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International Comparison of Guarded Hot Plate Facilities at Low Temperature on Mineral Wool Insulation Material

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ABSTRACT

In 2000, the Institute for Reference Materials and Measurements (IRMM) proposed the creation of a new European certified reference material for the thermal conductivity measurements IRMM-440. A specific production batch of high density (64–78 kg/m^3) resin-bonded glass fiber boards was manufactured and characterized by a group of six leading European laboratories with long-standing expertise in thermal conductivity measurements, including four national measurements institutes. The IRMM-440 has since been used as the European certified reference material for thermal conductivity measurements on thermal insulation covering temperatures between –10°C and 50°C, and most frequently to calibrate Heat Flow Meter apparatus making measurements at 10°C.

The aim of this article is to present an international interlaboratory comparison of thermal conductivity measurements made on IRMM-440 at temperatures between –170°C and 20°C. This is the first step to define an insulation reference material for low temperature applications. Indeed, there is currently no reference material available with certified thermal conductivity values for temperatures below –10°C.

Furthermore, a legal requirement for European conformity marking (CE) was introduced in the European Economic Area in August 2012 for thermal insulation products intended for use in building equipment and industrial installations. The manufacturers of these products are required to declare a "curve" of thermal conductivity against temperature for the whole temperature range at which the product is intended to be used. The values used to produce these "curves" need to be verified by an independent notified laboratory.

The data presented in this article comes from a report from the European Commission (2015): "Certification of a Resin-Bonded Glass Fibre Board for Thermal Conductivity between –10ºC and 50ºC, IRMM-440" (European Commission, 2000), which gives the thermal conductivity values of Dipartimento di Fisica Tecnica (DFT) from Italy. It also includes additional measurements made by National Physical Laboratory (NPL) from UK, Laboratoire National de Métrologie et d'Essais (LNE) from France, Forschungsinstitut für Wärmeschutz (FIW) from Germany and Instytut Mechanizacji Budownictwa i Górnictwa Skalnego (IMBiGS) of Poland. These last four laboratories have each made measurement on their own specimens of the IRMM-440 certified reference material. All measurements were carried out using Guarded Hot Plate apparatus designed for low temperature measurement and following specifications given in the (International Organization for Standardization [ISO] 8302).

Keywords: thermal conductivity, insulation material, mineral wool, low temperature

1. INTRODUCTION

New European product standards for insulation used in building equipment and industrial installations were published in 2009 and became effective in August 2012. These product standards provide a mandatory requirement for manufacturers to declare a "curve" of thermal conductivity against temperature that is verified by an independent notified laboratory. The notification is given by European Commission based on a national procedure of accreditation.

There are currently nine products standard in this field (EN 14303, EN 14304, EN 14305, EN 14306, EN 14307, EN 14308, EN 14309, EN 14313, and EN 14314). These standards gives rules for CE

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mark declaration for the following materials: mineral wool; elastomeric foam (FEF); cellular glass (CG); calcium silicate (CS); extruded polystyrene foam (XPS); polyurethane foam (PUR); polyisocyanurate foam (PIR); expanded polystyrene (EPS), polyethylene foam (PEF); and phenolic foam (PF). They cover the use of these materials from the low temperatures found in cryogenic systems, liquid natural gas storage, and climatic chambers and up to high temperatures found in industrial furnaces and steam pipes.

The thermal performance must be declared between the lowest and the highest temperature at which the product is intended to be used and the different standards give ranges for these temperatures: the lowest temperature given for CG with –265°C, FEF, PUR, PIR, PF is –200°C; XPS and EPS is –180°C, CS is –170°C, and finally PEF with –80°C.

There are very few laboratories within Europe that can perform the required thermal conductivity measurement below ambient temperatures. The calibration of a Guarded Hot Plate at low temperatures involves a number of specific technical challenges and the accuracy of measurements decreases at lower temperatures. Indeed, the thermal conductivity of insulation material is much lower at –150°C than at 20°C, which leads to lower rates of heat flux density and reduced accuracy at these temperatures. However, because of the need for CE marking of insulation products and the global need of reducing overall energy consumption, the requirement for very accurate thermal conductivity values at all temperatures has become far more important in recent years. The lack of certified reference material available for temperature below –10°C currently limits the control of quality within laboratories carrying out thermal conductivity measurements at these subambient temperatures.

The data presented in this article comes from a report from the European Commission (2000), giving thermal conductivity values measured by Dipartimento di Fisica Tecnica (DFT) from Italy, and also includes measurements made by National Physical Laboratory (NPL) from UK, Laboratoire National de Métrologie et d'Essais (LNE) from France, Forschungsinstitut für Wärmeschutz (FIW) from Germany, and Instytut Mechanizacji Budownictwa i Górnictwa Skalnego (IMBiGS) from Poland, on their own specimens of the Institute for Reference Materials and Measurement (IRMM)-440 certified reference material. All measurements were made using Guarded Hot Plate apparatus designed for low temperature measurement and following specifications given in the international standard ISO 8302.

2. LABORATORY APPARATUS AND TEST METHOD

Table 1 summarizes the major parameters of the Guarded Hot Plate apparatus used to provide data for this article. All apparatus were operated in a double-sided mode (heat flow through a pair of specimens).

The Guarded Hot Plate method, which has been standardized under the ISO 8302 and EN (Test Method EN 12667) determines steady-state thermal transmission properties of flat slab specimens having a low thermal conductivity. The standard test methods for double-sided Guarded Hot Plates utilize the one-dimensional steady-state thermal conductivity equation for the determination of thermal conductivity (λ):

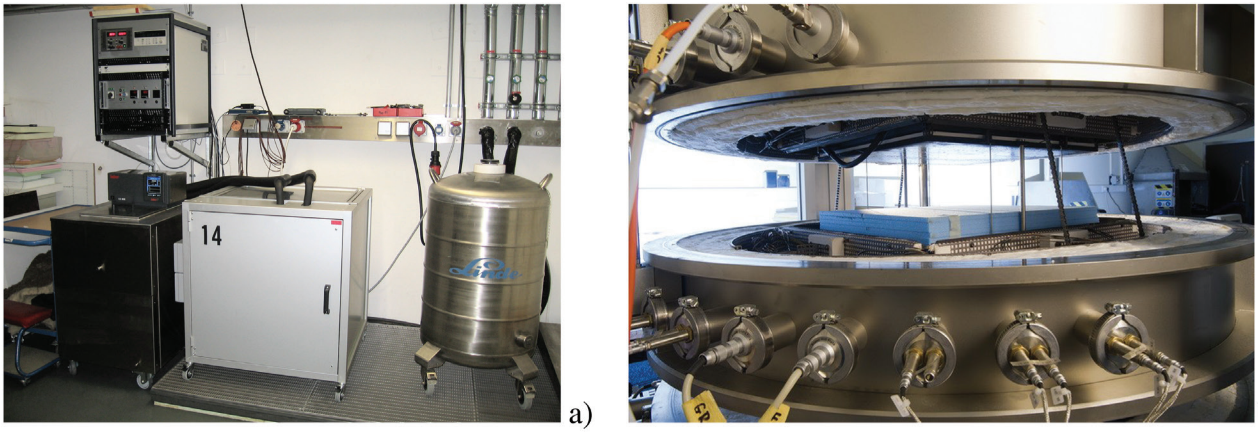
$$\lambda = \frac{Q\,L}{2A\,\Delta T} \tag{1}$$

Where Q is the time-rate of one-dimensional heat flow through the meter area of the guarded heater plate (W); A is the meter area of the apparatus normal to heat flow (m^2); ΔT (K) is the temperature difference between the specimen hot (T_h) and cold surfaces (T_c); and L(m) is the in-situ mean thickness of the pair

Table 1. Laboratory Guarded Hot -Plate apparatus.

Parameter	LNE	NPL	FIW	DFT	IMBiGS
Plate (mm)	500 × 500	305 × 305	340 × 340	300 × 300	300 × 300
Meter plate (mm)	247 × 247	152.5 × 152.5	200 × 200	148 × 158	148 × 148
Guard-center gap (mm)	3	2	2	2	2
Plate emittance	>0.9	>0.89	>0.9	_	>0.9
Temperature sensor	PT 100	T type	T type	_	PT 100
Operation mode	Two-sided	Two-sided	Two-sided	Two-sided	Two-sided

Notes: LNE, Laboratoire National de Métrologie et d'Essais, France; NPL, National Physical Laboratory, UK; FIW, Forschungsinstitut für Wärmeschutz, Germany; DFT, Dipartimento di Fisica Tecnica, Italy; IMBiGS, Instytut Mechanizacji Budownictwa i Górnictwa Skalnego, Poland.



a) b)

Figure 1. (a) Picture of the low temperature GHP used by Forschungsinstitut für Wärmeschutz (FIW) and (b) schematic diagram of the Netzsch apparatus [used by Laboratoire National de Métrologie et d'Essais (LNE) and Instytut Mechanizacji Budownictwa i Górnictwa Skalnego (IMBiGS)].

of specimens. Values of λ are reported at the mean temperature, $T_m = (T_h - T_c)/2$.

Guarded Hot Plates designed to make measurements at low temperature have cold plates that are cooled with a cooling fluid (in general liquid nitrogen) and heated to a specified temperature with an electrical heater (Figure 1). The heater plate is designed like a standard Guarded Hot Plate.

3. REFERENCE MATERIALS

The IRMM-440 is a resin-bonded glass fiber board, certified over the temperature range –10°C to 50°C, and with a quoted uncertainty of ±1%. Table 2 summarizes the reference materials by designation, material description, density, thickness, temperature range, and source.

The thermal conductivity (λ) of IRMM-440 is characterized statistically as a function of the mean temperature (T_m). Each sample is batch certified and is accompanied by a global certificate, having one or more property values certified by a procedure that establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence. Certified values of thermal conductivity for IRMM-440 (European Commission, 2000) are given by Equation (2):

$$\lambda(T) = a_0 + a_1 T + a_2 T^2 \tag{2}$$

Table 3 summarizes the regression coefficients (a_i) and expanded uncertainties (U) at a coverage factor of $k = 2$ for predicted values of IRMM-440.

Table 3. Regression coefficients for reference materials (λ in W/mK).

Designation	a_0	a_1	a_2	U ($k = 2$) W/mK
IRMM-440	0.0293949	0.0001060	2.047×10^{-7}	±0.00028

Note: IRMM, Institute for Reference Materials and Measurements.

4. PROTOCOL

Five laboratories from across Europe have carried out measurements on IRMM-440 at temperatures below 0°C. The DFT data came from a European Commission report (European Commission, 2000), and for NPL the data is from a paper published at the ITCC 2009 (Stacey, Salmon, & Simpkin, 2010). LNE, FIW and IMBiGS launched a test campaign in 2012 and 2013 to add additional data for low temperature.

Table 2. Reference materials.

Designation	Description	Density (kg/m³)	Thickness (mm)	Temperature (°C)	Source and Reference
IRMM[a]-440	Resin-bonded glass Fiber board	64–78	35	–10 to 50	(European Commission, 2015)

Note: IRMM, Institute for Reference Materials and Measurements.
[a]Certified reference material issued by IRMM.

All the laboratories provided thermal conductivity values for IRMM-440 from at least –150°C to 10°C, and two laboratories provided values down to –170°C. The protocol used in this study was to collect thermal conductivity data and then to fit them with a two-degreed polynomial. The comparison between the laboratories has been done at defined temperatures with the interpolated polynomial function.

5. RESULTS

The measurement data from the participating laboratories are presented in Table 4 and Figure 3. The values of bulk density (ρ) were rounded to the first decimal, the values of thickness (L) were rounded to the second decimal, and each value represents the average of a pair of specimens. The number of significant digits for the thermal conductivity parameters is tabulated as received.

Figure 2. Schematic diagram of the low temperature GHP used by National Physical Laboratory (NPL).

Table 4. Measured data.

	T_{mean} (°C)	λ (W/mK)	Δ*T*		T_{mean} (°C)	λ (W/mK)	Δ*T*
FIW Thickness 34.8 mm Density 68.6 kg/m³	10.2	0.0305	15.2	LNE Thickness 34.6 mm Density 72.6 kg/m³	10	0.03044	30
	–11.9	0.0282	15.1		0	0.02937	30
	–41	0.0249	15.4		–20	0.0273	30
	–65.4	0.0219	13.3		–110	0.0178	50
	–150.3	0.0136	47.6		–130	0.0154	50
					–150	0.0134	50
NPL Thickness 34.8 mm Density 68.4 kg/m³	50.4	0.0355	20	IMGiS Thickness 32.9 mm 71.5 kg/m³ (measured in March 2013)	20	0.03165	20
	0.2	0.0296	20		10	0.0306	20
	–49.1	0.0245	20		0	0.02953	20
	–97.2	0.019	20		–50	0.02447	20
	–149.2	0.0139	20		–100	0.01922	20
	–174.7	0.0117	20		–150	0.0137	20
DFT Density 66.7 kg/m³, Thickness 34.71 mm	33.45	0.03331					
	33.41	0.03329					
	20.09	0.03171					
	20.05	0.0317					
	10.13	0.03063					
	10.06	0.03061					
	0.22	0.02963					
	0.08	0.02962	Not available				
	–9.96	0.02858					
	–20.76	0.02739					
	–31.77	0.02624					
	–63.22	0.02295					
	–103.25	0.01871					
	–132.07	0.01557					
	–150.98	0.01347					
	–169.54	0.01141					

Notes: LNE, Laboratoire National de Métrologie et d'Essais, France; NPL, National Physical Laboratory, UK; FIW, Forschungsinstitut für Wärmeschutz, Germany; DFT, Dipartimento di Fisica Tecnica, Italy; IMBiGS, Instytut Mechanizacji Budownictwa i Górnictwa Skalnego, Poland.

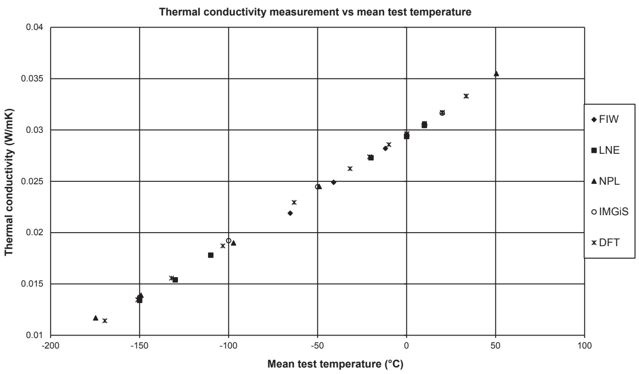


Figure 3. Thermal conductivity measurement made by the five participating laboratories versus mean test temperature.

5.1 Analysis of the results

In order to compare the curves for each laboratory it was decided to determine a two-degree polynomial regression by the method of least square. Coefficient a_0, a_1, a_2 are those defined in Equation (2). Table 5 gives the calculated coefficients of the interpolated polynomial. Table 6 gives the thermal conductivity calculated from the least square polynomial.

This method allows us to analyze the difference between the laboratories at each selected temperature: 10°C, –50°C, –100°C, and –150°C. Table 7 and Figure 4 give the relative deviation of the mean values versus temperature. Each laboratory is within ±3% of the mean values, which represents ±0.0004 W/mK.

Table 5. Coefficents a_i of the interpolated polynomial.

	a_0 (W/mK)	a_1 (W/mK^2)	a_2 (W/mK^3)	r^2
FIW	0.02942534	0.00011704	7.6992E–08	0.9994098
LNE	0.02938853	0.00010390	–2.0401E–08	0.9998960
NPL	0.02965936	0.00011327	5.6152E–08	0.9996816
IMBiGS	0.02959710	0.00010075	–3.406E–08	0.9999650
DFT	0.02963202	0.00010610	–5.7009E–09	0.9999069
IRMM*	0.0293949	0.00010600	2.05E–07	—

Notes: LNE, Laboratoire National de Métrologie et d'Essais, France; NPL, National Physical Laboratory, UK; FIW, Forschungsinstitut für Wärmeschutz, Germany; DFT, Dipartimento di Fisica Tecnica, Italy; IMBiGS, Instytut Mechanizacji Budownictwa i Górnictwa Skalnego, Poland; IRMM, Institute for Reference Materials and Measurements.
*Value useful only on the range –10°C to 50°C.

Table 7. Relative deviation versus mean temperature in relation to each polynomial function (%)

T_{mean} (°C)	FIW	LNE	NPL	IMBiGS	DFT
10	–0.07	–0.65	0.57	–0.07	0.22
0	–0.39	–0.51	0.40	0.19	0.31
–10	–0.69	–0.39	0.25	0.44	0.39
–50	–1.66	–0.10	–0.12	1.27	0.61
–100	–1.98	–0.37	0.15	1.68	0.53
–150	–0.26	–2.14	2.17	0.59	–0.36
–170		+2.54			–2.54

Notes: LNE, Laboratoire National de Métrologie et d'Essais, France; NPL, National Physical Laboratory, UK; FIW, Forschungsinstitut für Wärmeschutz, Germany; DFT, Dipartimento di Fisica Tecnica, Italy; IMBiGS, Instytut Mechanizacji Budownictwa i Górnictwa Skalnego, Poland.

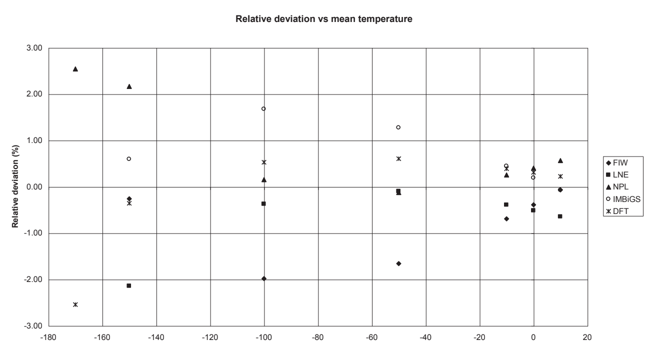


Figure 4. Relative deviation (calculated with mean values) versus mean temperature (%).

Table 6. Thermal conductivity (interpolated value).

T_{mean} (°C)	FIW	LNE	NPL	IMBiGS	DFT	IRMM
10	0.03060	0.03043	0.03080	0.03060	0.03069	0.03048
0	0.02943	0.02939	0.02966	0.02960	0.02963	0.02939
–10	0.02826	0.02835	0.02853	0.02859	0.02857	0.02836
–50	0.02377	0.02414	0.02414	0.02447	0.02431	
–100	0.01849	0.01879	0.01889	0.01918	0.01896	
–150	0.01360	0.01334	0.01393	0.01372	0.01359	
–170			0.01203		0.01143	

Notes: LNE, Laboratoire National de Métrologie et d'Essais, France; NPL, National Physical Laboratory, UK; FIW, Forschungsinstitut für Wärmeschutz, Germany; DFT, Dipartimento di Fisica Tecnica, Italy; IMBiGS, Instytut Mechanizacji Budownictwa i Górnictwa Skalnego, Poland; IRMM, Institute for Reference Materials and Measurements.

5.2 Proposition of new reference values for low temperature (below –10°C)

It is proposed that the mean values of thermal conductivity at temperatures below –10°C be used to determine a two-degree polynomial with the least square method. The value at –10°C will be the values of the actual IRMM certificate. Table 8 gives the regression coefficient of this polynomial.

TABLE 8. Coefficients a_i of the interpolated polynomial**.**

	a_0 (W/mK)	a_1 (W/mK^2)	a_2 (W/mK^3)	r^2
IRMM*	0.02945568	0.00010766	1.799E–08	0.99994072

**Note:* IRMM, Institute for Reference Materials and Measurements.

The combined uncertainty of the λ-certified values was determined from both contributions: uncertainty of the thermal conductivity measurements and uncertainty because of the fit λ versus temperature. Table 9 gives the calculated values versus temperature with the relative uncertainty (95% confidence interval $k = 2$).

The overall relative uncertainty of the λ-certified values is within [1.0%–4.9%], which corresponds to a constant limit of uncertainty at the 95% confidence level of ±0.0007 W/mK over the measurement range [–170°C to –10°C].

Each laboratory has measured the IRMM insulation material at, at least, 2.7% or 0.00038 W/mK (see Table 10 and Figure 5).

Table 9. Proposition of thermal conductivity of IRMM-440 for temperature below –10°C.

T_{mean} (°C)	λ (W/mK)	λ relative uncertainty (%) at the 95% confidence interval
–10	0.02836	2.83600×10^{-4}
–50	0.02412	2.69136×10^{-4}
–100	0.01887	2.58387×10^{-4}
–150	0.01371	2.20504×10^{-4}
–170	0.01167	8.72687×10^{-4}

Note: IRMM, Institute for Reference Materials and Measurements.

Table 10. Relative deviation versus mean temperature with the new proposal (%).

T_{mean} (°C)	FIW	LNE	NPL	IMBiGS	DFT
–10	–0.42	–0.12	0.53	0.72	0.67
–50	–1.46	0.10	0.08	1.48	0.81
–100	–2.00	–0.40	0.13	1.65	0.51
–150	–0.80	–2.67	1.61	0.05	–0.90
–170			3.03		–2.08

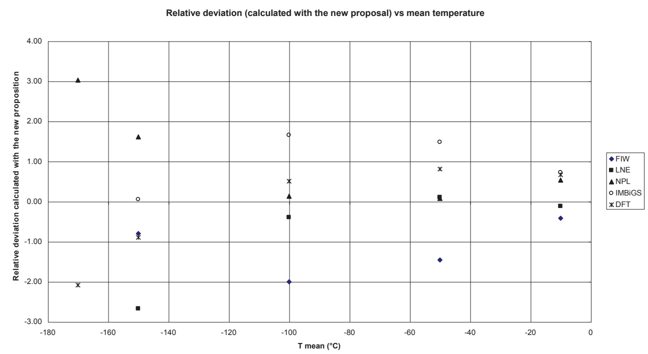


Figure 5. Relative deviation (calculated with new polynomial) versus mean temperature (%).

6. CONCLUSIONS

An intercomparison of thermal conductivity measurement at low temperature (between –170°C and 10°C) has been carried out by five experienced laboratories from across Europe. The results show a good agreement between these laboratories and a proposition is made to increase the temperature range of European certified reference material IRMM-440 to cover lower temperatures by using these results and an interpolated polynomial by the least square method. During the tests, the IRMM-440 material shows no type of deterioration at the low temperature range.

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