Report on the CCT Supplementary comparison S2 on thermal conductivity measurements of insulating materials by guarded hot plate

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Abstract

Seven National Metrology Institutes (NMIs) from France, United States, United Kingdom, Russia, Mexico, China and Germany participated in an inter-laboratory comparison on thermal conductivity measurements by the Guarded Hot Plate method. This action was part of a series of supplementary inter-laboratory comparisons (including infrared spectral emittance and thermal diffusivity) sponsored by the Consultative Committee on Thermometry (CCT) Task Group on Thermophysical Quantities (TG-ThQ). The objective of this collaborative work was to strengthen the consistency of thermal conductivity measurements carried out worldwide on low conductive materials. Measurements were conducted successively by all participants on the same sets of specimens of insulating materials (mineral wool and expanded polystyrene) at temperatures ranging from 10 °C to 40 °C, according to the International Standard ISO 8302. This protocol aimed to minimize issues of material variability by circulating the same pairs of specimens among the laboratories following the strict format of a round-robin test program. More than 120 data points (combinations of material, thickness and temperature) were compared. 92 % 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.

Keywords: Thermal conductivity; Comparison; Guarded Hot Plate; Insulating Materials

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1 Introduction

The guarded hot plate (GHP) method is recognized worldwide as the primary technique for the measurement of thermal conductivity of low conducting materials. This steady-state method, which is used especially for analyzing insulating materials for building applications, is standardized in the ISO 8302 [1] and ASTM C177 [2] standards. A number of the major National Measurement Institutes (NMIs) have developed measurement capabilities and standards to support industrial needs in these areas for accurate thermal conductivity measurements.

In recent years, several inter-laboratory comparisons of guarded hot plate measurements were performed by accredited testing organizations, insulating materials manufacturers and NMIs [3-5] in order to assess the consistency of their measurements. These previous 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.

A supplementary comparison on thermal conductivity of insulating materials was for the first time organized by the Bureau International des Poids et Mesures (BIPM). It was conducted by the Task Group for Thermophysical Quantities (CCT TG-ThQ), named formerly Working Group 9 "Thermophysical Properties", of the Consultative Committee for Thermometry (CCT). Seven National Metrology Institutes were involved: Laboratoire National de Métrologie et d'Essais (LNE), National Institute of Standards and Technology (NIST), National Physical Laboratory (NPL), National Institute of Metrology (NIM), Mendeleyev Institute for Metrology (VNIIM), Centro Nacional de *Metrología* (CENAM) and Physikalisch-Technische Bundesanstalt (PTB).

The thermal conductivities of two thermal insulating materials (mineral wool and expanded polystyrene) were measured at 10 °C, 23 °C, and 40 °C by using guarded hot plate (single-specimen or two-specimens) apparatus. The comparison was organized as a round-robin test. A set of specimens was circulated between the NMIs and was, thus, successively measured by them.

The primary goal of this supplementary comparison was to establish the state of the art of thermal conductivity measurements by the guarded hot plate method in National Metrology Institutes, by assessing in particular the variability and coherency of their thermal conductivity measurements. This report describes the inter-laboratory comparison protocol, the different GHP apparatus used by the participants, the selected materials, the tests results and the data analyses.

Remark: Table 1 shows a list identifying all of the variables and subscripts contained in this report.

Table 1 : List of variables and subscripts

Variable	Description
A	Area (m^2)
c	Correlation coefficient
D	Degree of equivalence or relative deviation from CRV
E	Error function
k	Coverage factor for expanded uncertainty
L	Thickness (m)
R_{th}	Thermal resistance ($m^2 \cdot K \cdot W^{-1}$)
T	Temperature (°C or K)
u	Standard uncertainty
U	Expanded uncertainty
λ	Thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$)
ρ	Density ($kg \cdot m^{-3}$)
Φ	Heat flow (W)
φ	Heat flux density ($W \cdot m^{-2}$)
ϕ	Diameter (mm)
Subscript	
c	Cold
h	Hot
i	Index of laboratory
k	Index of measurements
m	Mean
o	Normal conditions
add	Additional sources of uncertainty
adj	Adjusted to cut-off value if criteria is met
cut-off	Lower limit to which uncertainty is adjusted if criteria is met
CRV	Comparison Reference Value

2 Description of the comparison

2.1 Participating institutes

Table 2 summarizes the participant laboratories information. The laboratories are listed in the chronological order in which they performed their measurements.

Table 2 : Laboratory participants

ID	National Metrology Institute	Country	Contact person
NIST	National Institute of Standards and Technology	United States	Robert Zarr
LNE	Laboratoire National de Métrologie et d'Essais	France	Bruno Hay
NPL	National Physical Laboratory	United Kingdom	Clark Stacey
VNIIM	Mendeleyev Institute for Metrology	Russia	Nikolay Sokolov
NIM	National Institute of Metrology	China	Jintao Zhang
CENAM	Centro Nacional de Metrología	Mexico	Leonel Lira Cortes
PTB	Physikalisch-Technische Bundesanstalt	Germany	Ulf Hammerschmidt

Before starting the comparison, the participants were requested to give some technical information about their apparatus (type and size, working temperature, specimen dimensions, among other factors) in a specific questionnaire. The main features of these instruments are presented in Table 3. Examples of uncertainty budget, estimated by each laboratory according to [6], are given in section 2.4. Depending on the participant, the individual GHP apparatus operates either with a single specimen or a pair of specimens.

Table 3 : Laboratory guarded hot plate apparatus

Laboratory	NIST	LNE	NPL	VNIIM	NIM	CENAM	PTB
GHP type	Double	Double	Single	Double	Single	Double	Single
Specimen dimensions (mm)	ϕ 1016	610 x 610	610 x 610	ϕ 330	ϕ 337	ϕ 305	ϕ 100
Metering area (mm)	ϕ 406.4	300 x 300	305 x 305	ϕ 150	ϕ 200	ϕ 165	-
Mean temperature (°C)	7 to 65	0 to 50	5 to 40	-25 to 70	≥ 20	-5 to 60	-50 to 195
Specimen thickness (mm)	10 to 300	20 to 160	20 to 250	20 to 80	20 to 80	up to 50	5 to 25
Temperature difference (K)	5 to 30	5 to 40	10 to 30	5 to 20	5 to 30	5 to 30	3 to 20
Thermal conductivity (10^{-3} W·m^{-1}·K^{-1})	5 to 150	1.5 to 1500	≤ 100	20 to 200	50 to 2000	30 to 170	20 to 7000

2.2 Tested materials

Two different types of insulating material were selected for this comparison: mineral wool (MW) and expanded polystyrene (EPS). These materials were chosen due to low dispersion of density within a batch and for long-term stability.

- The mineral wool specimens were high density glass fiber boards (nominal density: 72 $kg \cdot m^{-3}$) with a thickness of 35 mm. They came from a batch of a certified reference material named IRMM-440, whose properties were characterized by six European laboratories in a framework of a certification project initiated by the Institute of Reference Materials and Measurements [7].
- Two special batches of expanded polystyrene boards (35 mm and 70 mm thick) with a nominal density of 22 $kg \cdot m^{-3}$ were specifically produced free of charge by Lafarge (France). It was a grey EPS containing graphite in order to avoid the "thickness effect" that is observed usually for normal white EPS (cf. EN 13163 standard [8]).

A pair of disk-shaped specimens (with diameter of 1016 mm) was prepared by LNE from each of the three materials characterized above. These specimens were identified MW35-1, MW35-2, EPS35-1, EPS35-2, EPS70-1 and EPS70-2 (see Figure 1). Two other pairs of EPS specimens of thicknesses 20 mm and 25 mm (identified EPS20-1, EPS20-2, EPS25-1 and EPS25-2) were specially machined for PTB because of their variant type of GHP apparatus (see Table 3).

Figure 1 : Studied specimens of mineral wool and expanded polystyrene

2.3 Comparison process

The comparison protocol was jointly drawn up by CCT Working Group 9 taking into account the major characteristics (specimen dimensions, temperature and thermal conductivity ranges, etc.) of the guarded hot plate apparatus involved in this comparison. The thermal conductivity measurements were carried out using GHP apparatus according to the International Standard ISO 8302 [1] by all laboratories except PTB (whose dimensions of the GHP differ from those recommended in [1]). In the case of two-specimen apparatus, the mean thermal conductivity λ of the pair of specimens is determined at steady-state conditions by using equation (1).

$$\lambda = \frac{\Phi \cdot L}{A \cdot \Delta T} \tag{1}$$

where Φ is half of the heat flow delivered by the electric heater (W) and passing through a surface of the metering area for the specimen; A (m^2) is the metering cross-section area; $\Delta T = T_h - T_c$ (K) is the measured temperature difference between each of the specimens' hot (T_h) and cold surfaces (T_c); and L (m) is the mean thickness of the specimens. Values of λ are indicated for the mean specimen temperature, $T_m = (T_h + T_c)/2$.

In a single-specimen apparatus, the second specimen was replaced by insulation and a guard plate. In this case, heat flow equals the heat flow delivered by the electric heater. Depending on its individual apparatus, each participant performed either just a part of the following program or the whole on each pair of specimens.

- Four successive runs at a fixed temperature of 23 °C with a temperature difference of 20 °C over a short period of time. After each run, the specimens were removed from the apparatus and then reassembled. This procedure yields information on the repeatability.
- One run at each of the two mean test temperatures of 10 °C and 40 °C under a temperature difference of 20 °C.

Table 4 summarizes each individual measurement programs. The same set of specimens was circulated between the different NMIs and was thus, successively measured by the participants (with the exception of PTB). Each participant sent the specimens back to the pilot laboratory after having performed their series of measurements.

Table 4 : Summary of the measurement program

Laboratory		NIST	LNE	NPL	VNIIM	NIM	CENAM	PTB
Temperatures (°C) and repetition	10	✓	✓	✓	✓		✓	
	23	✓	✓	✓	✓	✓	✓	✓
	40	✓	✓	✓	✓	✓	✓	✓
Materials and thickness (mm)	MW35	✓	✓	✓	✓	✓	✓	
	EPS35	✓	✓	✓	✓	✓	✓	✓ (*)
	EPS70	✓	✓	✓			✓	

(*) PTB had performed measurements with 20 and 25 mm thick EPS specimens. In this study, the results were analyzed with those obtained by other laboratories with 35 mm EPS specimens.

The pilot laboratory (LNE) supervised all specimens on a regular basis by recurrently measuring their densities. In addition, it arranged machining of the specimens according to the requests of each successive participant. The specimens were transported from laboratory to laboratory in closed flight containers. Initially, the specimens were measured by NIST that needed 1016 mm diameter specimens. Then the specimens were stepwise cut down, first to 610 mm x 610 mm and finally to 330 mm diameter. Each cutting process had to leave the central part of a specimen undisturbed. All cutting scraps were marked and retained at the pilot laboratory, in order to reassemble, as closely as possible, 610 mm x 610 mm specimens at the end of the comparison process. This procedure enabled the pilot laboratory to check the stability of the specimens by measuring their thermal conductivity at the beginning and at the end of the comparison.

Additional details of the comparison process are given in Appendix 4 “Technical Protocol”.

2.4 Instruments and uncertainties descriptions

2.4.1 NIST

In the frame of this inter-laboratory comparison, NIST has used the apparatus described in Figure 2. The apparatus plates, shown in the horizontal arrangement, are enclosed by an insulated environmental chamber. The plates are an aluminium alloy and the surfaces in contact with the specimens are flat to within 0.05 mm and are anodized black to have a total emittance of 0.89. The hot plate is rigidly mounted and each cold plate translates in the vertical direction for installation of the specimen. A clamping force is transmitted axially to each cold plate by a precision stepper motor and an in-line load cell measures the applied loading during the test.

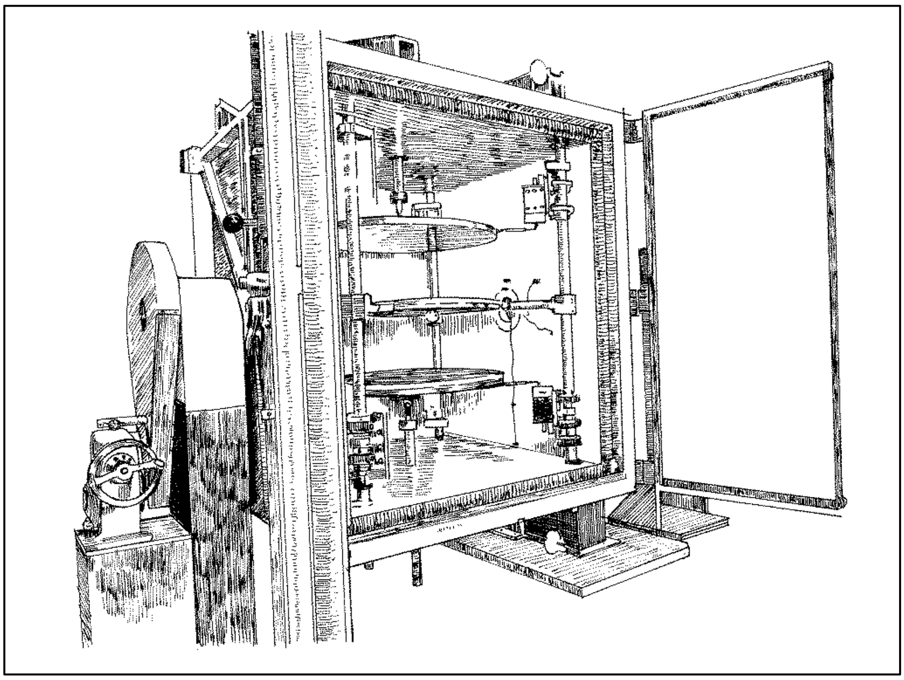


Figure 2 : NIST 1016 mm guarded hot plate

- The hot plate is monolithic in construction and is nominally 16.1 mm thick and consists of a meter plate 405.6 mm in diameter and a co-planar, concentric guard plate. The circular gap separating the meter plate and guard plate is 0.89 mm wide at the plate surface. The gap cross-sectional profile is diamond shaped to minimize lateral heat flow. The temperature difference across the gap is measured using an eight junction Type E thermopile. The meter-plate heater is located at a diameter of 287 mm and the inner and outer guard heaters are at diameters of 524.7 mm and 802.2 mm, respectively. The heating element for the meter plate is a thin nickel-chrome ribbon filament network, 0.1 mm thick and 4 mm wide, electrically insulated with polyimide, having an electrical resistance at room temperature of approximately 56 Ω.
- Each cold plate is 25.4 mm thick and contains flow channels arranged in a double spiral configuration that circulate a mixture of ethylene glycol and distilled water. The plate temperature is maintained by a dedicated refrigerated bath controlled to within ±0.05 K over a temperature range of –20 °C to 60 °C. The outer surfaces and edges of the cold plates are insulated with 102 mm of extruded polystyrene foam (not shown in Figure 2).

The primary temperature sensors for each plate are small capsule-type platinum resistance thermometers. The sensor is strain-free platinum wire and is supported in a gold-plated copper cylinder 3.18 mm in diameter by 9.7 mm long backfilled with helium gas and hermetically sealed. The sensors are placed in the metering section of each plate.

The meter plate electrical power is determined by measurement of:

- the direct-current voltage across the meter-plate heater by voltage taps welded to the heater leads in the center of the gap; and,
- the corresponding current in the circuit determined by a 0.1 Ω standard resistor in series with the heater that is placed in an oil bath at 25.0 °C.

Uncertainty budget

An example of uncertainty budget for EPS35 specimen measurements at 23 °C is shown in Table 5.

Table 5 : NIST uncertainty budget for EPS35 specimen at 23 °C

Components	**Value**	u_i	c_i (*)	$\lvert c_i \cdot u_i \rvert$
Heat flow Φ	2.360 W	$9\ 10^{-3}$ W	0.01352 $m^{-1} \cdot K^{-1}$	$1.2\ 10^{-4}$
Metering area A	0.1298 m^2	$2.866\ 10^{-5}$ m^2	-0.24571 $W \cdot m^{-3} \cdot K^{-1}$	$1.0\ 10^{-5}$
Specimen thickness L	0.03508 m	$6\ 10^{-5}$ m	0.90916 $W \cdot m^{-2} \cdot K^{-1}$	$6.0\ 10^{-5}$
Temperature diff. ΔT	20.00 K	$6.7\ 10^{-2}$ K	-0.00159 $W \cdot m^{-1} \cdot K^{-2}$	$1.1\ 10^{-4}$
Thermal conductivity λ from Eq (1)	0.03189 $W \cdot m^{-1} \cdot K^{-1}$	*Relative combined standard uncertainty (k=1) $(\Sigma \lvert c_i \cdot u_i \rvert^2)^{0.5}$*		0.00017 $W \cdot m^{-1} \cdot K^{-1}$

(*) $c_\Phi = \lambda/\Phi$, $c_A = -\lambda/A$, $c_L = \lambda/L$, $c_{\Delta T} = -\lambda/\Delta T$

2.4.2 LNE

In the frame of this inter-laboratory comparison, LNE has used the apparatus described in Figure 3. The apparatus has been designed for operation in the double-sided mode of operation and built in-house by LNE. The apparatus is symmetric, horizontal and is designed for operation at near room temperature. The apparatus is made of 3 different elements: two cold plates, one for each side, and one guarded hot plate.

- The guarded hot plate delivers heat through a spiral network of NiCr wires (4 mm large x 0.2 mm thick). The total resistance is 232 Ω. The heater provides a distributed heat flow through the uniformisation plates. These plates are made of aluminium with a roughness of 0.05 mm to avoid any parasitic contact resistance. Although the size of the plates is 610 x 610 mm, the metering area is 300 x 300 mm. In order to avoid/reduce heat losses, the guard is controlled by a series of 56 type K junctions mounted into a thermopile
- Each cold plate consists in a water loop (squared spiral shape for ensuring a uniform temperature profile) whose temperature is controlled by an external bath. The mass flow rate in each cold plate is controlled by a dedicated flowmeter. Each cold plate is 45 mm thick. In the standard double sided mode of operation, the two cold plates are maintained at equivalent temperatures.

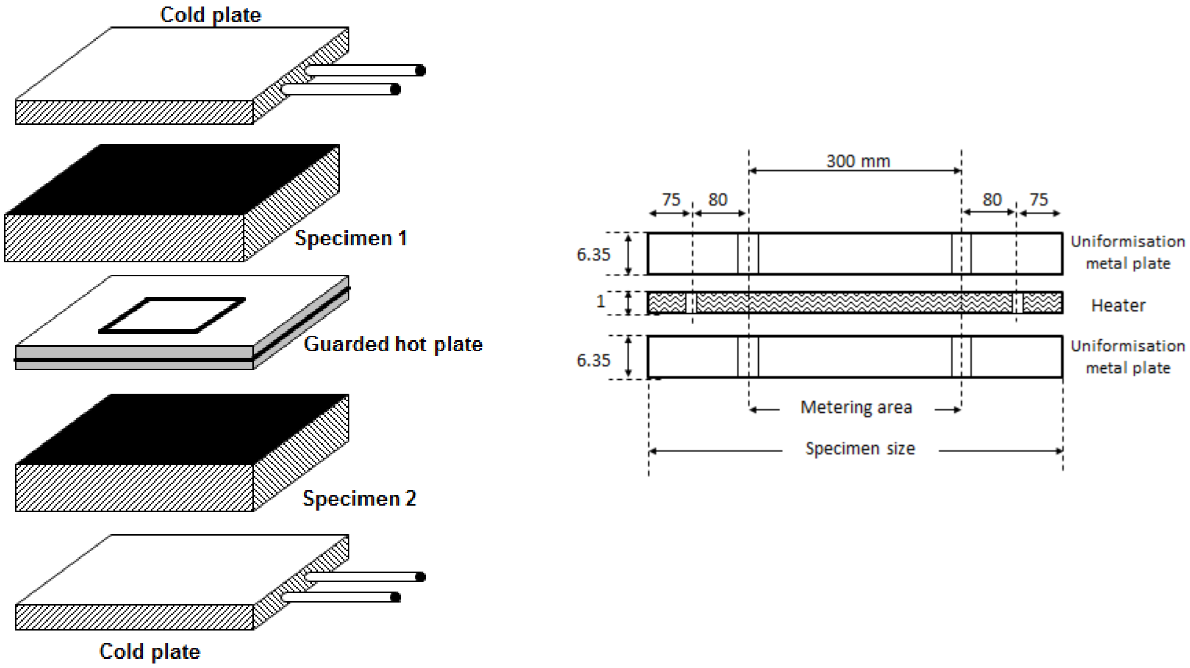


Figure 3 : LNE guarded hot plate (left) Schematic overview (right) Guarded hot plate

Type K thermocouples (Chromel/Alumel) are used to measure the temperature of the hot and cold plates: five thermocouples are located in the metering area of each uniformisation plate. Three thermocouples are used to measure temperature of the guard.

The power delivered by the heater is calculated by measuring:

- the current intensity going through the heater, thanks to 1Ω standard resistance,
- the voltage between the entry and exit wires of the resistance in the metering area.

Uncertainty budget

An example of uncertainty budget for EPS35 specimen measurements at 23 °C is shown in Table 6.

Table 6 : LNE uncertainty budget for EPS35 specimen at 23 °C

Components	Value	u_i	c_i (*)	$\lvert c_i \cdot u_i \rvert$
Heat flow Φ	1.6430 W	10^{-4} W	0.01942 $m^{-1}\cdot K^{-1}$	1.9 10^{-6}
Metering area A	0.09 m^2	7 10^{-5} m^2	-0.35457 $W\cdot m^{-3}\cdot K^{-1}$	2.5 10^{-5}
Specimen thickness L	0.03489 m	9 10^{-5} m	0.91463 $W\cdot m^{-2}\cdot K^{-1}$	8.2 10^{-5}
Temperature diff. ΔT	19.96 K	8.5 10^{-2} K	-0.00160 $W\cdot m^{-1}\cdot K^{-2}$	1.4 10^{-4}
Thermal conductivity λ from Eq (1)	0.03191 $W\cdot m^{-1}\cdot K^{-1}$	*Relative combined standard uncertainty (k=1)* $(\Sigma \lvert c_i \cdot u_i \rvert^2)^{0.5}$		0.00016 $W\cdot m^{-1}\cdot K^{-1}$

(*) $c_\Phi = \lambda/\Phi$, $c_A = -\lambda/A$, $c_L = \lambda/L$, $c_{\Delta T} = -\lambda/\Delta T$

Values u_i comes from an analysis of each element of the GHP apparatus:

- u_Φ includes the defect of thermal equilibrium inside the guard rings and the correction due to heat losses from the cold wires,
- u_L includes uncertainty due to thickness variation with temperature,
- $u_{\Delta T}$ includes the accuracy and the drift of the temperature sensors, the non-uniformity of the temperature at the plates surfaces.

2.4.3 NPL

In the frame of this inter-laboratory comparison, NPL used the apparatus described in Figure 4. The NPL apparatus was a 610 x 610 mm single-sided Guarded Hot Plate (NPL 610GHP) with a 305 x 305 mm metering area and linear temperature gradient edge-guards. In this apparatus the specimen was mounted horizontally with heat flow upwards and a heat flux transducer incorporated in the cold plate as an additional monitor of heat flow through the specimen. The apparatus was designed for measurements in the temperature range 5 °C to 40 °C and conformed to ISO 8302 and EN 12667. Its use was normally restricted to specimens between 20 mm and 250 mm thick having a thermal conductivity up to 0.1 $W \cdot m^{-1} \cdot K^{-1}$ and thermal resistance down to 0.2 $m^2 \cdot K \cdot W^{-1}$.

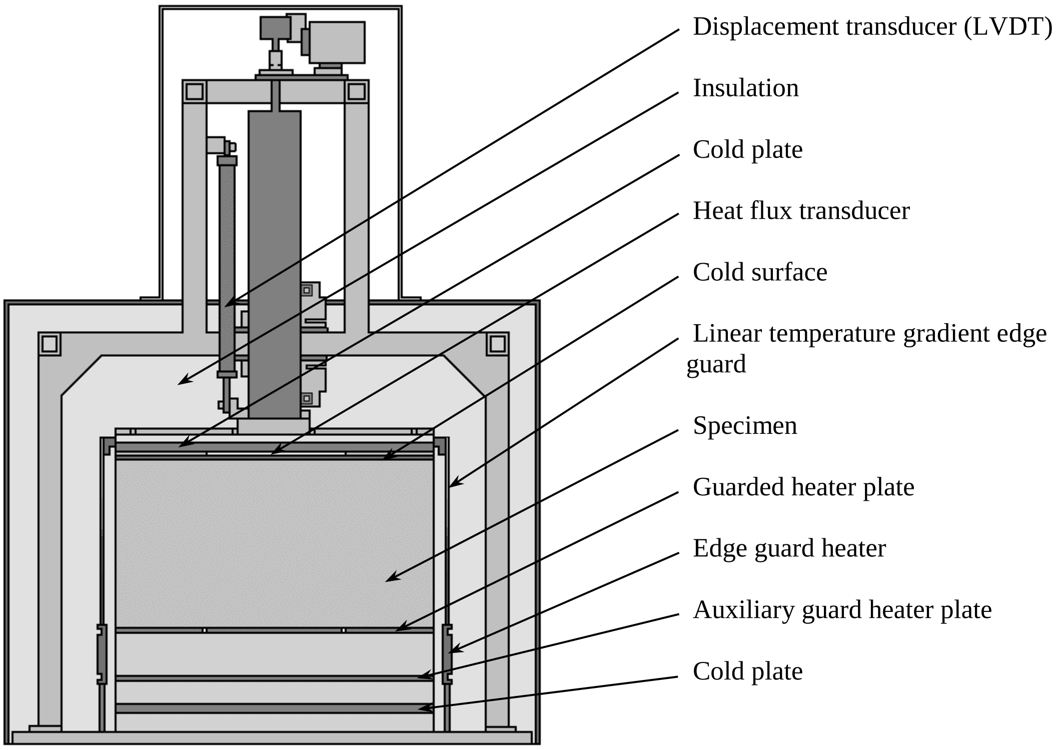


Figure 4 : NPL guarded hot plate

The guarded heater plate was made of copper and had lateral dimensions of 610 x 610 mm, with a central metering area of 305 x 305 mm and an air gap of 2 mm. Temperature balance between the metering area and lateral guard was maintained using the output of a 20-junction thermopile to control the power supplied to the lateral guard heater. Linear temperature gradient edge-guards were used to further reduce edge heat gains or losses. The cold plate of the apparatus was also made of copper and its temperature was maintained during measurements by a combination of fluid circulation and electrical heating. The surfaces of the guarded heater plate and cold surface plate had an estimated total hemispherical emittance of 0.9 and all the temperature sensors (Type E thermocouples) and electrical instruments used were calibrated with traceability to United Kingdom national standards. An auxiliary guard was maintained at the same temperature as the guarded heater plate in order to ensure that the heat generated by the guarded heater plate flowed upwards through the specimen. The auxiliary guard was made from 12 mm thick copper plate and separated from the guarded heater plate by 75 mm thick foam insulation. The heat flux transducer, which had a measuring area of 250 x 250 mm and was mounted on the surface of the cold plate, was used to confirm that extraneous edge heat gains or losses had been reduced to an acceptable level.

Uncertainty budget

An example of uncertainty budget for EPS35 specimen measurements at 23 °C is shown in Table 7.

Table 7 : NPL uncertainty budget for EPS35 specimen at 23 °C

Components	**Value**	u_i	c_i (*)	$\lvert c_i \cdot u_i \rvert$
Heat flow Φ	1.7205 W	$3.08\ 10^{-3}$ W	$0.01875\ \mathrm{m^{-1} \cdot K^{-1}}$	$5.76\ 10^{-5}$
Metering area A	$0.09315\ \mathrm{m^2}$	$6.02\ 10^{-5}\ \mathrm{m^2}$	$-0.34625\ \mathrm{W \cdot m^{-3} \cdot K^{-1}}$	$2.08\ 10^{-5}$
Specimen thickness L	0.03482 m	$2.68\ 10^{-4}$ m	$0.92629\ \mathrm{W \cdot m^{-2} \cdot K^{-1}}$	$2.48\ 10^{-4}$
Temperature diff. ΔT	19.94 K	$1.82\ 10^{-2}$ K	$-0.00161\ \mathrm{W \cdot m^{-1} \cdot K^{-2}}$	$2.95\ 10^{-5}$
Thermal conductivity λ from Eq (1)	$0.03226\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$	*Relative combined standard uncertainty (k=1) $(\Sigma \lvert c_i \cdot u_i \rvert^2)^{0.5}$*		$0.00026\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$

(*) $c_\Phi = \lambda/\Phi$, $c_A = -\lambda/A$, $c_L = \lambda/L$, $c_{\Delta T} = -\lambda/\Delta T$

Values u_i comes from an analysis of each element of the GHP apparatus:

- u_Φ includes resolution, calibration of electrical power measurement, metering/guard balance, metering/auxiliary balance and edge heat gains/losses.
- u_A includes resolution, calibration and alignment.
- u_L includes resolution, calibration, parallelism, linearisation and stability.
- $u_{\Delta T}$ includes resolution, calibration, linearisation and spread.

2.4.4 VNIIM

In the frame of this inter-laboratory comparison, VNIIM used a homemade apparatus. It is symmetric and horizontal, and is made of three different elements: two cold plates, one for each side, and one guarded hot plate (Figure 5).

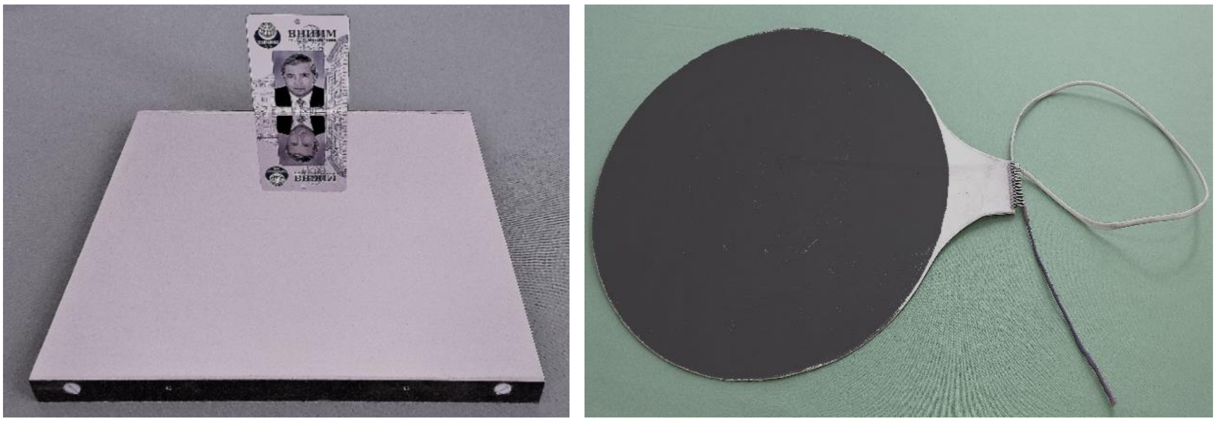


Figure 5 : VNIIM cold plate (left) and guarded hot plate (right)

The guarded hot plate delivers heat through a spiral network of copper wires (diameter 0.15 mm). The heater resistance of the metering area is 173 Ω and is connected to a 4-wire circuit. The guarded hot plate is made of plastic and has a thickness of 0.67 mm and a roughness less than 0.01 mm ($R_z = 0.1$). The diameters of the guarded hot plate and metering area are 300 mm and 150 mm respectively. It contains 8 thermocouples for measuring temperature and 16 differential thermocouples (copper-constantan). The cold junctions of eight thermocouples are at the temperature of melting ice or at the triple point of water. Each cold plate has a water loop of squared shape spiral for ensuring a uniform temperature profile. An external bath controls the temperature of the water loop. The thickness of the cold plate is 23 mm. In the standard mode, the two cold plates are maintained at equal temperatures. Working surfaces have a protective coating of hafnium and have a roughness R_z of 0.1 (mirror). Platinum resistance thermometers are positioned in the metering area of cold plates. They enable before measurement to calibrate thermocouples at hot and cold temperatures without dismantling.

Uncertainty budget

Table 8 shows an example of the uncertainty budget for measurements of EPS35 samples at 23 °C.

Table 8 : VNIIM uncertainty budget for EPS35 specimen at 23 °C

Components	Value	u_i	c_i (*)	$\|c_i \cdot u_i\|$
Heat flow Φ	0.3342 W	10^{-4} W	0.09783 $m^{-1} \cdot K^{-1}$	9.8 10^{-6}
Metering area A	0.017663 m^2	2 10^{-5} m^2	-1.851 $W \cdot m^{-3} \cdot K^{-1}$	3.7 10^{-5}
Specimen thickness L	0.0348 m	9 10^{-5} m	0.9395 $W \cdot m^{-2} \cdot K^{-1}$	8.5 10^{-5}
Temperature diff. ΔT	20.14 K	1.5 10^{-2} K	-0.00162 $W \cdot m^{-1} \cdot K^{-2}$	2.4 10^{-5}
Thermal conductivity λ from Eq (1)	0.03270 $W \cdot m^{-1} \cdot K^{-1}$	*Relative combined standard uncertainty (k=1) $(\Sigma \|c_i \cdot u_i\|^2)^{0.5}$*		10^{-4} $W \cdot m^{-1} \cdot K^{-1}$

(*) $c_\Phi = \lambda/\Phi$, $c_A = -\lambda/A$, $c_L = \lambda/L$, $c_{\Delta T} = -\lambda/\Delta T$

Values u_i comes from an analysis of each element of the GHP apparatus:

- u_{Φ} includes the defect of thermal equilibrium inside the guard rings and the correction due to heat losses from the cold wires,
- u_L includes uncertainty due to thickness variation with temperature,
- $u_{\Delta T}$ includes the accuracy and the drift of the temperature sensors, the non-uniformity of the temperature at the plates surfaces.

2.4.5 NIM

In the frame of this inter-laboratory comparison, NIM has used a single-sided Guarded Hot Plate. The diameters of the guarded hot plate and metering area are 337 mm and 200 mm respectively. The apparatus is designed for measurements above 20 °C for specimens between 20 mm and 80 mm thick.

Uncertainty budget

An example of uncertainty budget for EPS35 specimen measurements at 23 °C is shown in Table 9.

Table 9 : NIM uncertainty budget for EPS35 specimen at 23 °C

Components	Value	u_i	c_i (*)	$\|c_i \cdot u_i\|$
Heat flow Φ	0.6063 W	/	/	/
Metering area A	0.0314 m^2	/	/	/
Specimen thickness L	0.03462 m	/	/	/
Temperature diff. ΔT	19.69 K	/	/	/
Thermal conductivity λ from Eq (1)	0.03395 $W \cdot m^{-1} \cdot K^{-1}$	*Relative combined standard uncertainty (k=1) $(\Sigma \|c_i \cdot u_i\|^2)^{0.5}$*		0.00025 $W \cdot m^{-1} \cdot K^{-1}$

(*) $c_\Phi = \lambda/\Phi$, $c_A = -\lambda/A$, $c_L = \lambda/L$, $c_{\Delta T} = -\lambda/\Delta T$

2.4.6 CENAM

In the frame of this inter-laboratory comparison, CENAM used the apparatus described Figure 6. It was built in-house by CENAM and operated in steady state with double-sided mode of measuring. The apparatus is symmetric, horizontal and is designed for operation at room temperature. It has 3 plates: two cold plates, one for each side, and one central guarded hot plate. The plates, 305 mm in diameter, are made of copper with a roughness of 0.05 mm. Cold plates are 27 mm in thickness and the guarded hot plate has a thickness of 12mm.

- The line heat source of the guarded hot plate is an electric heater of 94.3 Ω; this heater maintains a uniform plate average surface temperature of the hot plate. The size of the hot plate is 305 mm in diameter and the metering area diameter is 150 mm. The temperature of the plate is controlled for 7 thermocouples type T connected in a thermopile configuration.
- Temperature of cold plates is controlled by an external bath which recirculates water in a loop; cold plates are maintained at same temperature.

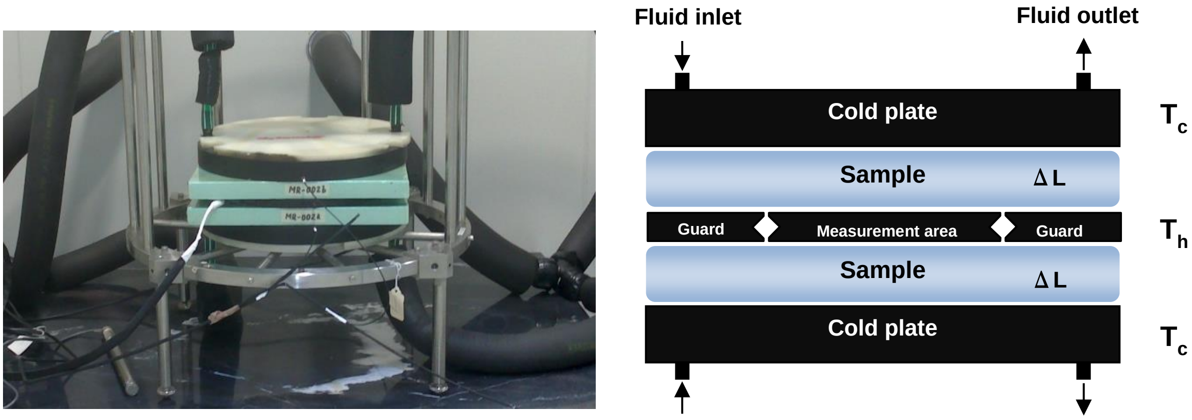


Figure 6 : CENAM guarded hot plate

Type T thermocouples are used to measure the temperature of the hot and cold plates, three of them are located in the metering area of each plate and three more are placed in the guard. The power delivered by heaters is calculated by measuring the electrical current and the applied voltage in the heater.

Uncertainty budget

An example of uncertainty budget for EPS35 specimen measurements at 23 °C is shown in Table 10.

Table 10 : CENAM uncertainty budget for EPS35 specimen at 23 °C

Components	Value	u_i	c_i (*)	$\lvert c_i \cdot u_i \rvert$
Heat flow Φ	0.3831 W	$5\ 10^{-5}$ W	0.08496 K^{-1}	$4.25\ 10^{-6}$
Metering area A	0.021404 m^2	$2\ 10^{-6}$ m^2	-1.52073 $W{\cdot}m^{-3}{\cdot}K^{-1}$	$3.04\ 10^{-6}$
Specimen thickness L	0.03455 m	$1\ 10^{-4}$ m	0.94211 $W{\cdot}m^{-2}{\cdot}K^{-1}$	$9.42\ 10^{-5}$
Temperature diff. ΔT	19.00 K	0.162 K	-0.00171 $W{\cdot}m^{-1}{\cdot}K^{-2}$	$2.78\ 10^{-4}$
Thermal conductivity λ from Eq (1)	0.03278 $W{\cdot}m^{-1}{\cdot}K^{-1}$	*Relative combined standard uncertainty (k=1) $(\Sigma \lvert c_i{\cdot}u_i \rvert^2)^{0.5}$*		0.00029 $W{\cdot}m^{-1}{\cdot}K^{-1}$

(*) $c_\Phi = \lambda/\Phi$, $c_A = -\lambda/A$, $c_L = \lambda/L$, $c_{\Delta T} = -\lambda/\Delta T$

Values of u_i were calculated from an analysis of each element of the GHP apparatus:

- u_{Φ} includes the defect of thermal equilibrium inside the guard rings and the correction due to heat losses though cold wires,
- u_L includes uncertainty due to thickness variation of specimen with temperature,
- $u_{\Delta T}$ includes the accuracy and the drift of the temperature sensors, the non-uniformity of the temperature at the plates surfaces.

2.4.7 PTB

The measurements on EPS20 and EPS25 were carried out using the single plate GHP apparatus "GHP-S". The instrument is a PTB-specific development for use on small cylindrical specimens (∅ 100 mm x 5…25 mm) only. Therefore, it is not covered by one of the relevant testing or construction standards. According to Figure 7, the specimen (A) is sandwiched between the upper hot plate (B) and the lower thermostated (J) cold plate (C). The guard plate (D) on top of the stack and the lateral guard ring (E) surround the hot plate to ensure a unidirectional and uniform heat flow of the imposed rate. The latter components (B…E) are made from nickel-plated copper. A special rigid edge-insulation (F), surrounding the specimen, avoids significant lateral heat losses. The entire stack can be tightly packed without any compaction by the outer push rod (H). The gross heat flow is determined from the electric input power of the hot plate. Here, the voltage drop is directly measured and the current is indirectly determined from the voltage drop across a calibrated four-pole resistor of 1 Ω. The basic working temperature of the specimen is set and maintained constant by immersion of the whole instrument in a thermostated bath (J). All temperatures are determined by copper-constantan thermocouples (∅ 0.2 mm) that are located within bore holes inside the respective copper components. The temperature stations are identified by the numbered points in Figure 7. The instrument is operated fully automatically. Input values have to be set for the actual diameter and thickness of the specimen as well as for its intended temperature difference and mean temperature. During a run the electrical power to the hot plate is adjusted and controlled accordingly. To satisfy the steady-state condition typically takes about six hours. Then, the instrument performs by itself a number of predefined successive runs. Finally, it displays the mean value for the thermal conductivity of the specimen at the predefined mean temperature and, additionally, all relevant data for the corrections to the underlying ideal model (Equation 1).

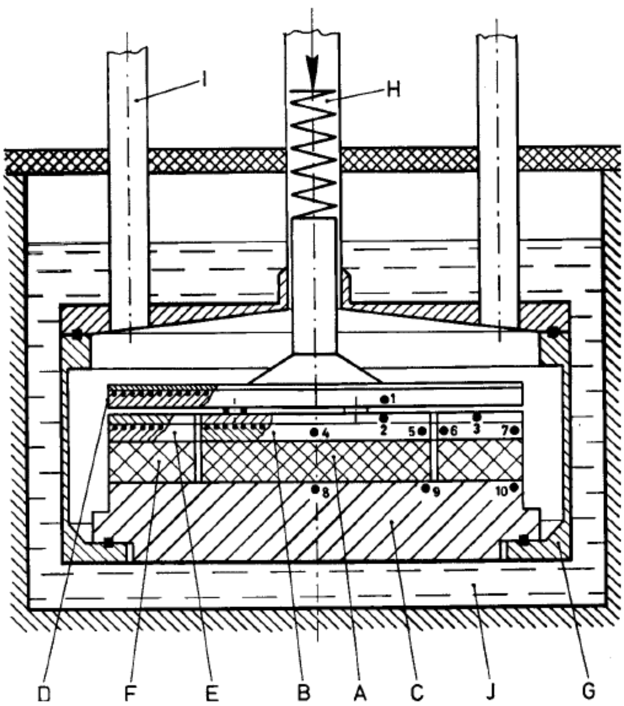


Figure 7 : Schematic of the PTB Guarded Hot Plate Apparatus "GHP-S"

A: specimen, B: hot plate, C: cold plate, D: guard plate, E: guard ring, F: edge insulation, G: casing, H: push rod, I: ducts, J: thermostated bath, 1-10: thermocouples.

Uncertainty budget

The simplified uncertainty budget for the EPS20 specimen measurements at 23 °C is shown in Table 11. For a complete budget including all necessary corrections please see [9].

Table 11 : PTB uncertainty budget for EPS20 specimen at 23 °C

Components	Value	u_i	c_i (*)	$\lvert c_i \cdot u_i \rvert$
Heat flow Φ	0.2467 W	$2.3 \cdot 10^{-4}$ W	0.127 $m^{-1} \cdot K^{-1}$	$2.9 \cdot 10^{-5}$
Metering area A	0.007854 m^2	$7.0 \cdot 10^{-5}$ m^2	-3.997 $W \cdot m^{-3} \cdot K^{-1}$	$2.8 \cdot 10^{-4}$
Specimen thickness L	0.01997 m	$9.0 \cdot 10^{-5}$ m	1.571 $W \cdot m^{-2} \cdot K^{-1}$	$1.4 \cdot 10^{-4}$
Temperature diff. ΔT	20.00 K	$2.0 \cdot 10^{-1}$ K	-0.0016 $W \cdot m^{-1} \cdot K^{-2}$	$3.2 \cdot 10^{-4}$
Thermal conductivity λ from Eq (1)	0.0314 $W \cdot m^{-1} \cdot K^{-1}$	*Relative combined standard uncertainty (k=1) $(\Sigma \lvert c_i \cdot u_i \rvert^2)^{0.5}$*		0.00045 $W \cdot m^{-1} \cdot K^{-1}$

(*) $c_\Phi = \lambda/\Phi$, $c_A = -\lambda/A$, $c_L = \lambda/L$, $c_{\Delta T} = -\lambda/\Delta T$

2.5 Processing of the comparison data

2.5.1 Data pre-processing for potential correction/compensation

In this comparison, two factors (bulk density and specimens stability) have been checked to identify potential needs of compensation, in order to properly compare results from all participants.

Bulk density measurements

Prior to the analysis of the results, particular attention has been paid to coherence between measurements of specimen densities by each partner. Density is a key factor in heat transfer by conduction and it must be checked whether the specimens have not been damaged by compression or stretching over their elastic limit during their transportation or by partners and thus remain identical between each measurement. Density measurements also provide information on the degree of homogeneity of the specimen materials. This point must be checked because the comparison protocol assumes that the thermal conductivity of the tested materials remain constant whatever the size of the specimens. In the case of MW35, a homogeneity study, which was done prior to the certification process of the IRMM-440, demonstrated that the thermal conductivity of this mineral wool was the same at a given temperature for specimens having different dimensions and whose density was in the range 64 $kg \cdot m^{-3}$ to 78 $kg \cdot m^{-3}$. In that case, the uncertainty on thermal conductivity measurements due to non-homogeneity of the material was negligible compared to the other sources of uncertainty [7].

Figure 8 plots the relative differences between bulk density measurements and the grand mean value $\bar{\bar{\rho}}_J$ as a function of laboratory. The grand mean value is defined for each specimen (MW35-1, MW35-2, EPS35-1, EPS35-2, EPS70-1, EPS70-2) by equation (2) as the mean value of the I involved laboratories measurements:

$$\bar{\rho} = \frac{1}{I} \sum_i \rho_i \qquad (2)$$

Measured values of ρ_i for each material are displayed in Table 17 and 18 in Appendix 1. It has to be noticed that bulk density measurements performed by PTB on 20 and 25 mm specimens are included in EPS35-1 data set for the analysis. In this report, all the results performed by PTB with 20 mm and 25 mm have been analysed with EPS35 data set.

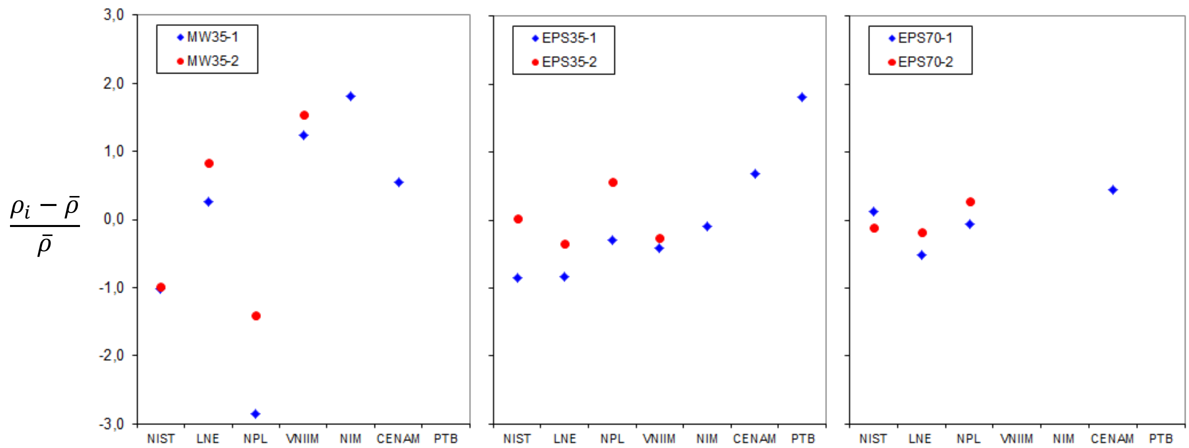


Figure 8 : Relative variation in specimen bulk density versus laboratory

The density measurements for MW35 specimens (a semi-rigid solid) are more dispersed than those carried out on expanded polystyrene (a rigid solid), probably due to difficulties in accurately measuring the dimensions of MW35 specimen because of their compressibility. The relative variations in density of MW35, EPS35 and EPS70 specimens vary respectively from -3 % to +2 %, -0.5 % to +1 % and -0.5 % to +0.5 %. PTB has measured mean values for bulk density of 22.05 kg·m^{-3} and 22.00 kg·m^{-3} for EPS20 and EPS25 specimens.

The values obtained by the participants for MW35 are nevertheless within the density range (64 kg·m^{-3} to 78 kg·m^{-3}) recommended in the certificate of analysis of the IRMM-440. In addition, Figure 8 shows that there is no correlation between the density measurements and the laboratories, because no systematic behaviour appears for a laboratory for all specimens. For example, NPL measured the lowest density for MW35-1 and MW35-2 specimens, and the highest ones for EPS35-2 and EPS70-2.

In conclusion, the bulk density of each specimen, and therefore its thermal conductivity, can be considered as reasonably stable with time whatever the specimen dimensions. Only one case (EPS35-1) seems showing a drift of density values with time or with the laboratories (both factors being correlated).

Specimen stability

In order to investigate potential aging effect, measurements have been performed on the same specimens of each material, with the same equipment and the same operator once they have been brought back to the pilot laboratory (LNE) 40 months after its first measurement.

Table 12 : Additional thermal conductivity measurements for MW35, EPS35 and EPS70

Laboratory	Specimen	Date	T_m [°C]	$\lambda \cdot 10^{-3}$ [W·m^{-1}·K^{-1}]	2010 vs 2007 Rel. Dev [%]	$U(\lambda) \cdot 10^{-3}$ [W·m^{-1}·K^{-1}]
LNE	MW35-1 + MW35-2	May 2007	10.00	30.02	+ 1.07	0.30
		August 2010	10.00	30.34		0.30
		May 2007	23.00	31.54	+ 1.15	0.32
		August 2010	23.00	31.90		0.32
		May 2007	40.00	33.60	+ 0.95	0.34
		August 2010	40.00	33.92		0.34
	EPS35-1 + EPS35-2	May 2007	10.00	30.47	+ 1.15	0.30
		August 2010	10.00	30.82		0.30
		May 2007	23.00	31.90	+ 1.02	0.32
		August 2010	23.00	32.22		0.32
		May 2007	40.00	33.76	+ 1.10	0.34
		August 2010	40.00	34.13		0.34
	EPS70-1 + EPS70-2	May 2007	10.00	30.63	+ 1.04	0.30
		August 2010	10.00	30.95		0.30
		May 2007	23.00	32.02	+ 0.93	0.32
		August 2010	23.00	32.32		0.32
		May 2007	40.00	33.87	+ 1.06	0.34
		August 2010	40.00	34.23		0.34

These additional measurements have been performed on the three pairs of specimens at 10 °C, 23 °C, and 40 °C (one thermal-conductivity measurement per temperature level) at the end of the comparison. The final specimens of 330 mm diameter (dimensions needed by VNIIM, NIM and CENAM) were reassembled with the corresponding cutting scraps, which were retained at LNE, in order to obtain specimens having the required dimensions (610 mm x 610 mm). Table 12 presents the measurements performed by LNE in May 2007 and in August 2010 for each pair of specimens. As these additional measurements were not performed by strictly following the comparison process (only one measurement performed at 23 °C instead of four, and measurements performed on a specimen in a state slightly different than the initial one), the corresponding results were not used in the calculation of the mean thermal conductivity values attributed to LNE in this inter-laboratory comparison.

The mean values obtained by LNE at the end of the comparison are approximately 1 % higher than those obtained three years earlier. The observed long-term variations are small compared to the differences between the results of the different laboratories, and are within the measurement uncertainties of LNE. This might prove the stability of the materials and the specimens.

In order to perform the measurements in August 2010, each specimen has been reassembled with the pieces successively cut for fitting with guarded hot plates of the successive laboratories. For studying the effect of cutting and reassembly, other measurements have been performed by LNE in March 2011 on new EPS and MW specimens obtained from the same batches as those studied in this comparison. These new specimens were measured before and after cutting/reassembly process, in order to quantify the influence of the cutting on the thermal conductivity measurements. The results of these measurements, presented in Table 13, show a systematic increase of the thermal conductivity values ranging from 0.5 % to 1 % depending on the pair of specimens. This could explain a part of the variation observed between thermal conductivity values determined by LNE at the beginning and at the end of the comparison. This fact reinforces the previous conclusion about the generally good stability of the tested specimens in terms of their inherent thermal conductivity.

Table 13 : Effect of the cutting/reassembly process on thermal conductivity results

Laboratory	Specimen	Date	T_m [°C]	$\lambda \cdot 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	Rel. Dev. [%]	$U(\lambda) \cdot 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]
LNE	MW35	Initial state	10.00	30.20	+0.66	0.30
		After cutting	10.00	30.40		0.30
	EPS35	Initial state	10.00	30.56	+0.49	0.30
		After cutting	10.00	30.71		0.30
	EPS70	Initial state	10.00	30.43	+0.95	0.30
		After cutting	10.00	30.72		0.30

2.5.2 Statistical analysis procedure

After preparation of the data for each participant, the results are compared. The methodology recommended by the Consultative Committee on Photometry and Radiometry (CCPR) Working Group on Key Comparisons (WG KG) [10][11] has been employed using weighted mean averaging with cut-off to obtain a nominal Comparison Reference Value (CRV) λ_{CRV} for each material and temperature, given by equation (3). In the weighted mean calculation, each participant's values are weighted by their quoted standard uncertainties down to a cut-off point $u_{cut-off}$, which is given by the average of the standard uncertainties less than or equal to the median of all the standard uncertainties, given by equation (4).

$$\lambda_{CRV} = \frac{\sum_i \lambda_i u_{adj,i}^{-2}}{\sum_i u_{adj,i}^{-2}} \tag{3}$$

Where

$$\begin{cases} u_{adj,i} = u(\lambda_i)\ for\ u(\lambda_i) \geq u_{cut-off} \\ u_{adj,i} = u_{cut-off}\ for\ u(\lambda) < u_{cut-off} \\ u_{cut-off} = average\{u(\lambda_i)\}\ for\ u(\lambda_i) \leq median\{u(\lambda_j)\} \end{cases} \tag{4}$$

The uncertainty for the CRV is given by equation (5):

$$u(\lambda_{\mathrm{CRV}}) = \frac{\sqrt{\sum_{\mathrm{i}} \frac{u^2(\lambda_i)}{u_{\mathrm{adj}}^4(\lambda_i)}}}{\sum_{\mathrm{i}} u_{adj}^{-2}(\lambda_i)} \tag{5}$$

The individual participant's results are then compared to the λ_{CRV} values. The relative deviations from the λ_{CRV}, D_i, and their associated uncertainties, U_{i} are the unilateral Degrees of Equivalence (DoE), which are calculated according to equations (6) and (7):

$$D_{\mathrm{i}} = \frac{\lambda_i - \lambda_{\mathrm{CRV}}}{\lambda_{\mathrm{CRV}}} \tag{6}$$

$$U_{\mathrm{i}} = \lambda_{\mathrm{CRV}}^{-1} \cdot k(=2) \cdot \sqrt{u^2(\lambda_i) + u^2(\lambda_{\mathrm{CRV}}) + u_{\mathrm{add}}^2 - 2 \cdot \frac{\frac{u^2(\lambda_i)}{u_{\mathrm{adj}}^2(\lambda_i)}}{\sum_i u_{\mathrm{adj}}^{-2}(\lambda_i)}} \tag{7}$$

where the additional uncertainty component u_{add} is used to account for the drift due to the specimen stability. For each material and temperature, u_{add} is calculated with equations (8) by using values of Table 23:

$$u_{add} = \frac{\lambda_{August\ 2010} - \lambda_{May\ 2007}}{\sqrt{3}} \tag{8}$$

The error function is defined by equation (9) as:

$$E_{\mathrm{i}} = \frac{|D_{\mathrm{i}}|}{U_{\mathrm{i}}} \tag{9}$$

Any $E_\mathrm{i} \leq 1$ is considered as a consistent value. For $E_\mathrm{i} > 1$, a Chi-square consistency check is performed. If the test fails with a level of confidence of 1 % (i.e. if the associated p-value is lower than 0.01), then the corresponding data from the laboratory with the highest E_i (named $max\{E_i\}$ in the rest of the report) are removed and the DoE are recalculated.

The observed chi-squared is calculated as:

$$\chi^2_{obs} = \sum_i \frac{(\lambda_i - \lambda_{\mathrm{CRV}})^2}{u^2_{\mathrm{adj}}(\lambda_i)} \tag{10}$$

This process is summarized in Figure 9.

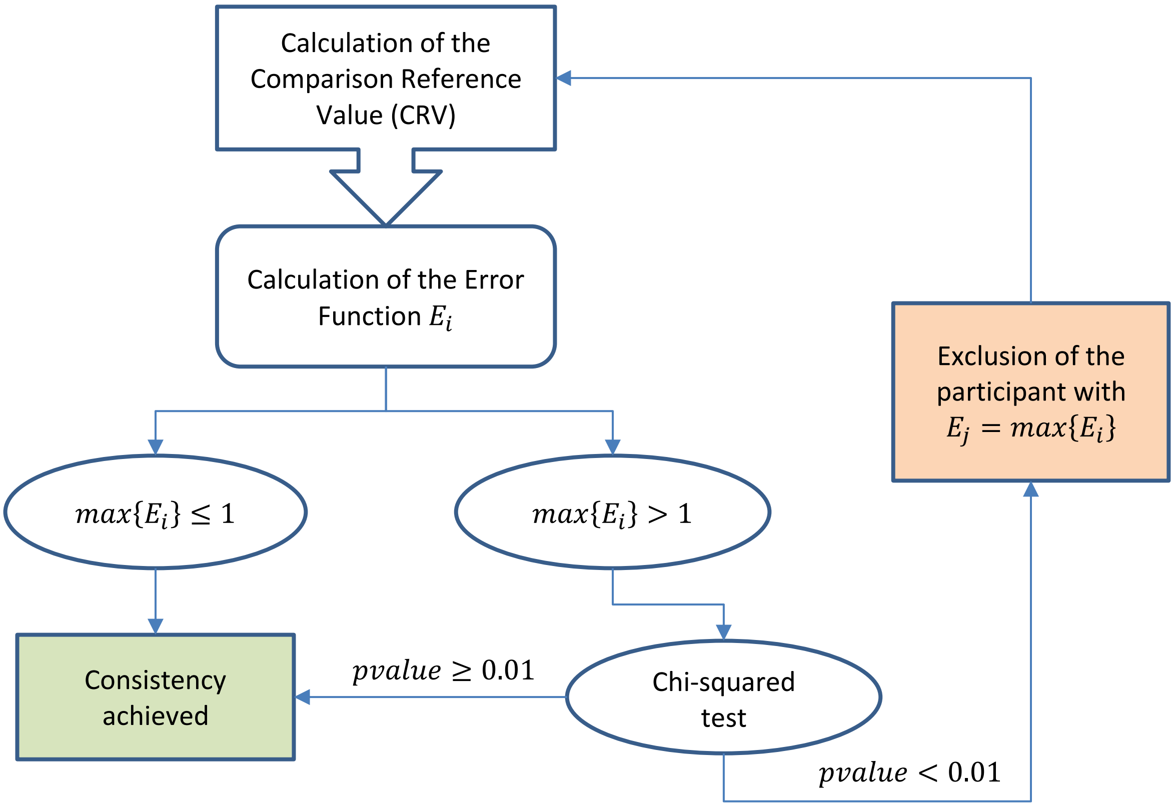


Figure 9 : Flowchart of the procedure for excluding inconsistent data

3 Results

3.1 Summary of the statistical procedure

Table 14 summarizes the successive iterations of the statistical procedure. No outlier is identified for MW35 and EPS70. For EPS35 at 23 °C, it was required to exclude NIM and VNIIM to reach consistency and at 40 °C, it was required to exclude NIM and PTB.

Table 14 : Iterations of the statistical procedure for MW35, EPS35 and EPS70 at 10 °C, 23 °C and 40 °C

Material	*Temp. (°C)*	*Iteration 1*	*Iteration 2*	*Iteration 3*
MW35	10	$max\{E_i\} = 1.03$ χ^2 test ✔		
	23	$max\{E_i\} = 0.87$ ✔		
	40	$max\{E_i\} = 0.81$ ✔		
EPS35	10	$max\{E_i\} = 0.97$ ✔		
	23	$max\{E_i\} = 3.15$ χ^2 test ✖ ⇨ NIM is excluded	$max\{E_i\} = 1.18$ χ^2 test ✖ ⇨ VNIIM is excluded	$max\{E_i\} = 1.00$ ✔
	40	$max\{E_i\} = 2.65$ χ^2 test ✖ ⇨ NIM is excluded	$max\{E_i\} = 1.16$ χ^2 test ✖ ⇨ PTB is excluded	$max\{E_i\} = 0.83$ ✔
EPS70	10	$max\{E_i\} = 0.39$ ✔		
	23	$max\{E_i\} = 0.78$ ✔		
	40	$max\{E_i\} = 0.67$ ✔		

3.2 Comparison reference value (CRV)

For each of the nine configurations (3 materials x 3 temperatures), the Comparison Reference Values have been calculated from equations (3) and (4). Results are displayed in Table 15.

Table 15 : Comparison Reference Values of thermal conductivity

Specimen	λ_{CRV} 10^{-3} [W·m^{-1}·K^{-1}] at 10 °C	at 23 °C	at 40 °C
MW35	30.28	31.81	33.83
EPS35	30.76	32.03	34.16
EPS70	30.73	32.20	34.10

As expected, Comparison Reference Values of thermal conductivity increase with temperature. Thermal conductivity values for EPS35 and EPS70 are very similar whatever the temperature. This indicates that the sensitivity of thermal conductivity to the thickness of specimen is weak or negligible here for measurements performed for expanded polystyrene.

3.3 Raw results

In this section, the raw results are reported on the graphs for all participants. They are compared with the CRV provided in Section 3.2.

3.3.1 Mineral wool

Measurements on mineral wool have been performed by 6 of 7 laboratories. Five of these six laboratories have carried out tests at 10 °C, 23 °C and 40 °C. At 10 °C and 40 °C conditions, only one measurement has been done whereas 4 measurements were carried out at 23 °C by each partner. Figure 10 presents the values of thermal conductivity obtained by the participating laboratories at 10 °C and 40 °C, and the mean values calculated at 23 °C from the 4 repeated measurements. The associated bars represent the expanded uncertainty U determined from the adjusted standard uncertainties obtained from equation (4) with a coverage factor $k = 2$ (cf. equation 11). For each temperature, the red line identifies the CRV.

$$U = k \times u_{adj,i} \tag{11}$$

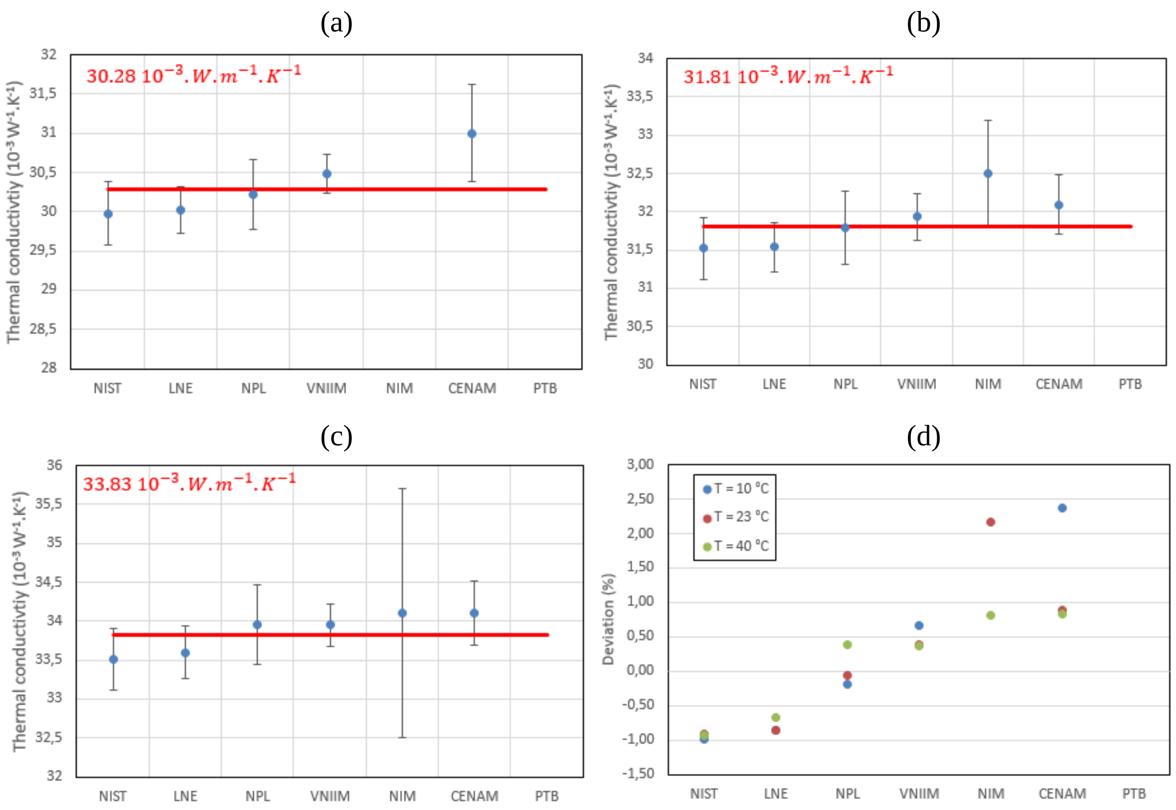


Figure 10 : Thermal conductivity measurements of MW35
(a) 10 °C (b) 23 °C (c) 40 °C (d) Relative difference for each temperature

The CRV for each temperature have been estimated respectively to 30.28 10^{-3} $W{\cdot}m^{-1}{\cdot}K^{-1}$ (10 °C), 31.81 10^{-3} $W{\cdot}m^{-1}{\cdot}K^{-1}$ (23 °C) and 33.83 10^{-3} $W{\cdot}m^{-1}{\cdot}K^{-1}$ (40 °C). Whatever the temperature and the laboratory, the discrepancy to the CRV remains between -1.5 % and +2.5 %. Figure 10-d shows that the behaviour of the laboratories does not change from one temperature to another, since the relative position of each laboratory (for both mean values and dispersions) is constant on the three graphs.

3.3.2 Expanded polystyrene

Measurements have been performed on expanded polystyrene at 23 °C and 40 °C by all laboratories. Four laboratories have carried out the measurements with 35 mm and 70 mm thick materials. Only one measurement has been done at 10 °C and 40 °C, whereas 4 measurements were carried out at 23 °C by each partner.

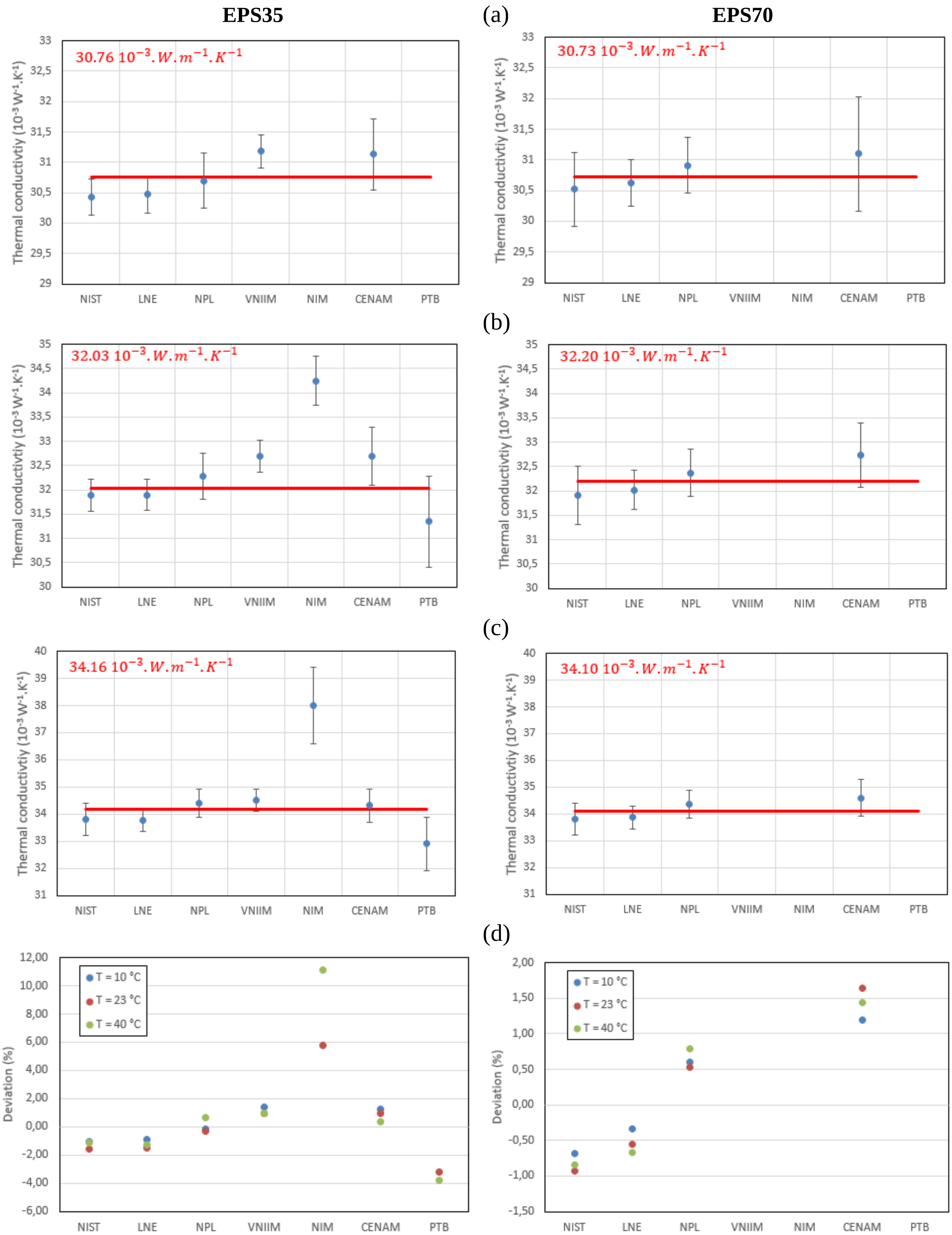


Figure 11 : Thermal conductivity measurements of EPS35 and EPS70
(a) 10 °C (b) 23 °C (c) 40 °C (d) relative deviation for each temperature

Figure 11 presents the raw values of thermal conductivity obtained by the partners on EPS35 and EPS70 at 10 °C and 40 °C, and the mean values calculated at 23 °C from the 4 repeated measurements. The associated bars represent the expanded uncertainty U determined from the adjusted standard uncertainties obtained from equation (4) with a coverage factor k = 2 (cf. equation 11). The CRV calculated from equation (3) is used to determine how spread the results obtained by each partner are.

The CRV for the two thicknesses (35 mm and 70 mm) at each temperature (10 °C, 23 °C and 40 °C) are close to each other. The differences between the values obtained for the two thicknesses are lower than 0.55 %.

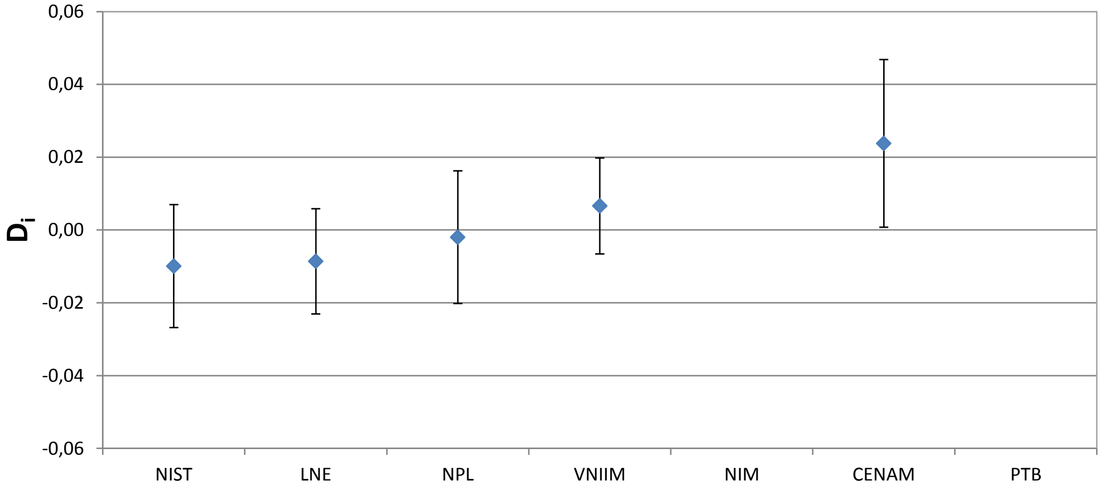
For a given temperature and a given thickness, the measurement results which have been identified as consistent (cf. table 14) vary from -2.1 % to +2.4 % around the CRV. The relative difference to a CRV for a laboratory is not a random value: NIM and CENAM, for instance, always provide values higher than the CRV. For each partner, it means that the discrepancy to the CRV is a systematic error probably due to the equipment or operations procedures.

3.4 Degree of equivalence

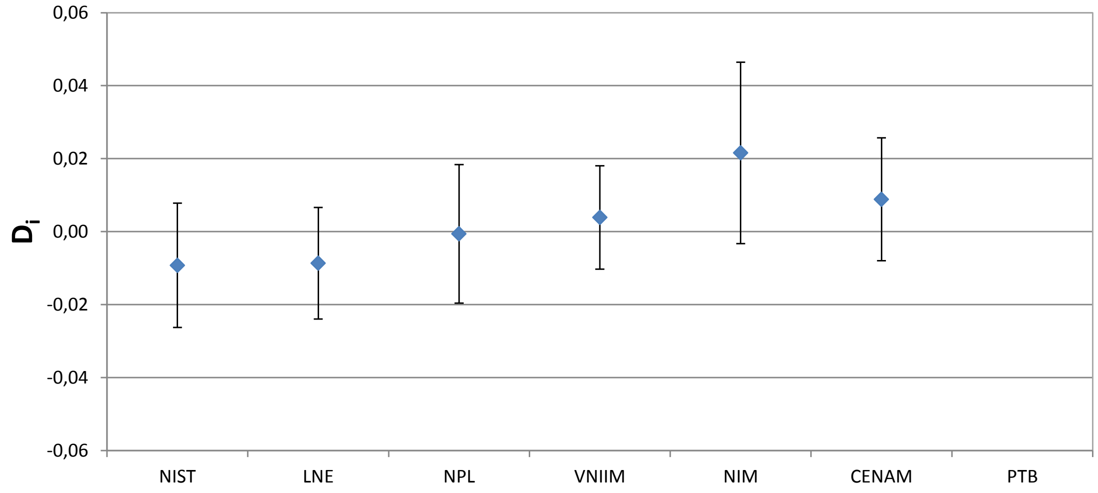
The degrees of equivalence have been calculated for each configuration by using equation (6), taking into consideration only the measurement results considered as consistent. Results for MW35, EPS35 and EPS70 are respectively shown on Figure 12, Figure 13 and Figure 14.

- For MW35, the degrees of equivalence vary between -0.01 and +0.025 (without error bars). The error bars increase this range to -0.04 to + 0.05. The measurement values are therefore very close to λ_{CRV}.
- For EPS35, the degrees of equivalence varies between -0.025 and +0.021 (without error bars). The error bars increase this range to -0.06 to + 0.04. The measurement values are therefore very close to λ_{CRV}.
- For EPS70, the degrees of equivalence vary between -0.01 and +0.016 (without error bars). The error bars increase this range to -0.03 to + 0.045. The measurement values are thus very close to λ_{CRV}.

(a)



(b)



(c)

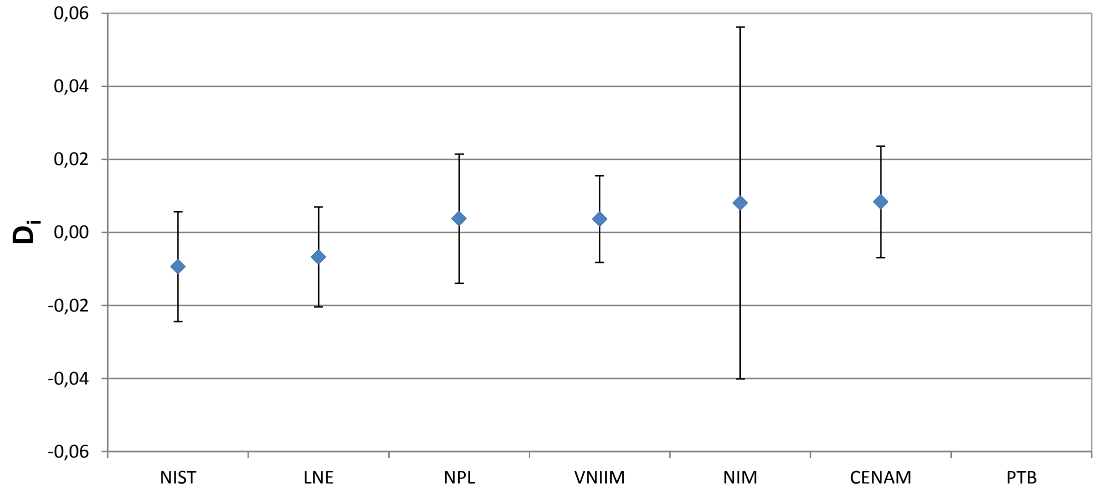
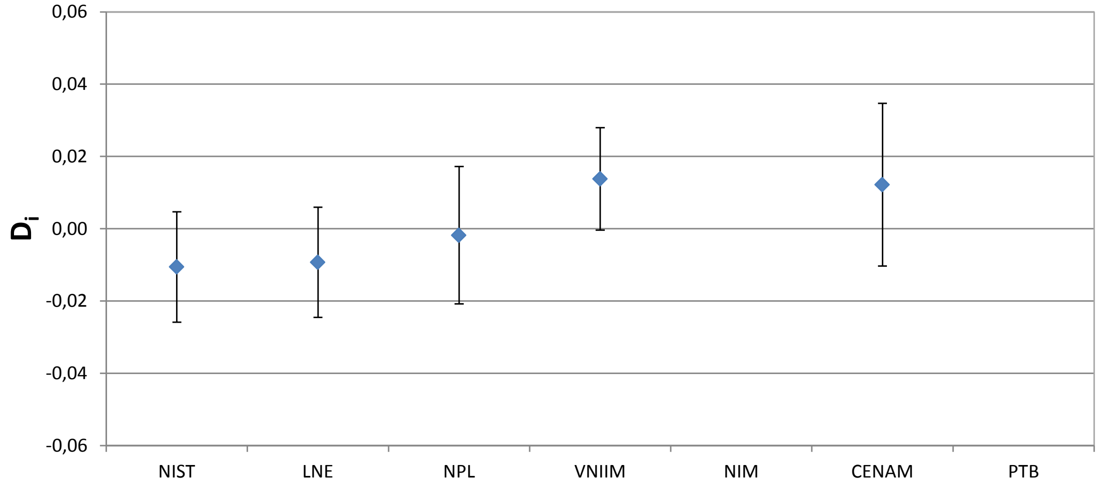
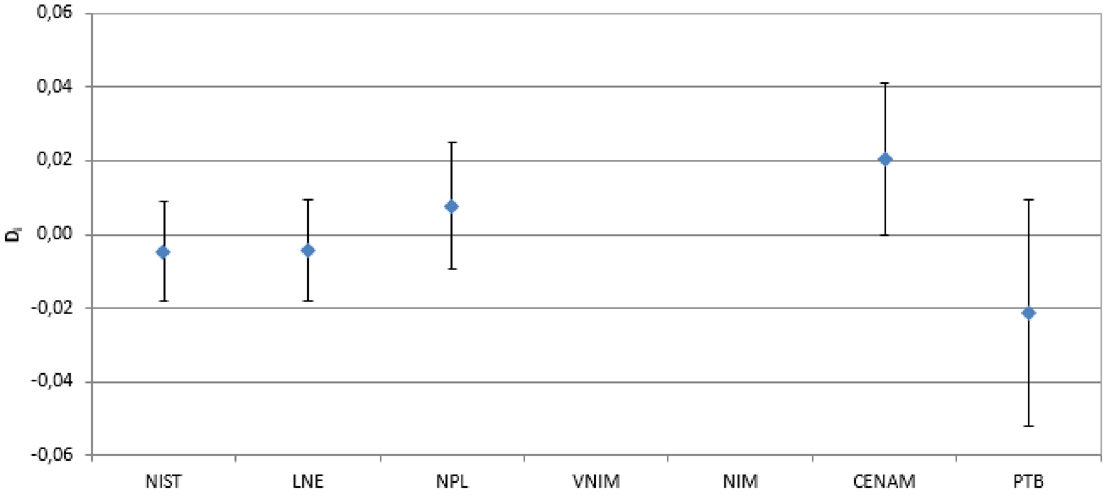


Figure 12 : Degrees of Equivalence for MW35 at (a) 10 °C (b) 23 °C and (c) 40 °C

(a)



(b)



(c)

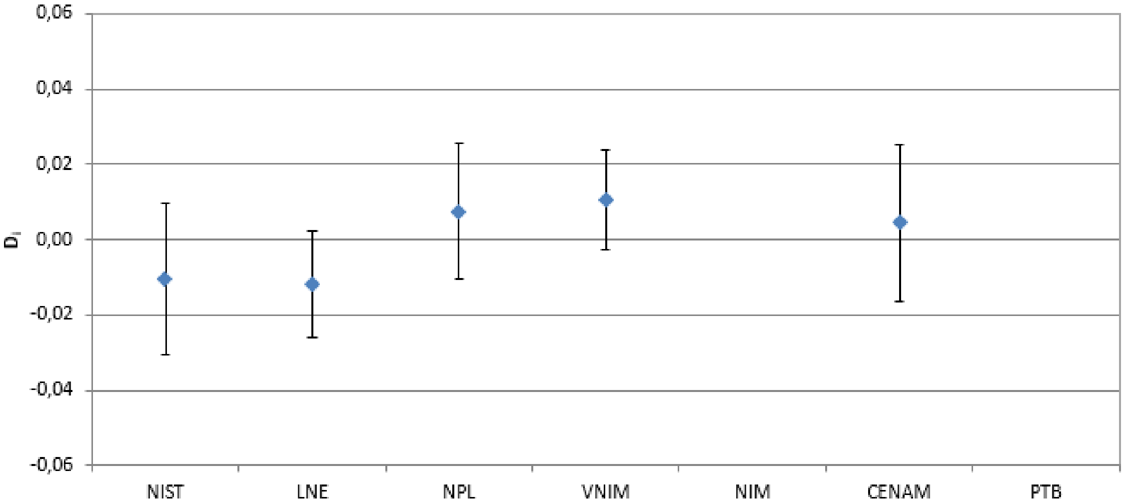
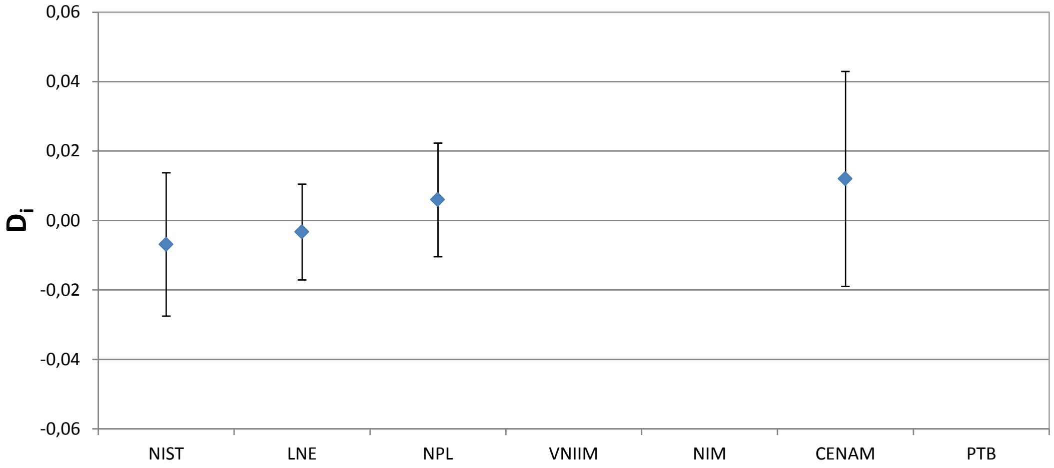
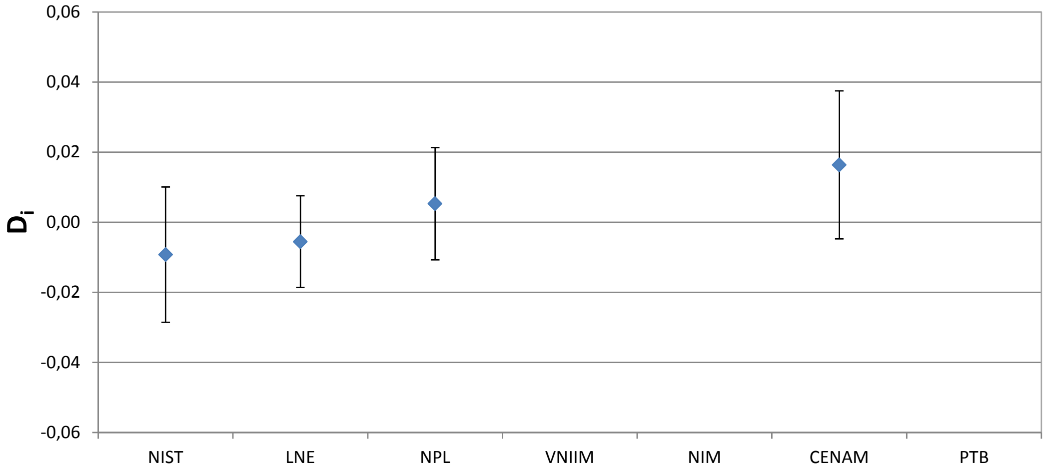


Figure 13 : Degrees of Equivalence for EPS35 respectively at 10 °C, 23 °C and 40 °C

(a)



(b)



(c)

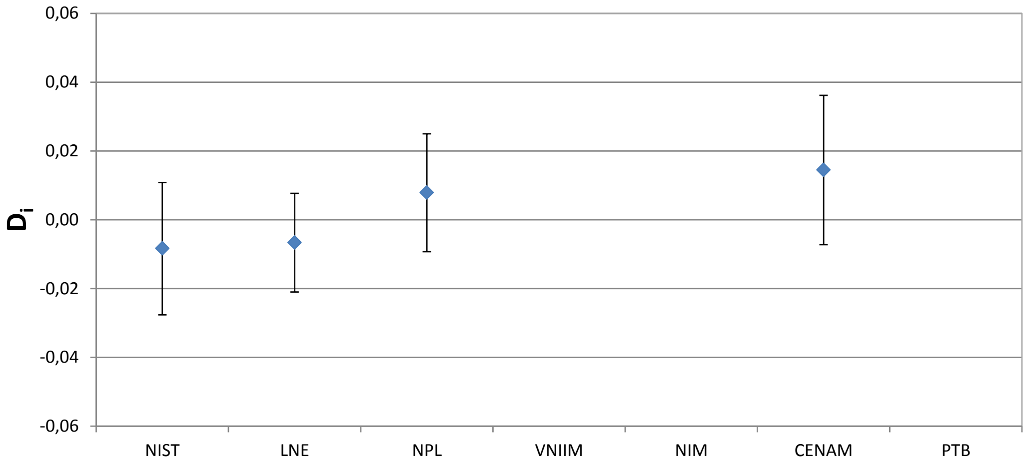


Figure 14 : Degrees of Equivalence for EPS70 respectively at 10 °C, 23 °C and 40 °C

3.5 Error function results

In order to analyse the DoE presented in the previous section, the error functions given by equation (9) have been calculated. The error function is a ratio between the absolute value of the relative deviations D_i and the associated expanded uncertainty U_i. The values of error function are plotted in Figure 15 to Figure 17 for MW35, EPS35 and EPS70 respectively and the corresponding values are summarized in Table 16.

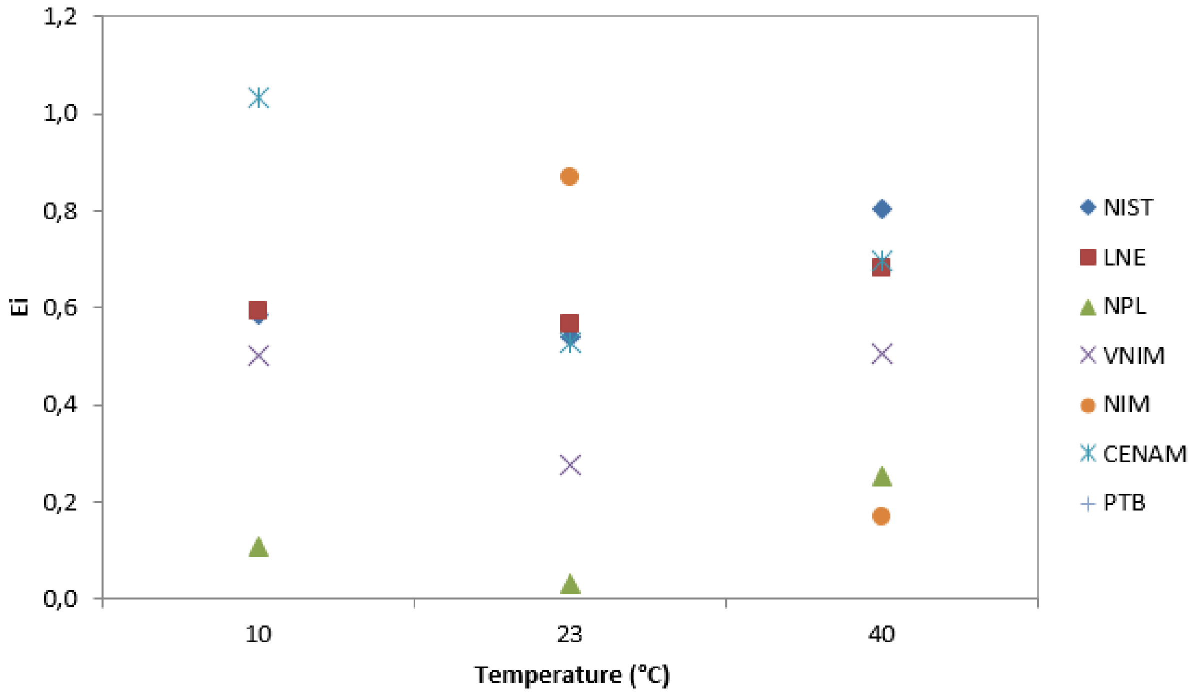


Figure 15 : Error function for MW35

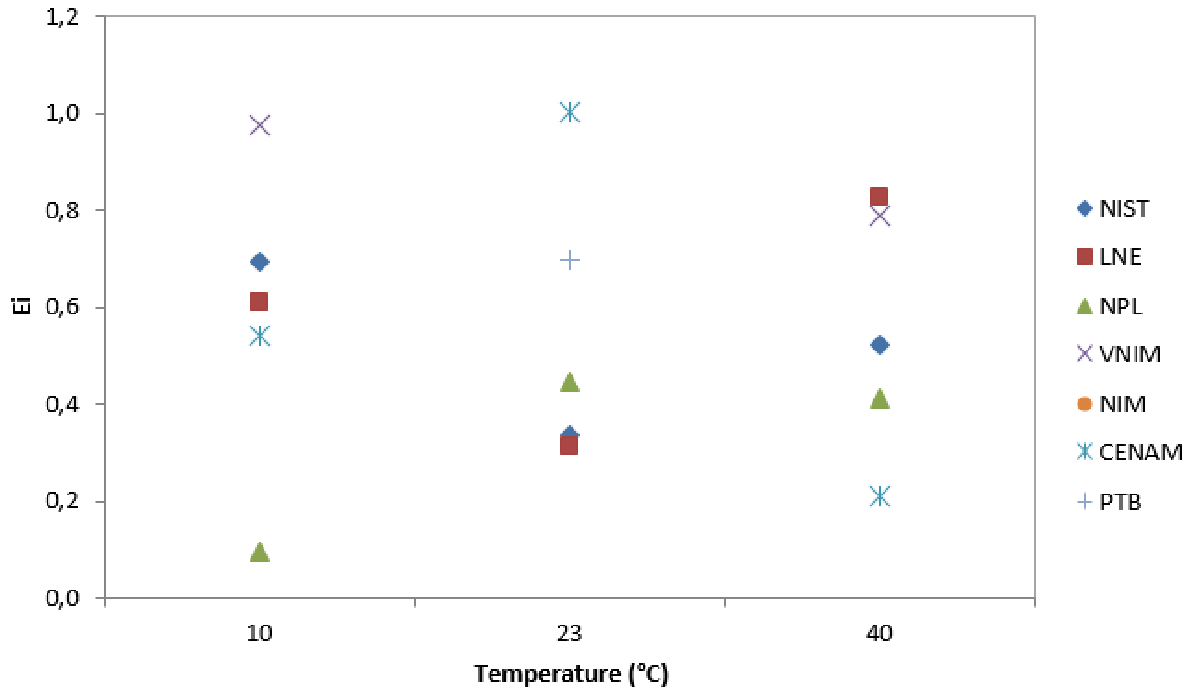


Figure 16 : Error function for EPS35

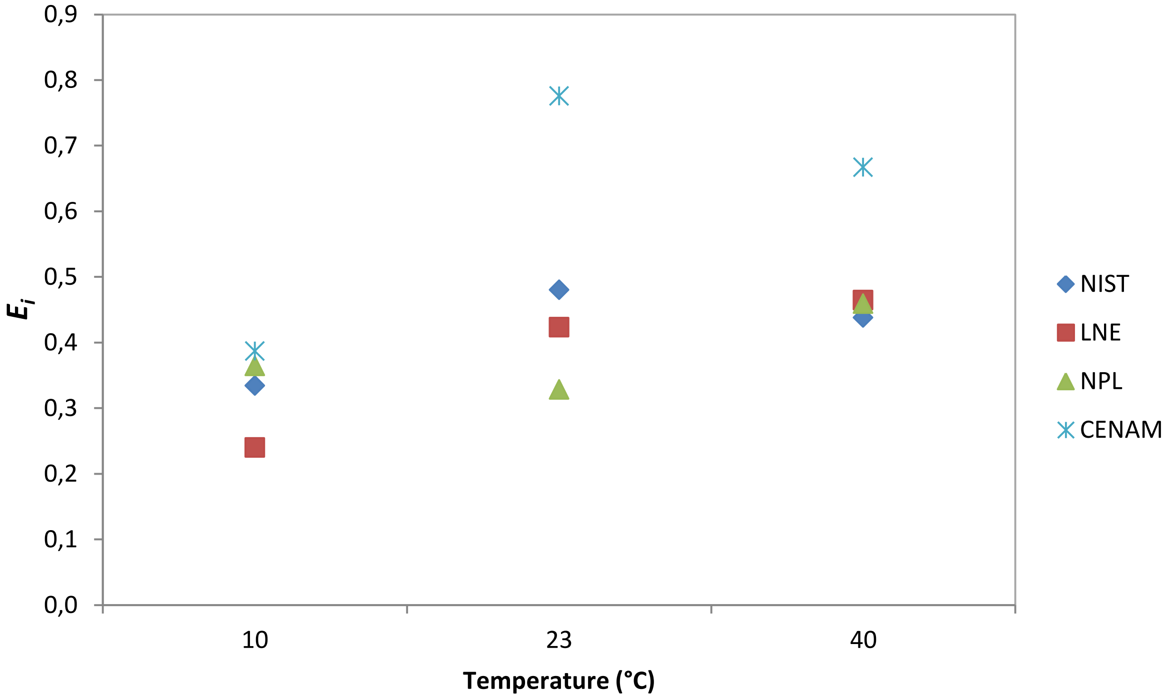


Figure 17 : Error function for EPS70

Graphs of error functions show that there is no correlation between temperature and error functions: no particular trend is visible. Error functions are rather well spread between the partners. There is no ordering between the partners with one having low E_i and another having always the highest E_i. It means that the partners have roughly the same ability to measure the thermal conductivity whatever the dimensions (metering area, thickness), the number of specimens per apparatus.

Table 16 : Summary of all error function results

Laboratory	MW35			EPS35			EPS70		
	10 °C	23 °C	40 °C	10 °C	23 °C	40 °C	10 °C	23 °C	40 °C
NIST	0.587	0.541	0.805	0.693	0.337	0.524	0.334	0.480	0.438
LNE	0.594	0.567	0.683	0.608	0.311	0.827	0.240	0.423	0.464
NPL	0.109	0.030	0.252	0.095	0.445	0.411	0.364	0.328	0.459
VNIIM	0.500	0.276	0.504	0.974		0.789			
NIM		0.869	0.170						
CENAM	1.032	0.527	0.698	0.542	1.000	0.212	0.387	0.776	0.667
PTB					0.698				

The statistical procedure here applied enables to get consistent data and to calculate the error function values in Table 16. It can be observed that participants have only been considered as outliers for EPS35.

4 Conclusion

This CCT supplementary comparison investigated the agreement and the variability in thermal conductivity measurements performed by seven NMIs by using the Guarded Hot Plate method. All laboratories (except for PTB) measured the same sets of specimens of insulating materials (mineral wool and expanded polystyrene) in order to avoid any potential influence of specimen heterogeneity from sampling.

The results obtained at 10 °C, 23 °C and 40 °C on the three pairs of specimens (MW35, EPS35 and EPS70) indicate that there is no laboratory-material interaction. It has been also demonstrated that the deviations observed in few cases between thermal conductivity results obtained by the partners cannot be ascribed to density variations or to an ageing phenomena.

The analysis of the results indicated good agreement between the NMIs considering the relative deviations and their uncertainties. From the interpretation of the error functions, only four values (NIM and VNIIM for EPS35 at 23 °C and NIM and PTB for EPS35 at 40 °C) among 48 were considered to be 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 thermal conductivity measurement by GHP apparatus.

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Appendix 1 – Tables of bulk density measurements

Table 17 : Physical characteristics of MW35 specimens determined by the laboratories

Laboratory	Specimen reference	Thickness [mm]	Diam./length [mm]	Width [mm]	Weight [g]	Density [$kg \cdot m^{-3}$]
NIST	MW35-1	35.50	1015	/	2018.6	70.3
	MW35-2	35.40	1015	/	2028.0	70.8
	mean value					70.6
LNE	MW35-1	34.50	612	611	917.0	71.2
	MW35-2	34.58	612	611	931.0	72.1
	mean value					71.7
NPL	MW35-1	35.50	612	612	916.9	69.0
	MW35-2	35.29	611	612	930.6	70.5
	mean value					69.8
VNIIM	MW35-1	34.50	330	/	211.9	71.9
	MW35-2	34.50	330	/	214.0	72.6
	mean value					72.3
NIM	MW35-1	34.23	330	/	211.7	72.3
CENAM	MW35-1	34.62	330	/	211.6	71.4
Grand mean $\bar{\bar{\rho}}$ [$kg \cdot m^{-3}$]	MW35-1					71.0
	MW35-2					71.5
Grand stand. dev. [$kg \cdot m^{-3}$]	MW35-1					1.2
	MW35-2					1.0
Relative grand SD [%]	MW35-1					1.7
	MW35-2					1.4
Range [$kg \cdot m^{-3}$]	MW35-1					3.3
	MW35-2					2.1

Table 18 : Physical characteristics of EPS35 and EPS70 specimens

Laboratory	Specimen reference	Thickness [mm]	Diam./length [mm]	Width [mm]	Weight [g]	Density [kg·m^{-3}]
NIST	EPS35-1	35.10	1015	/	608.8	21.44
	EPS35-2	34.90	1015	/	615.7	21.80
	mean value					21.62
	EPS70-1	69.80	1015	/	1219.6	21.59
	EPS70-2	69.90	1015	/	1248.6	22.08
	mean value					21.84
LNE	EPS35-1	34.98	611	611	280.0	21.44
	EPS35-2	34.84	612	611	283.0	21.72
	mean value					21.58
	EPS70-1	69.77	611	610	558.0	21.46
	EPS70-2	69.70	611	611	574.0	22.06
	mean value					21.76
NPL	EPS35-1	34.72	611	611	279.4	21.56
	EPS35-2	34.40	612	612	282.4	21.92
	mean value					21.74
	EPS70-1	69.35	611	611	558.0	21.55
	EPS70-2	69.29	611	611	573.2	22.16
	mean value					21.86
VNIIM	EPS35-1	34.86	330	/	64.2	21.55
	EPS35-2	34.74	330	/	64.6	21.74
	mean value					21.64
NIM	EPS35-1	34.75	330	/	64.2	21.60
CENAM	EPS35-1	34.55	330	/	64.2	21.77
	EPS70-1	69.36	330	/	128.3	21.66
PTB	EPS20-2	19.98	100	/	3.469	22.00
	EPS20-3	20.35	101	/	3.603	22.10
	mean value					22.00
	EPS25-1	25.12	100	/	4.363	22.00
	EPS25-2	25.20	100	/	4.375	22.00
	mean value					22.00
Grand mean $\overline{\overline{\rho}}$ [kg·m^{-3}]	EPS35-1					21.56
	EPS35-2					21.80
	EPS70-1					21.57
	EPS70-2					22.10
Grand stand. dev. [kg·m^{-3}]	EPS35-1					0.12
	EPS35-2					0.09
	EPS70-1					0.09
	EPS70-2					0.05
Relative grand SD [%]	EPS35-1					0.57
	EPS35-2					0.40
	EPS70-1					0.40
	EPS70-2					0.24
Range [kg·m^{-3}]	EPS35-1					0.33
	EPS35-2					0.20
	EPS70-1					0.20
	EPS70-2					0.10

Appendix 2 – Tables of thermal conductivity measurements

Table 19 : Repeated measurements at 23 °C for MW35 specimens

Lab.	Specimen	T_m [°C]	e [mm]	T_H [°C]	T_C [°C]	A [m^2]	φ [W·m^{-2}]	R_{th} $^{(*)}$ [m^2·K·W^{-1}]	$\lambda \cdot 10^{-3}$ [W·m^{-1}·K^{-1}]	U(λ)$\cdot 10^{-3}$ [W·m^{-1}·K^{-1}]
NIST	MW35-1 + MW35-2	23.00	34.51	33.00	13.00	0.1297	18.28	1.090	31.54	0.40
		23.00	34.52	33.00	13.00	0.1297	18.27	1.090	31.52	0.40
		23.00	34.51	33.00	13.00	0.1297	18.27	1.090	31.52	0.40
		23.00	34.51	33.00	13.00	0.1297	18.26	1.100	31.50	0.40
LNE	MW35-1 + MW35-2	23.09	34.54	33.17	13.01	0.0900	18.38	1.097	31.50	0.32
		23.00	34.54	33.00	12.99	0.0900	18.32	1.093	31.61	0.32
		22.99	34.54	32.95	13.02	0.0900	18.20	1.095	31.54	0.32
		22.90	34.54	32.91	12.89	0.0900	18.26	1.096	31.50	0.32
NPL	MW35-1	22.90	34.58	32.90	12.90	0.0930	18.39	1.087	31.81	0.48
		23.00	34.64	32.90	13.00	0.0930	18.29	1.089	31.80	0.48
		23.10	34.62	33.00	13.10	0.0930	18.31	1.089	31.79	0.48
		23.00	34.66	33.00	13.00	0.0930	18.33	1.091	31.78	0.48
	MW35-2	23.00	34.48	32.90	13.00	0.0930	18.38	1.083	31.84	0.48
VNIIM	MW35-1 + MW35-2	23.03	34.50	33.05	13.02	0.0177	18.54	1.080	31.93	0.20
		23.05	34.50	33.04	13.05	0.0177	18.54	1.080	31.93	0.20
		23.01	34.50	33.00	13.01	0.0177	18.55	1.080	31.95	0.20
		23.03	34.50	33.04	13.02	0.0177	18.54	1.080	31.94	0.20
NIM	MW35-1	23.28	34.25	32.99	13.57	0.0314	18.58	1.048	32.70	0.70
		23.24	34.21	32.93	13.56	0.0314	18.32	1.059	32.30	0.70
		23.26	34.20	32.95	13.56	0.0314	18.62	1.043	32.80	0.70
		23.24	34.20	32.94	13.54	0.0314	18.29	1.062	32.20	0.70
CENAM	MW35-1 + MW35-2	23.30	34.50	33.70	13.00	0.0214	38.50	1.070	32.13	0.39
		23.20	34.50	33.40	13.00	0.0214	37.80	1.070	31.97	0.38
		23.20	34.50	33.40	13.00	0.0214	37.80	1.070	32.09	0.39
		23.20	34.50	33.30	13.00	0.0214	37.80	1.070	32.19	0.39

$^{(*)}$ $R_{th} = (T_H - T_C)/\varphi$

Table 20 : Repeated measurements at 23 °C for EPS35 and EPS70 specimens

Lab.	Specimen	T_m [°C]	e [mm]	T_H [°C]	T_C [°C]	A [m^2]	φ [W·m^{-2}]	R_{th} (*) [m^2·K·W^{-1}]	$\lambda\cdot10^{-3}$ [W·m^{-1}·K^{-1}]	U(λ)$\cdot10^{-3}$ [W·m^{-1}·K^{-1}]
NIST	EPS35-1 + EPS35-2	23.01	35.08	33.00	13.01	0.1297	18.18	1.100	31.88	0.30
		23.00	35.08	33.00	13.00	0.1297	18.19	1.100	31.90	0.30
		23.00	35.07	33.00	13.00	0.1297	18.18	1.100	31.89	0.30
		23.00	35.07	33.00	13.00	0.1297	18.18	1.100	31.87	0.30
	EPS70-1 + EPS70-2	23.00	70.00	33.00	13.00	0.1297	9.12	2.190	31.90	0.60
		23.00	69.99	33.00	13.00	0.1297	9.12	2.190	31.90	0.60
		23.00	69.98	33.00	13.00	0.1297	9.12	2.190	31.91	0.60
		23.00	69.96	33.00	13.00	0.1297	9.12	2.190	31.90	0.60
LNE	EPS35-1 + EPS35-2	22.97	34.89	33.00	12.94	0.0900	18.35	1.094	31.88	0.32
		23.02	34.89	33.00	13.04	0.0900	18.26	1.093	31.91	0.32
		23.02	34.89	33.03	13.00	0.0900	18.32	1.093	31.92	0.32
		23.02	34.89	33.04	12.99	0.0900	18.31	1.095	31.87	0.32
	EPS70-1 + EPS70-2	22.98	69.73	33.01	12.95	0.0900	9.21	2.178	32.02	0.32
		22.99	69.73	33.02	12.95	0.0900	9.21	2.179	32.01	0.32
		23.01	69.73	32.98	13.03	0.0900	9.17	2.176	32.04	0.32
		22.98	69.73	32.96	12.99	0.0900	9.17	2.178	32.02	0.32
NPL	EPS35-1	22.95	34.98	32.90	13.00	0.0930	18.34	1.084	32.29	0.48
		22.95	34.82	32.90	13.00	0.0930	18.47	1.079	32.26	0.48
		22.95	34.98	32.90	13.00	0.0930	18.33	1.083	32.29	0.48
		22.95	34.94	32.90	13.00	0.0930	18.38	1.083	32.27	0.48
	EPS35-2	23.00	34.70	33.00	13.00	0.0930	18.57	1.073	32.33	0.48
	EPS70-1	22.75	69.52	32.90	12.60	0.0930	9.43	2.151	32.33	0.48
		22.80	69.53	33.00	12.60	0.0930	9.48	2.147	32.39	0.49
		22.75	69.51	32.90	12.60	0.0930	9.47	2.148	32.36	0.49
		22.85	69.53	33.10	12.60	0.0930	9.52	2.146	32.40	0.49
	EPS70-2	22.85	69.18	33.00	12.70	0.0930	9.51	2.140	32.32	0.48
VNIIM	EPS35-1 + EPS35-2	23.06	34.80	33.12	13.00	0.0177	18.91	1.065	32.69	0.20
		23.06	34.80	33.13	13.00	0.0177	18.93	1.064	32.71	0.20
		23.08	34.80	33.15	13.00	0.0177	18.94	1.064	32.70	0.20
		23.08	34.80	33.15	13.00	0.0177	18.94	1.065	32.69	0.20
NIM	EPS35-1	23.30	34.70	33.06	13.55	0.0314	19.19	1.015	34.20	0.50
		23.36	34.62	33.21	13.52	0.0314	19.31	1.018	34.00	0.50
		23.40	34.67	33.17	13.63	0.0314	19.33	1.011	34.30	0.50
		23.39	34.67	33.21	13.58	0.0314	19.50	1.005	34.50	0.50
CENAM	EPS35-1 + EPS35-2	22.60	34.55	32.10	13.10	0.0214	17.90	1.050	32.78	0.59
		22.90	34.55	32.80	13.00	0.0214	18.90	1.040	33.08	0.60
		22.95	34.55	32.90	13.00	0.0214	18.90	1.050	32.84	0.59
		22.90	34.55	33.10	12.70	0.0214	18.90	1.070	32.06	0.58
	EPS70-1 + EPS70-2	22.90	69.36	32.80	13.00	0.0214	9.40	2.110	32.83	0.66
		22.90	69.36	32.70	13.10	0.0214	9.20	2.130	32.51	0.65
		22.70	69.36	32.40	13.00	0.0214	9.20	2.110	32.91	0.66
		22.80	69.36	32.60	13.00	0.0214	9.20	2.120	32.66	0.65

(*) $R_{th} = (T_H - T_C)/\varphi$

Table 20 (continued): Repeated measurements at 23 °C for EPS20 specimens

Lab.	Specimen	T_m [°C]	e [mm]	T_H [°C]	T_C [°C]	A (**) [m²]	φ [W·m⁻²]	R_{th} (*) [m²·K·W⁻¹]	$\lambda \cdot 10^{-3}$ [W·m⁻¹·K⁻¹]	$U(\lambda) \cdot 10^{-3}$ [W·m⁻¹·K⁻¹]
PTB	EPS20-2	23.00	19.98	33.00	13.00		33.28	0.603	33.10	0.99
		23.00	19.98	33.00	13.00		33.44	0.600	33.30	1.00
		23.00	19.98	33.00	13.00		32.82	0.610	32.70	0.98
		23.00	19.98	33.00	13.00		33.10	0.602	33.30	1.00
	EPS20-3	23.01	20.35	33.00	13.01		31.40	0.643	31.40	0.94
		23.01	20.35	33.00	13.01		31.40	0.644	31.30	0.94
		23.01	20.35	33.00	13.01		32.10	0.630	32.00	0.96
		23.00	20.35	33.00	13.00		31.30	0.645	31.30	0.94
	EPS25-1	23.00	25.12	33.00	13.00		24.30	0.825	30.40	0.91
		23.00	25.12	33.00	13.00		24.30	0.826	30.30	0.91
		23.00	25.12	33.00	13.00		24.20	0.829	30.20	0.91
		23.00	25.12	33.00	13.01		24.80	0.808	31.00	0.93
	EPS25-2	22.98	25.20	33.00	12.97		24.20	0.830	30.30	0.91
		22.99	25.20	33.00	12.98		23.40	0.856	29.40	0.88
		22.99	25.20	33.00	12.99		24.70	0.812	31.00	0.93
		23.00	25.20	33.00	13.00		24.40	0.822	30.60	0.92

(*) $R_{th} = (T_H - T_C)/\varphi$
(**) *Not reported*

Table 21 : Measurements at 10 °C and 40 °C for MW35 specimens

Lab.	Specimen	T_m [°C]	e [mm]	T_H [°C]	T_C [°C]	A [m²]	φ [W·m⁻²]	R_{th} (*) [m²·K·W⁻¹]	$\lambda \cdot 10^{-3}$ [W·m⁻¹·K⁻¹]	$U(\lambda) \cdot 10^{-3}$ [W·m⁻¹·K⁻¹]
NIST	MW35-1 MW35-2	10.00	34.51	20.00	0.01	0.1297	17.38	1.150	29.98	0.40
		40.00	34.52	50.00	30.00	0.1297	19.40	1.030	33.51	0.40
LNE	MW35-1 MW35-2	10.03	34.54	19.98	0.07	0.0900	17.31	1.151	30.02	0.30
		40.02	34.54	50.03	30.01	0.0900	19.48	1.028	33.60	0.34
NPL	MW35-1	10.00	34.52	20.00	0.00	0.0930	17.49	1.142	30.23	0.45
		40.10	34.62	50.10	30.00	0.0930	19.64	1.020	33.93	0.51
	MW35-2	10.00	34.42	19.90	0.00	0.0930	17.51	1.139	30.21	0.45
		40.15	34.54	50.10	30.20	0.0930	19.65	1.017	33.98	0.51
VNIIM	MW35-1 MW35-2	10.21	34.45	20.27	0.15	0.0177	17.80	1.130	30.48	0.20
		40.15	34.58	50.33	29.97	0.0177	19.99	1.019	33.95	0.20
NIM	MW35-1	40.60	34.30	49.90	31.30	0.0314	18.51	1.006	34.10	1.60
CENAM	MW35-1 MW35-2	10.18	34.50	20.40	-0.04	0.0214	36.70	1.110	31.00	0.62
		40.05	34.50	50.30	29.80	0.0214	40.40	1.010	34.11	0.41

(*) $R_{th} = (T_H - T_C)/\varphi$

Table 22 : Measurements at 10 °C and 40 °C for EPS35 and EPS70 specimens

Lab.	Specimen	T_m [°C]	e [mm]	T_H [°C]	T_C [°C]	A [m²]	φ [W·m⁻²]	R_{th} (*) [m²·K·W⁻¹]	$\lambda \cdot 10^{-3}$ [W·m⁻¹·K⁻¹]	U(λ)$\cdot 10^{-3}$ [W·m⁻¹·K⁻¹]
NIST	EPS35-1	10.00	34.99	20.00	0.00	0.1297	17.41	1.150	30.43	0.30
	EPS35-2	40.00	35.16	50.00	30.00	0.1297	19.21	1.040	33.80	0.40
	EPS70-1	10.00	69.88	20.00	0.00	0.1297	8.74	2.290	30.52	0.60
	EPS70-2	40.00	70.10	50.00	30.00	0.1297	9.64	2.070	33.81	0.60
LNE	EPS35-1	10.00	34.89	20.01	-0.02	0.0900	17.49	1.145	30.47	0.30
	EPS35-2	40.03	34.89	49.99	30.07	0.0900	19.27	1.033	33.76	0.34
	EPS70-1	10.01	69.73	20.07	-0.05	0.0900	8.83	2.277	30.63	0.30
	EPS70-2	39.97	69.74	49.94	30.00	0.0900	9.68	2.059	33.87	0.34
NPL	EPS35-1	10.00	34.93	20.00	0.00	0.0930	17.52	1.139	30.68	0.46
		40.10	35.02	50.10	30.10	0.0930	19.66	1.016	34.46	0.52
	EPS35-2	10.00	34.65	20.00	0.00	0.0930	17.73	1.128	30.72	0.46
		40.10	34.77	50.10	30.10	0.0930	19.78	1.011	34.37	0.52
	EPS70-1	10.00	69.48	20.30	-0.40	0.0930	9.19	2.245	30.94	0.46
		39.90	69.60	50.10	29.70	0.0930	10.09	2.023	34.41	0.52
	EPS70-2	9.90	69.11	20.10	-0.30	0.0930	9.12	2.237	30.89	0.46
		40.20	69.25	50.50	29.80	0.0930	10.25	2.018	34.32	0.51
VNIIM	EPS35-1	10.16	34.78	20.29	0.03	0.0177	18.16	1.116	31.18	0.20
	EPS35-2	40.25	34. 82	50.51	29.98	0.0177	20.35	1.009	34.52	0.20
NIM	EPS35-1	41.36	34.84	50.43	32.30	0.0314	19.73	0.917	38.00	1.40
CENAM	EPS35-1	9.20	34.55	18.40	0.00	0.0214	16.60	1.110	31.13	0.59
	EPS35-2	40.00	34.55	50.20	29.80	0.0214	20.20	1.000	34.31	0.62
	EPS70-1	8.60	69.36	17.40	-0.30	0.0214	7.90	2.230	31.10	0.93
	EPS70-2	40.20	69.36	50.60	29.80	0.0214	10.40	2.000	34.59	0.69
PTB	EPS20-1	40.00	19.98	50.01	30.00	(**)	35.22	0.569	35.10	1.05
	EPS20-2	40.00	20.35	50.01	30.00	(**)	32.60	0.620	32.50	0.98
	EPS25-1	40.00	25.12	50.002	30.005	(**)	25.60	0.783	32.00	0.96
	EPS25-2	40.00	25.20	50.004	30.003	(**)	25.20	0.786	32.00	0.96

(*) $R_{th} = (T_H - T_C)/\varphi$
(**) *Not reported*

Appendix 3 – Degrees of equivalence results tables

Table 23 : Degrees of equivalence and error functions for MW35 specimens

MW35, 10°C								
Lab.	$\lambda_i \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$U(\lambda_i) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	D_i	U_i	E_i	$\lambda_{CRV} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u(\lambda_{CRV}) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u_{cut\text{-}off} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]
NIST	29.98	0.40	-0.010	0.017	0.587			
LNE	30.02	0.30	-0.009	0.014	0.594			
NPL	30.22	0.45	-0.002	0.018	0.109			
VNIIM	30.48	0.20	0.007	0.013	0.500	30.28	0.059	0.125
NIM								
CENAM	31.00	0.62	0.024	0.023	1.032			
PTB								

U_{add}*=0.185* 10^{-3} $W \cdot m^{-1} \cdot K^{-1}$

MW35, 23°C								
Lab.	$\lambda_i \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$U(\lambda_i) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	D_i	U_i	E_i	$\lambda_{CRV} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u(\lambda_{CRV}) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u_{cut\text{-}off} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]
NIST	31.52	0.40	-0.009	0.017	0.541			
LNE	31.54	0.32	-0.009	0.015	0.567			
NPL	31.80	0.48	-0.001	0.019	0.030	31.81	0.054	0.150
VNIIM	31.94	0.20	0.004	0.014	0.276			
NIM	32.50	0.70	0.022	0.025	0.869			
CENAM	32.10	0.39	0.009	0.017	0.527			
PTB								

U_{add}*=0.208* 10^{-3} $W \cdot m^{-1} \cdot K^{-1}$

MW35, 40°C								
Lab.	$\lambda_i \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$U(\lambda_i) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	D_i	U_i	E_i	$\lambda_{CRV} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u(\lambda_{CRV}) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u_{cut\text{-}off} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]
NIST	33.51	0.40	-0.009	0.012	0.805			
LNE	33.60	0.34	-0.007	0.010	0.683			
NPL	33.96	0.51	0.004	0.015	0.252	33.83	0.057	0.135
VNIIM	33.95	0.20	0.004	0.007	0.504			
NIM	34.10	1.60	0.008	0.047	0.170			
CENAM	34.11	0.41	0.008	0.012	0.698			
PTB								

U_{add}*=0.092* 10^{-3} $W \cdot m^{-1} \cdot K^{-1}$

Table 24 : Degrees of equivalence and error functions for EPS35 specimens

EPS35, 10°C								
Lab.	$\lambda_i \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$U(\lambda_i) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	D_i	U_i	E_i	$\lambda_{CRV} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u(\lambda_{CRV}) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u_{cut\text{-}off} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]
NIST	30.43	0.30	-0.011	0.015	0.693	30.76	0.056	0.133
LNE	30.47	0.30	-0.009	0.015	0.608			
NPL	30.70	0.46	-0.002	0.019	0.095			
VNIIM	31.18	0.20	0.014	0.014	0.974			
NIM								
CENAM	31.13	0.59	0.012	0.022	0.542			
PTB								

U_{add}=0.202 10^{-3} $W \cdot m^{-1} \cdot K^{-1}$

EPS35, 23°C								
Lab.	$\lambda_i \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$U(\lambda_i) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	D_i	U_i	E_i	$\lambda_{CRV} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u(\lambda_{CRV}) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u_{cut\text{-}off} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]
NIST	31.89	0.30	-0.005	0.014	0.337	32.03	0.075	0.183
LNE	31.90	0.32	-0.004	0.014	0.311			
NPL	32.28	0.48	0.008	0.017	0.445			
VNIIM								
NIM								
CENAM	32.69	0.60	0.021	0.021	1.000			
PTB	31.35	0.94	-0.021	0.031	0.698			

U_{add}=0.185 10^{-3} $W \cdot m^{-1} \cdot K^{-1}$

EPS35, 40°C								
Lab.	$\lambda_i \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$U(\lambda_i) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	D_i	U_i	E_i	$\lambda_{CRV} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u(\lambda_{CRV}) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u_{cut\text{-}off} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]
NIST	33.80	0.60	-0.011	0.020	0.524	34.16	0.051	0.177
LNE	33.76	0.34	-0.012	0.014	0.827			
NPL	34.42	0.52	0.007	0.018	0.411			
VNIIM	34.52	0.20	0.011	0.013	0.789			
NIM								
CENAM	34.31	0.62	0.004	0.021	0.212			
PTB								

U_{add}=0.214 10^{-3} $W \cdot m^{-1} \cdot K^{-1}$

Table 25 : Degrees of equivalence and error functions for EPS70 specimens

EPS70, 10°C								
Lab.	$\lambda_i \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$U(\lambda_i) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	D_i	U_i	E_i	$\lambda_{CRV} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u(\lambda_{CRV}) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u_{cut\text{-}off} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]
NIST	30.52	0.60	-0.007	0.021	0.334	30.73	0.091	0.190
LNE	30.63	0.30	-0.003	0.014	0.240			
NPL	30.92	0.46	0.006	0.016	0.364			
VNIIM								
NIM								
CENAM	31.10	0.93	0.012	0.031	0.387			
PTB								

U_{add}*=0.185* 10^{-3} $W \cdot m^{-1} \cdot K^{-1}$

EPS70, 23°C								
Lab.	$\lambda_i \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$U(\lambda_i) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	D_i	U_i	E_i	$\lambda_{CRV} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u(\lambda_{CRV}) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u_{cut\text{-}off} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]
NIST	31.90	0.60	-0.009	0.019	0.480	32.20	0.095	0.203
LNE	32.02	0.32	-0.006	0.013	0.423			
NPL	32.37	0.49	0.005	0.016	0.328			
VNIIM								
NIM								
CENAM	32.73	0.66	0.016	0.021	0.776			
PTB								

U_{add}*=0.173* 10^{-3} $W \cdot m^{-1} \cdot K^{-1}$

EPS70, 40°C								
Lab.	$\lambda_i \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$U(\lambda_i) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	D_i	U_i	E_i	$\lambda_{CRV} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u(\lambda_{CRV}) \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]	$u_{cut\text{-}off} \times 10^{-3}$ [$W \cdot m^{-1} \cdot K^{-1}$]
NIST	33.81	0.60	-0.008	0.019	0.438	34.10	0.100	0.215
LNE	33.87	0.34	-0.007	0.014	0.464			
NPL	34.37	0.52	0.008	0.017	0.459			
VNIIM								
NIM								
CENAM	34.59	0.69	0.014	0.022	0.667			
PTB								

U_{add}*=0.208* 10^{-3} $W \cdot m^{-1} \cdot K^{-1}$

Appendix 4 – Technical Protocol

CCT-S2 Supplementary comparison on thermal conductivity measurement by guarded hot plate

CONTENTS

1. INTRODUCTION

Under the Mutual Recognition Arrangement (MRA) [1] the metrology equivalence of national measurement standards will be determined by a set of key comparisons chosen and organized by the Consultative Committees of the CIPM working closely with the Regional Metrology Organizations (RMOs).

In May 2003 the Consultative Committee for Thermometry (CCT), formed a new Working Group 9 (WG-9) for Thermophysical Quantities, which was tasked to determine the need for key comparisons in the field of thermophysical quantities. At its meeting in June 2005 CCT-WG-9 identified a need for comparisons of three quantities: thermal conductivity, thermal diffusivity, and emittance. It was decided to initiate three supplementary comparisons of these quantities as a preliminary step to eventual key comparisons. In particular, a supplementary comparison on thermal conductivity measurement by guarded hot plate (GHP) was initiated, and LNE (France) was appointed as the pilot laboratory. The supplementary comparison was accepted by the CIPM and assigned the identifier CCT-S2. Seven NMIs confirmed in June 2006 their agreement to participate in this comparison: CENAM, LNE, NIM, NIST, NPL, PTB and VNIIM.

The procedures outlined in this document cover the technical procedure to be followed during the measurements of the transfer specimens. The procedure follows the guidelines established by the BIPM [1].

2. ORGANIZATION

2.1. Participants

CENAM	Leonel Lira Cortes Área Eléctrica, Division de Termometría Centro Nacional de Metrología Km 4,5 Carretera a los Cues, El Marques, C.P. 76241 Querétaro, Mexico	llira@cenam.mx
LNE	Bruno Hay Laboratoire National de Métrologie et d'Essais Division Thermique et Optique, 29, Avenue Roger Hennequin F-78197 Trappes, FRANCE	bruno.hay@lne.fr
NIM	Jintao Zhang Heat Division, National Institute of Metrology Bei San Huan Dong Lu 18, Beijing 100013 China	zhangjint@nim.ac.cn
NIST	Robert Zarr Engineering Laboratory, National Institute of Standards and Technology 100 Bureau Drive, Gaithersburg, MD 20899-8632 USA	rzarr@nist.gov
NPL	Clark Stacey Thermal Performance Group, Materials Division National Physical Laboratory Teddington, Middlesex TW11 0LW, England, UK	clark.stacey@npl.co.uk
PTB	Ulf Hammerschmidt Physikalisch-Technische Bundesanstalt AG 1.74, Bundesallee 100, 38116 Braunschweig Germany	Ulf.Hammerschmidt @ptb.de
VNIIM	Nikolay Sokolov D.I. Mendeleyev Institute for Metrology Moskovsky Prospect 19, St. Petersburg 190005 Russia	N.A.Sokolov@vniim.ru

In accordance with the guidelines established at the CCT, participants must be members of CCT, and have made an independent realization of their thermal conductivity scale. By their declared intention to participate in this supplementary comparison, the laboratories accept the general instructions and the technical protocols written in this document and commit themselves to follow the procedures strictly. Once the protocol and list of participants has been agreed to, no change to the protocol or list of participants may be made without prior agreement of all participants.

2.2. Form of comparison

The comparison will be carried out through the measurements of transfer standard artefacts. The same set of specimens will circulate between the different NMIs and will be successively measured by the participants (with the exception of PTB). This protocol aims to minimize issues of material variability. Each participant will send the specimens back to the pilot laboratory after having performed their series of measurements.

The pilot laboratory will supervise all specimens on a regular basis by recurrently measuring their densities. In addition, it will arrange machining of the specimens according to the requests of each successive participant. The specimens will be measured first by NIST that needs 1016 mm diameter specimens. Then the specimens will be stepwise cut down, first to 610 mm x 610 mm and finally to 330 mm diameter. Each cutting process has to leave the central part of a specimen undisturbed. All cutting scraps will be marked and retained at the pilot laboratory, in order to reassemble, as closely as possible, 610 mm x 610 mm specimens at the end of the comparison process. This procedure will enable the pilot laboratory to check the stability of the specimens by measuring their thermal conductivity at the beginning and at the end of the comparison. Figure 1 gives a chronological scheme of the circulation of the specimens.

Each laboratory will have 6 months for measurements and transportation. The deadline for returning the artefacts will be advised when the specimens are shipped to the participants. Final results must be submitted directly to the pilot laboratory within 3 months after completing measurements. If for some reason, the NMI is not ready or customs clearance takes too much time in a country, the participant laboratory must contact the pilot laboratory. Exclusion of a participant's results from the report may occur if the results are not available in time to prepare the draft report.

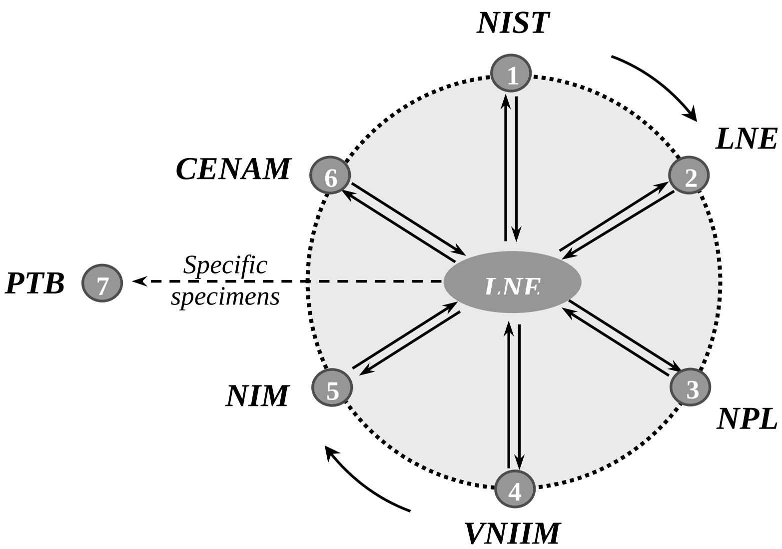

Figure 1 : Chronological circulation of the specimens

2.3. Comparison Schedule

Activity	Date
Confirmation of participants	June 2006
Technical protocol sent to participants	July 2006
Technical protocol and schedule approved by participants	September 2006
Preparation of the specimens	August to December 2006
Circulation of the specimens and thermal conductivity measurements from labs 1 to 7	January 2007 to Dec. 2009
Second measurements by the pilot laboratory at the end of the comparison	March 2010
Pre-Draft A process starts	May 2010
Draft A comparison report circulated	May 2012
Draft B comparison report submitted to CCT	May 2014

2.4. Transportation of the artefacts

The specimens will be transported from laboratory to laboratory in closed flight containers provided by the pilot laboratory. The pilot laboratory covers the costs for transportation to the participant laboratory. Arrangement of the transportation back to the pilot laboratory is each participant laboratory's responsibility. Each participating laboratory covers the costs for its own measurements, transportation and any customs charges as well as for any damage that may have occurred within its country.

3. DESCRIPTION OF THE ARTEFACTS

Two different types of insulating material will be used for this comparison: mineral wool (MW) and expanded polystyrene (EPS). These materials are chosen because they best meet the selective criteria for thermal conductivity measurements: low dispersion of density within a batch and long-term stability.

- The mineral wool specimens will be high density glass fiber boards having a thickness of 35 mm. They will come from a batch of the certified reference material IRMM-440, whose properties were characterized by six European laboratories in a framework of a certification project initiated by the Institute of Reference Materials and Measurements [2].
- Two special batches of expanded polystyrene boards (35 mm and 70 mm thick) with a nominal density of 22 kg·m^{-3} will be specifically produced by Lafarge (France). It will be a grey EPS containing graphite in order to avoid the "thickness effect" that is observed usually for normal white EPS (cf. EN 13163 standard [3]).

A pair of disk-shaped specimens (with diameter ϕ of 1016 mm) will be prepared by LNE from each of the three materials characterized above. Two other pairs of EPS specimens of thicknesses 20 mm and 25 mm will be specially machined for PTB because of their variant type of GHP apparatus.

4. MEASUREMENT PROCEDURE

4.1. Traceability

Temperature measurements should be made using the International Temperature Scale of 1990 (ITS-90).

4.2. Measurand

The measurand is the thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$). Depending on the participant, the individual GHP apparatus operates either with a single specimen or a pair of specimens. The thermal conductivity measurements will be carried out using guarded hot plate apparatus according to the International Standard ISO 8302 [4] by all laboratories except PTB (whose dimensions of the GHP differ from those recommended in [4]). In the case of two-specimen apparatus, the mean thermal conductivity λ of the pair of specimens is determined at steady-state conditions by using equation (1).

$$\lambda = \frac{\Phi \cdot L}{A \cdot \Delta T} \tag{1}$$

where Φ is the measured rate of heat flow (W) passing through two surfaces of the metering area for the specimen pair; A (m^2) is the metering cross-section area; $\Delta T = T_h - T_c$ (K) is the measured temperature difference between each of the specimens' hot (T_h) and cold surfaces (T_c); and L (m) is the mean thickness of the pair of specimens. Values of λ are indicated for the mean specimen temperature, $T_m = (T_h + T_c)/2$.

In a single-specimen apparatus, the second specimen is replaced by insulation and a guard plate. In this case, equation (1) is modified slightly by removal of the constant coefficient 2.

4.3. Measurement instructions

Before performing thermal conductivity measurements, participants are requested to:

- Condition specimens at (23 ± 2) °C and (50 ± 5) % relative humidity for a minimum of 5 days, in order to reach the thermal equilibrium with the environment (i.e. constant mass). This equilibrium is judged as having been obtained when two successive mass measurements within a 24 h interval do not differ by more than ± 0.5 %,
- Measure the thickness of the specimens inside the apparatus (under a clamping pressure of about 1000 Pa). In the case of mineral wool, adequate spacers are used in order to avoid thickness variation (and density change) during the thermal conductivity measurements,
- Calculate the specimen density from the measurements of the mass and volume.

Depending on its individual apparatus, each participant performs either just a part of the following program or the whole on each pair of specimens.

- Four successive runs at a fixed temperature of 23 °C with a temperature difference of 20 °C over a short period of time. After each run, the specimens are removed from the apparatus and then reassembled. This procedure yields information on the repeatability.
- One run at each of the two mean test temperatures of 10 °C and 40 °C under a temperature difference of 20 °C.

No information relating to the comparison, such as measurement results, obtained by a participant during the course of the comparison shall be communicated to any party other than the pilot laboratory. The pilot laboratory will be responsible for disseminating information to other participants and any other release of information. In the latter case the pilot laboratory will seek permission of all the participants before releasing information.

5. MEASUREMENT UNCERTAINTY

Measurement uncertainty shall be estimated according to the ISO Guide to the Expression of Uncertainty in Measurement [5] by taking into account the uncertainty components associated to the measurement of the following quantities:

- Heat flow Φ (W),
- Average thickness of the pair of specimens L (m),
- Metering cross-section area A (m^2),
- Temperature difference between hot and cold surfaces of the specimen ΔT (K),

The participant laboratories should provide to the pilot laboratory an example of uncertainty budget in the case of EPS35 (35 mm thick expanded polystyrene board). Table 1 gives an example of table for the uncertainty budget.

EPS35 - 23 °C				
Input Quantity X_i	Estimate x_i	Sensitivity coefficient cx_i	Std uncertainty $u(x_i)$	$cx_i \cdot u(x_i)$ ($W \cdot m^{-1} \cdot K^{-1}$)
Φ (W)				
A (m^2)				
L (m)				
ΔT (K)				
λ ($W \cdot m^{-1} \cdot K^{-1}$)		u_c (λ) (k=1)		

Table 1 : Example of table for the uncertainty budget

6. REPORTING OF RESULTS

The final results should be submitted to the pilot laboratory at the latest within three months from completion of measurements. The results should be submitted in the spreadsheet form, which is distributed to the participants by the pilot laboratory (cf. "Measurement report" in annex). The form requires entry of the specimen characteristics, the results of measurements, the thermal conductivity values and the associated expanded uncertainty (k=2).

Following receipt of all measurement reports from the participating laboratories, the pilot laboratory will follow the procedure outlined in the Guidelines for CCPR Comparison Report Preparation [6].

7. ANALYSIS OF COMPARISON RESULTS

7.1. Introduction

In the Technical Supplement to the Mutual Recognition Arrangement (MRA) [7], key comparisons are identified as the technical basis for the arrangement. One primary goal of this supplementary comparison CCT-S2, is to follow the process of a key comparison as closely as possible, including the technical deliverables, which are outlined as:

- Reference values, known as comparison reference values (CRV)
- Unilateral degree of equivalence (DoE) of each national measurement standard, both its deviation from the CRV and the uncertainty of that deviation at the 95 % level of confidence.

As the key comparisons are the technical basis for the MRA, the results reported should be the basis upon which CMCs are validated and subsequently evaluated. For instance, the CCPR Guidelines state that all participants should be able to "check the consistency of their CMCs with the KC results" ([6], §8.1). This means that the comparison should determine the value of each participant's bias (DoE) and the uncertainty associated with that value in order to give some indication as to whether a participant has adequately estimated the likely magnitude of that bias.

7.2 Data analysis

Weighted mean averaging with cut-off will be employed to obtain a nominal comparison reference value (CRV) for each temperature. 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 [8].

The individual participant's results will then be compared to the CRVs. The relative deviations and their uncertainties are the unilateral relative Degrees of Equivalence (DoE). To determine whether any of the results can be considered outliers, the error function will be calculated and presented.

REFERENCES

[1] MRA, Mutual Recognition Arrangement, BIPM, 1999. T.J. Quinn, “Guidelines for CIPM key comparisons” BIPM, Paris, (1999, modified 2003).

[2] Quin S., Venuti G., DePonte F. and A. Lamberty, “Certification of a resin-bonded glass fibre board for thermal conductivity between -10 °C and +50 °C - IRMM440”, Report EUR 19572 EN (2000).

[3] EN 13163 - “Thermal insulation products for buildings - Factory made products of expanded polystyrene (EPS) - Specification” (2008).

[4] ISO 8302 - “Thermal Insulation - Determination of steady-state thermal resistance and related properties - Guarded hot plate apparatus” (2001).

[5] BIPM IEC IFCC ILAC ISO IUPAC IUPAP and OIML 2008 *Guide to the Expression of Uncertainty in Measurement* (Geneva: International Organization for Standardization) available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf.

[6] Consultative Committee on Photometry and Radiometry Key Comparison Working Group, Guidelines for CCPR Comparison Report Preparation, 2009 available at http://www.bipm.org/utils/common/pdf/Guidelines_for_CCPR_KC_Reports.pdf.

[7] International Committee for Weights and Measures, 2003, Mutual Recognition of National Measurement Standards and of Calibration and Measurement Certificates Issued by National Measurement Institutes (Paris: Comité International des Poids et Mesures), available at http://www.bipm.org/utils/en/pdf/mra_2003.pdf .

[8] CCPR-G2 Rev.3, 2013 *Guidelines for CCPR Key Comparison Report Preparation.*

ANNEX: MEASUREMENT REPORT

Laboratory: **Country:** **Contact:** **Material:**

SPECIMEN CHARACTERISTICS Arrival date of the sample : Conditioning conditions :

Specimen reference	Date of the measurement	Thickness (mm)	Diameter (mm)	Width (mm)	Weight (g)	Density (kg/m3)

THERMAL CONDUCTIVITY MEASUREMENTS

Measurement N°	Nominal temperature Tn (°C)	Specimen reference	Date of the measurement	Thickness * (mm)	Mean temperature Tm (°C)	Hot Surface temperature Th (°C)	Cold Surface temperature Tc (°C)	Density of heat flow (W/m2)	Thermal resistance R (m2.K/W)	Thermal conductivity λ (mW/(m.K))	u(λ) (k=2) (mW/(m.K))
1	23 °C										
2	23 °C										
3	23 °C										
4	23 °C										
5	10 °C										
6	40 °C										

* during λ measurement

Observations: