertainty of thickness (u_{th}), the plates separation of HFM, was 0.5%. It come from the standard uncertainty of

repeatability (u_{ath}), the certificate of the jig's calibration (u_{certh}) and thickness resolution of HFM (u_{rth}). There was 0.5%, 0.001% and 0.0004%, respectively.

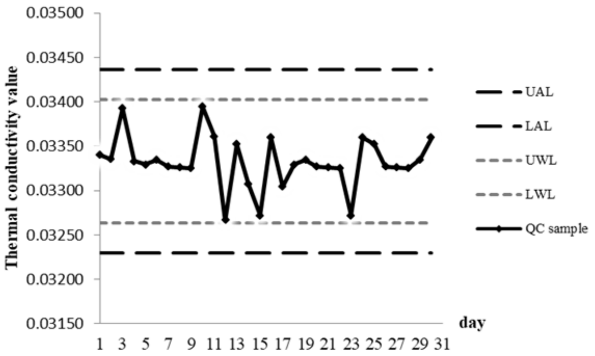

Fig. 2 Control chart of precision

- The resolution of HFM for CRM measurement is 0.00001 W/m·K. The standard uncertainty from resolution of HFM (u_r) calculated as (9) was 0.01%.

In Fig. 3, several sources of standard uncertainties take part in standard uncertainty of CRM.

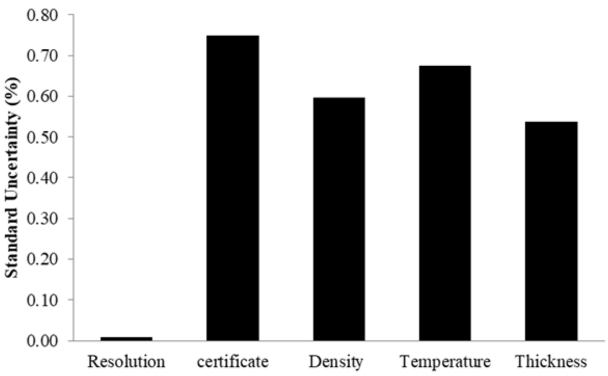

Fig. 3 Sources of standard uncertainties of CRM measurement

For assessment of method and laboratory bias (4), the bias value was insignificant. Hence, it was negligible for correction.

For standard uncertainty of method and laboratory bias (u_{bias}), it consisted of the bias value, the repeatability and the standard uncertainty of CRM (u_{crm}). The standard uncertainty of CRM is a major uncertainty because there are several sources of uncertainties happened in CRM measurement process of bias assessment. The standard uncertainty of method and laboratory bias is presented in Table I.

TABLE I
DATA FOR LABORATORY AND METHOD BIAS ASSESSMENT

Source of Uncertainty	Standard uncertainty (%)
Bias	-0.4
Repeatability	0.3
CRM	1.3

For sample measurement, the standard uncertainty of repeatability (u_a) calculated as (11) was 0.1%. The density of sample ($u_{d(x)}$) calculated as (6) was 0.1%. The resolution of HFM ($u_{r(x)}$) was 0.001%. Each standard uncertainty of sample measurement is displayed in Fig. 4. The combined standard uncertainty calculated as (12) was 1.7%.

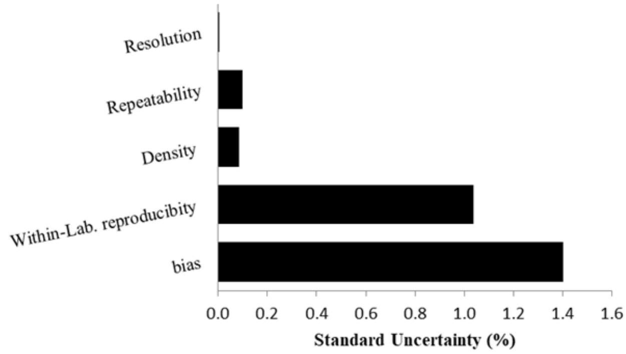

Fig. 4 Standard uncertainties of sample measurement by single laboratory validation approach

The uncertainty of laboratory and method bias becomes a highest uncertainty of measurement sample. However, laboratory could be developed the measurement system, used high accuracy of device and improved competency for better uncertainty.

The measurement result of sample with expanded uncertainty (13) was 0.03367 W/m·K ± 3.5% (k = 2) at mean temperature 23.5 °C.

The uncertainty sources of thermal conductivity measurement by single laboratory validation approach are shown in Fig. 5.

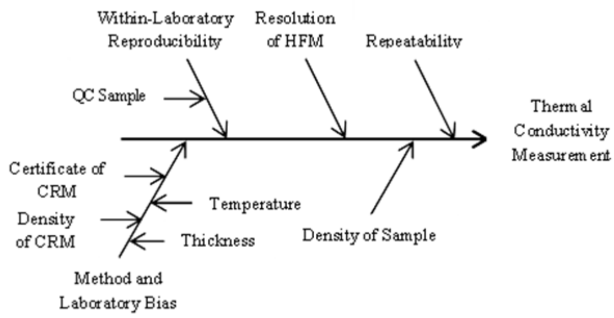

Fig. 5 Fish bone diagram of thermal conductivity measurement uncertainty by single laboratory validation approach

IV. CONCLUSION

The single laboratory validation approach is an alternative method for uncertainty estimation. It is appropriate for routine testing laboratory. However, there are varieties of process measurement for thermal conductivity. Therefore, the uncertainty values come from several sources depending on instruments, measurement procedures, including sampling or sample preparation. The result of thermal conductivity with uncertainty of sample is beneficial for measurement technique improvement, the quality manage of laboratory and potential for trade.

ACKNOWLEDGMENT

This work was supported facilities by Thermal Insulation Testing Laboratory, Department of Science service.

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