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Thermal conductivity measurement of insulating innovative building materials by hot plate and heat flow meter devices: a Round Robin Test

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Abstract

The characterization of thermal insulation properties of construction materials represents a fundamental step on the building insulation assessment. In recent years innovative materials have been introduced in the market to fulfill the continuously growing requirements of energy saving and sustainability, and their performance is not so reliable and mature as it happens for traditional insulators. The work presents a Round Robin Test realised among six European laboratories hosting hot plates devices to measure the thermal conductivity of four different materials: aerogel, vacuum insulation panels, polystyrene and birch wood fibre insulation boards. After the definition of the common measurement protocol, the tests campaign was executed and the results were checked with a consistency analysis. Data showed that the hot plate apparatuses result suitable for the measurement of the tested innovative materials, both in terms of absolute values retrieved and repeatability. The reproducibility is satisfactory as well, except for vacuum insulation

panels, the most insulating samples, which present values of standard deviations quite high, at least in relative terms, so showing that the thermal properties of high performance materials must be assessed with particular care.

Keywords

Hot plate; thermal conductivity; aerogel; vacuum insulation panels, polystyrene, birch wood boards.

Nomenclature

A	area of the sample	[m ²]
c	calibration factor	[-]
d	deviation for each laboratory	[W/m·K]
EPS	expanded polystyrene panel	
f	correction coefficient	[K ⁻¹]
h	between-laboratory consistency	
j	laboratory number	
k	within-laboratory consistency	
n	number of the tests executed	
q	heat flow per time unit	[W]
Q	heat flow per time and area unit	[W/m ²]
r	repeatability	
R	reproducibility	
RRT	Round Robin Test	
s	standard deviation of the averages	[W/m·K]
T	temperature	[K]
V	voltage output	[V]
VIP	vacuum insulation panel	

Greek symbols

λ thermal conductivity [W/m·K]

Subscripts

a average

c cold

h hot

m measured

r reference

T temperature

1. Introduction

The energy saving requirements in the building sector pushes the research on highly insulating materials towards continuously increasing performance. Wall thermal insulation properties, as well as transparent surfaces transmittance, play a fundamental role for the definition of the heating consumption, as showed by sensitivity analyses conducted to assess the reliability of building simulation tools [1]. Besides, in recent years, the environment-friendly materials gained a growing success, as the sustainability is becoming a central issue on the construction sector [2] [3].

The thermal conductivity of most common building materials could be retrieved from standard sources of data [4], [5]; nevertheless, the direct measurement (with its uncertainty) constitutes the most reliable way to assess this thermophysical property [6], [7], [8].

One of the most accurate approach for the measurement of homogenous materials thermal conductivity in steady-state conditions is the guarded hot-plate method [9] [10], [11], [12], [13], [14], [15].

The principle is quite simple: the sample is positioned among two surfaces kept at known constant temperatures T_h and T_c ; registering the stationary heat flow passing through the plates, it is possible to obtain

the thermal conductivity of the material analysed by means of the one-dimensional integral form of the Fourier equation [16], [17]:

$$\lambda = \frac{qL}{A(T_h - T_c)} \quad (1)$$

where q is the heat flow per time unit and A is the area of the sample facing the plates.

The guarded hot plate method proved useful for the assessment of the thermal behaviour of particular samples, such as phase change materials [18], flat evacuated glazing [19] and vacuum insulation panels.

The scientific community is well aware of the issue of comparing the results coming from different laboratories: the Keymark group [20] defined scheme rules to ensure laboratories conformity, stating that registered laboratories shall produce results on the measurements of λ at 10°C respect to the reference material within $\pm 1,5\%$. Extending the working temperature from 300 K up to 1,000 K, Ebert et al. [21] conducted an interlaboratory campaign on a calcium silicate material; various types of instruments were used in these measurements: from guarded hot plates to hot wires, and a self-built apparatus. An increase of uncertainty emerged at higher temperatures. The temperature dependence of thermal conductivity of expanded glass granulate was also investigated by Schreiner et al. [22] through a Round Robin Test (RRT) executed again with different devices. It was demonstrated that the transient methods show very good compliance with the Keymark reference curve.

In recent years a particular attention has been dedicated to the super-insulating materials thermal tests. In the EBC Annex 65 [23], for instance, the theme of repeatability was the object of a common-exercise on Insulation Panels and Advanced Porous Materials. Within this framework, a series of different samples of Vacuum Insulation Panels (VIP) and aerogels produced by the main market players were tested by the research centres involved, highlighting a scarce reproducibility for the tested surveys. It was not strictly a RRT, but a common exercise as each laboratory tested a different sample, although made of the same material and by the same producer. VIPs were also the subject of a RRT conducted according to the Standard ASTM C1484-00 [24]; a satisfactory agreement among the results was found, showing at the same time the importance of a proper assessment of the edge effect.

The present work is aimed at performing a RRT among various laboratories hosting different hot plate devices, using four types of materials (the same samples for all the participants, which is an important feature of the tests). According to the Standard ASTM E 691 for interlaboratory studies [25], six laboratories were

involved in the RRT; the participant came from six different European Universities: four Italian, one Spanish and one Latvian.

The materials described below were chosen for the tests:

- *aerogel*, composed of nanoporous silicon molecules with trapped air, deposited on a reinforced glass fiber matrix [26]; nominal thickness of each panel: 10 mm; apparent density: 92 kg/m³.
- *Expanded polystyrene panel (EPS)*, a solid polystyrene closed-cell foam, one of the most common insulation material; nominal thickness of each panel: 40 mm; apparent density: 8 kg/m³.
- *Vacuum insulation panels (VIPs)*, made of a microporous insulation material consisting of inorganic oxides, which main constituent is fumed silica [27]; nominal thickness of each panel: 25 mm; apparent density: 167 kg/m³.
- *Birch wood fibre board*, derived from the birch veneer waste and produced by a Latvian manufacturer [28]; nominal thickness of each panel: 50 mm; apparent density: 55 kg/m³.

The selection criterion obeyed to the following considerations: aerogel and VIPs represent the innovative high-insulation solutions, birch wood fibre insulation boards stand for the sustainable answer, and the common polystyrene panels fulfilled the function of the reference insulating material.

Four samples for each material were tested, except for the VIPs, as only two samples were available.

2. Methodology and description of the hot plate apparatuses

2.1 Measurement protocol

A seven steps measurement protocol was shared among the partners, with the purpose of controlling, as far as possible, the variables linked to the measurement procedures.

- Step 1 (panels conditioning): the panels to be tested were conditioned in a climatic chamber (or similar devices) for the humidity evaporation between 105 °C and 110 °C, except for EPS panels because of possible structural damages at these temperatures. The conditioning lasted no less than 24 hours and it has been considered effective if the panel weight difference between two conditioning periods was lower than 0.1 kg/m³ or 0.01% by volume.
- Step 2 (thickness measurement): the thickness of each panel on its four sides was measured and the mean value was registered.

- Step 3 (register environmental conditions): the room temperature and relative humidity were registered, fixing them to values close to 20 °C and 50%, respectively.
- Step 4 (panel setup): the lateral edges of the panels to be tested were covered with a tape, at the aim of preventing air humidity to enter the panels themselves.
- Step 5 (pressure set): the pressure was set to 3,000 Pa or the closer value each device allowed to reach.
- Step 6 (temperature set): the hot plate temperatures were set to 60 °C (hot side) and 15 °C (cold side), or to the closer values that each device allowed to reach.
- Step 7 (measurement): retrieve the thermal conductivity and repeat the procedure for all the samples.

During the RRT, the results of each laboratory remained unknown to all other participants, to avoid reciprocal influence on the measurements.

2.2 Description of the hot plate and heat flow meter apparatuses

Each hot plate device used in the RRT differs from the others for various characteristics. In the next paragraphs a short description of each apparatus is provided.

2.2.1 University of Bologna

The experimental set-up is based on the heat flux meter method with a single-specimen symmetrical configuration; the schematic of the apparatus is shown in figure 1. Water from two separate thermostatic baths (Techne Tempette Mod. RB-54) (1 and 2 in figure 1) is circulated by two pumps (3 and 4 in figure 1) through two serpentine coils in thermal contact with a thick copper plate each (5 and 6 in figure 1). The plates are thus kept at a constant but different temperature and strive to provide two isothermal boundaries for the specimen to be tested. The surface temperature on the plates is measured by ten K-type thermocouples. Two heat flux meters are placed between the two plates and the test specimen. These assemblies allows the calculation of the specific heat flux through the metering area, A , from a measurement of their voltage output, V , and a calibration factor, c , which is a function of the average temperature of the heat flux meter face in contact with the test specimen, T_h or T_c , respectively. Hence, for each flow meter, the following obtains:

$$q = c(T_{h,c})V \quad (1)$$

On the flow meter side facing the specimen, six K-type thermocouples are located, following the arrangement suggested by EN 12667 [17] in order to calculate the average temperature on each side of the test specimen, T_c and T_h respectively. The average temperature of the test specimen, T_a , is obtained averaging these two values. The ambient air temperature is also measured by another thermocouple placed close to the set-up. All thermocouples cold junctions are kept at the reference temperature (0 °C) by a Peltier cooler ice point (Kaye 170, 12 in figure 1). Data relative to heat flux meter and thermocouple output are recorded continuously during the measurement period using a HP 3488A switcher, a HP 3458A multimeter and stored in a personal computer for processing.

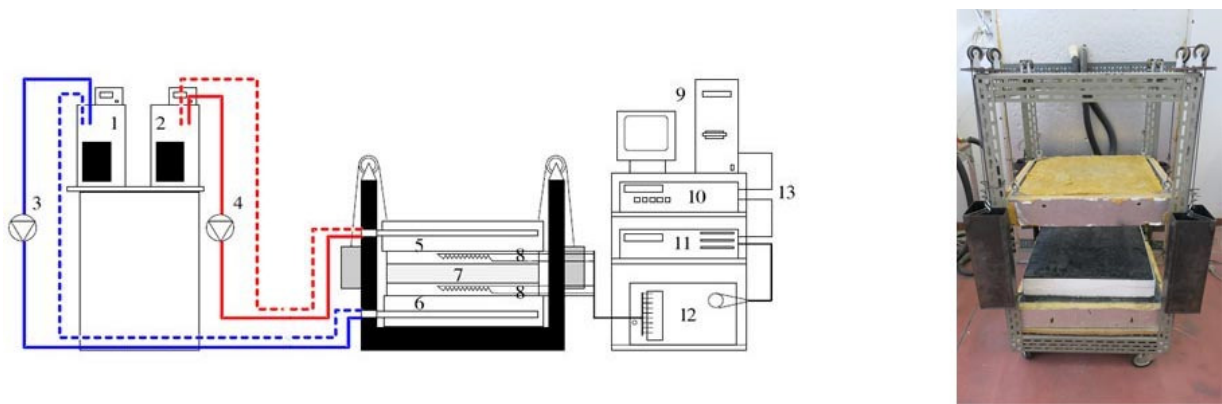


Figure 1 - Illustrative chart and general view of Bologna heat flow meter apparatus. 1), 2) thermostatic baths; 3), 4) circulator pumps; 5), 6) heating and cooling plates; 7) test specimen; 8) heat flow meters; 9) personal computer; 10) multimeter; 11) switcher unit; 12) ice point; 13) general purpose interface bus.

The experimental setup has been checked in order to verify its compliance with the standard requirements for conductivity measurements. The most significant controls were performed on the following parameters: flatness, emissivity and temperature distribution of the heating and cooling plates, number and location of temperature sensors and emissivity of the two heat flow meters.

2.2.2 University of Latvia

The Department of Physics, Faculty of Physics and Mathematics, University of Latvia hosts a Taurus TCA 500 P measuring instrument. It is a system for determining the thermal conductivity of samples by the heat flow meter method (figure 2). The test sample, with dimensions of 50 × 50 cm, is placed between the cooling plate and the heater plate; the heat flows from the heater plate through the sample to the cooling plate from where it is carried off. A passive protection zone surrounds the heat flow meter to prevent, as far as possible,

lateral heat transfer. Depending on the thickness of the sample, the width of the protection zone considerably influences the uncertainty of the measuring set-up. The minimum thickness of the test specimen is 20 mm; the maximum is 200 mm.

The instrument includes the following main functional units:

- measuring chamber;
- one cooling plate with heat flow meter;
- one hot plate with heat flow meter;
- one isothermal block.

The heater and cooling plate temperature is adjusted by means of a Peltier cryostat to establish a temperature gradient from the heater plate across the specimen of $5 \div 30\text{ }^{\circ}\text{C}$. The average sample temperature can be changed between 0 and $60\text{ }^{\circ}\text{C}$.

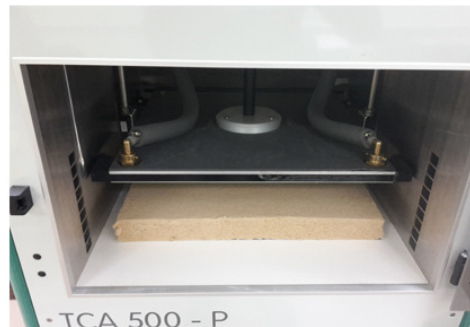
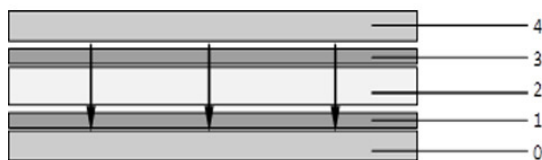


Figure 2 – Illustrative chart and general view of Latvian laboratory system. 0) heater plate; 1) heat flow meter 2; 2) specimen; 3) heat flow meter 1; 4) cooling plate.

The thermocouples of each measurement plane are embedded in the surface of the heating and cooling plate, respectively, in fixed positions. When measuring solid samples, sponge rubber mats with known thermal conductivity are used as compensating layers. A total of 10 thermocouples are used for the direct determination of the average measurement temperature difference.

The purpose of the isothermal block is to provide compensation for the thermocouple voltages for the transition from thermal material to copper conductors, which also have thermal voltages.

In order to keep the effort for compensating these error voltages low, all transitions from thermal conductors to copper conductors are made in an environment kept at a uniform temperature. This environment is the isothermal block, designed to keep the temperature gradient within the block very small. The temperature

within the isothermal block is determined by means of a highly accurate PT100 measuring resistor and it is used for correcting the thermal voltage error.

The device is equipped with a lifting equipment (the upper cold plate is moved by an electric lift device) and force (resolution 1.0 N) and thickness (resolution 0.1 mm) sensors.

2.2.3 Universidad Politécnica de Madrid

At Universidad Politécnica de Madrid the thermal conductivity λ of the samples was measured by means a heat flow meter model HFM 436 *Lambda*. The HFM 436/Lambda complies with the standards ASTM C518-17 [29] and EN 12667 [17].

In the HFM 436 the sample is placed between two heated plates, set at different temperatures. For the measurement, the dimension of the samples is 30 x 30 cm. The heat flow Q per time and area unit trough the sample is measured by a calibrated heat flux transducer.

The test is executed after reaching a thermal equilibrium. The sample is placed between two heated plates controlled to a user-defined average sample temperature and temperature drop.

The plate temperatures are controlled by bidirectional Heating/Cooling Peltier systems, coupled with a closed loop fluid flow with an integrated forced air heat exchanger as shown in figure 3.

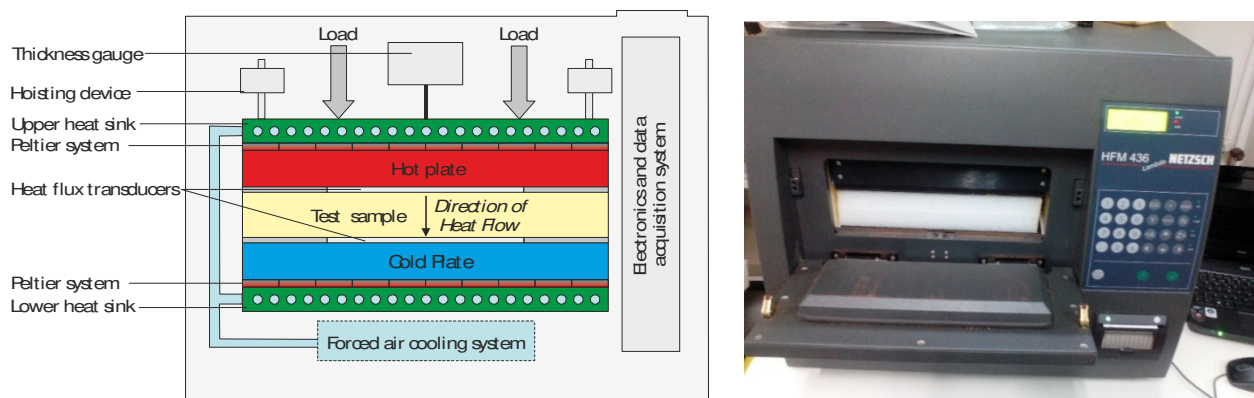


Figure 3 - Illustrative chart and general view of Madrid system.

In the HFM 436, one of two heat flow transducers measure the heat flow though the sample. The signal of a heat flow transducer (in volts) is proportional to the heat flow through the transducer (see equation 1).

2.2.4 University of Modena and Reggio Emilia

The Guarded Hot Plate apparatus (GJHP) is a "double-specimen" type, with frontal dimensions 300 mm x 300 mm. Figure 4 shows an exploded sketch of the GHP. The heaters are divided in a central square section to which a heat flow rate is provided through embedded resistors fed with direct current, and a contour section provided also with a heat flow rate, and maintained at the same temperature of the central section by a closed-loop control system.

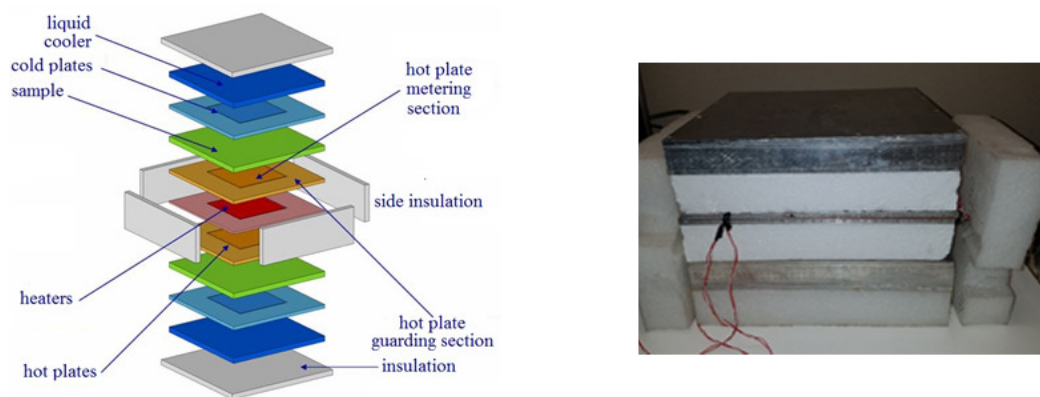


Figure 4 - Illustrative chart and general view of Modena and Reggio Emilia guarded hot plate.

Building the sandwich from the ham to the two bread slices, the central and contour heaters have over and under themselves two couples of aluminium plates (hot plate metering and guarding sections) that follow their contours. In these plates, 5 sensors are housed; continuing outwards, the spaces for the samples to be tested are located. Continuing again, other couples of aluminium plates are located following the contours of the central square and the guard. Two sensors are located in the central aluminium elements (cold plates metering section). At the top and bottom of the apparatus two liquid cooled metal plates are located. A thick layer of expanded polyethylene insulates all the apparatus.

The apparatus is provided with 14 shielded RTD Pt100 elements, divided as follows:

- 8 positioned within the measurement zone;
- 6 positioned inside the ring guard near the main heating element.

All the sensor are connected with a National Instrument NI-4251 PCI board with TBX-68T and their signal is collected through a LabVIEW custom software that is also in charge of managing the temperature control of the hot plates. The electric power fed into the central zone, completely converted into heat due to Joule effect, is measured by a professional digital wattmeter.

2.2.5 University of Perugia

The apparatus hosted in the Laboratory of Thermotechnics of the University of Perugia is a "single-specimen" type, the frontal dimensions are 50 x 50 cm; figure 5 shows an illustrative chart and a general view of the guarded hot plate. The main heating element (hot plate) is divided into a central square component (measurement area) that provides a specific power through resistors fed with direct current, and a contour element (guard ring) maintained at the same temperature of the central part by the control system. Below the heating element, sandwiched between two panels of insulating materials, a second guard hot plate is installed; also this plate is maintained at the same temperature of the central part by the control system. All the apparatus is aimed at the achievement of a one-dimensional heat flow within the measurement zone. The specimen is interposed between the main heating element and a water cooled metal plate (cooling unit).

The apparatus is provided with 34 shielded J-type thermocouples, divided as follows:

- 17 thin-wire thermocouples positioned within the measurement zone;
- 8 thin-wire thermocouples positioned inside the ring guard near the separation from the measurement zone;
- 8 immersion thermocouples positioned inside the cooling unit in direct contact with the specimen;
- 1 shielded thermocouple inserted inside the bottom guard hot plate.

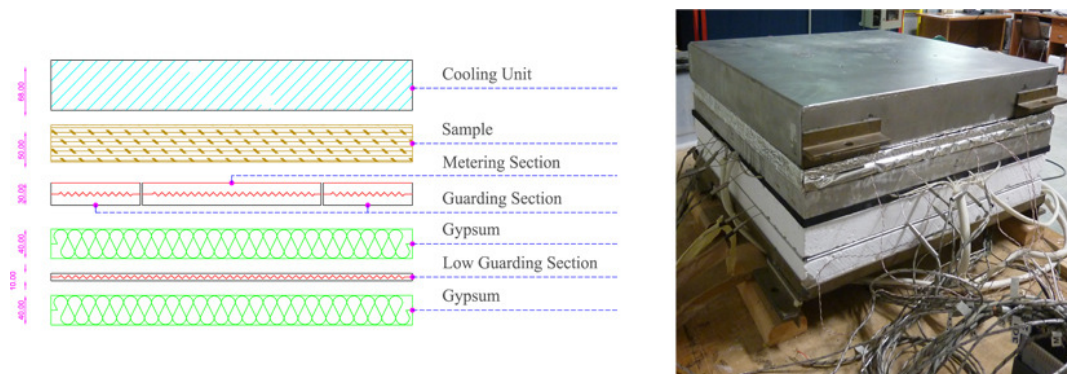


Figure 5 - Illustrative chart and general view of Perugia guarded hot plate.

The sensors can be divided into two subgroups:

- a group that monitors the thermal imbalance between the measuring area and the guard zones;
- a second group to measure the average temperature of the hot and cold face of the specimen, at the aim of defining an average cross temperature difference.

The sensors installed to balance the temperatures between the various elements of the apparatus are connected to the control panel, which is made of 6 temperature controllers capable of operating both in PID and ON / OFF modalities. The monitoring apparatus and the measurement of the various temperatures elements is implemented by 3 DAQ data acquisition systems, four channels each, for a total of twelve sensors acquired. The measurement of the power fed into the central zone is carried out with a professional digital multimeter.

2.2.6 Politecnico di Torino

The apparatus hosted in the Energy Department of Politecnico di Torino is a single sample Dynamic Heat Flow Meter apparatus (DHFM) “Lasercomp FOX 600” conforming to the standards ASTM C518-17 [29] and EN 12667 [17]. The apparatus can operate in steady state conditions (measurement of the thermal conductivity and thermal resistance), and dynamic thermal conditions can be used for the characterization of the dynamic thermal properties (volumetric specific heat and enthalpy according to ASTM C1784-14 [30]). The experimental apparatus was calibrated by the manufacturer with a reference expanded polystyrene sample certified by NIST (1450b NIST SRM). Moreover, a second calibration process was carried out in Politecnico di Torino by using a Pyrex glass “Pyrex50mmIRMN” certified by National Physical Laboratory (NPL) of Teddington UK.

The measurement plates have dimensions of 60 x 60 cm and are both equipped with thermocouples (temperature resolution ± 0.01 °C) and heat flux transducers (measuring area of 254 x 254 mm) that allow measuring samples with a minimum size of 30 x 30 cm. The HFM device is heated/cooled by Peltier elements that utilize an external chiller system as a heat exchanger. Linear optical encoders are located in the corners of the apparatus to measure the sample thickness. The scheme of the apparatus and the picture of the measurement rig are reported in figure 6.

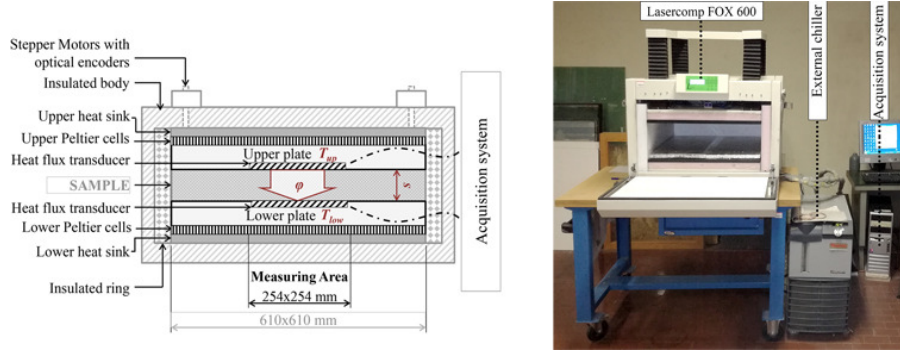


Figure 6 - Illustrative chart and general view of the Politecnico di Torino heat flow meter apparatus.

2.3 Statistical analysis

The results of all the experimental tests have been subjected to a statistical analysis, as indicated in the Standard ASTM E 691 [25]. For each laboratory partner j involved in the RRT, the thermal conductivity average value $\bar{\lambda}_j$ of each material tested is calculated as follows:

$$\bar{\lambda}_j = \sum_{i=1}^n \frac{\lambda_i}{n} \quad (2)$$

where λ_i represents the individual test result in the laboratory j and n is the number of the tests executed in one material in that laboratory; the correspondent standard deviation (SD) is described by equation (3):

$$s_j = \sqrt{\frac{\sum_{i=1}^n (\lambda_i - \bar{\lambda}_j)^2}{(n-1)}} \quad (3)$$

The relative SD is the percentage ratio between the standard deviation and the average value.

As far as intermediate statistics, the average of the thermal conductivity averages for each material is defined by the next relation:

$$\bar{\bar{\lambda}} = \sum_{j=1}^p \frac{\bar{\lambda}_j}{p} \quad (4)$$

where p is the number of the laboratories involved (six).

Thus, it is possible to evaluate the deviation for each laboratory j :

$$d_j = \bar{\lambda}_j - \bar{\bar{\lambda}} \quad (5)$$

and the standard deviation of the averages:

$$s_{\bar{\lambda}} = \sqrt{\frac{\sum_{j=1}^p d_j^2}{(p-1)}} \quad (6)$$

Once the average of the averages and its standard deviation are retrieved, the precision statistics have to be introduced, in terms of repeatability standard deviation s_r and reproducibility standard deviation s_R [31]. The first is obtained with equation (7):

$$s_r = \sqrt{\frac{\sum_{j=1}^p s^2}{p}} \quad (7)$$

While the latter is the maximum value between $(s_R)^*$ and s_r , with $(s_R)^*$ following defined:

$$(s_R)^* = \sqrt{(s_\lambda)^2 + \frac{(s_r)^2(n-1)}{n}} \quad (8)$$

The within-laboratory consistency starts from the definition of the parameter k_j :

$$k_j = \frac{s_j}{s_r} \quad (9)$$

High values of the term k_j indicate within-laboratory imprecision, while very low ones may be linked, for instance, to an insensitive measurement scale.

The between-laboratory consistency h_j , a parameter indicating at a glance the variability of the test method and particular laboratories that exceed or result close to the critical values, is calculated by the next equation:

$$h_j = \frac{d_j}{s_\lambda} \quad (9)$$

The k and h critical values at the 0.5 % significance level depend on the number of laboratories and the number of replicate test results [25]: since not all samples were tested in all laboratories and the number of samples available is not the same for all materials, the critical values result different from one type of material to another (table 1).

If the data consistency analysis shows that no inconsistent results emerge, the 95% repeatability and reproducibility limits (r and R) could be calculated without excluding any measurement:

$$r = 2.8s_r \quad (10)$$

$$R = 2.8s_R \quad (11)$$

The relative 95% repeatability and reproducibility are represented by the percentage ratio between the r and R and the average of the thermal conductivity averages $\bar{\lambda}$.

Table 1 - Within-laboratory consistency k and between-laboratory consistency h critical values for the materials tested.

Material	Number of samples	Number of labs	Within-laboratory consistency k	Between-laboratory consistency h
Aerogel*	4	4	1.73	1.49
Polystyrene	4	6	1.84	1.92
VIP**	2	4	1.95	1.49
Birch wood	4	6	1.84	1.92

* in Bologna lab, the four samples of aerogel were put together in order to reach the minimum thickness required by the apparatus, so retrieving only one value, not included in the consistency analysis; furthermore, it was not possible to test aerogel in Latvian lab.

** Only two VIPs were available; in Modena Lab the two samples of VIP were put together, as required by the "double-specimen" type apparatus, so retrieving only one value, not included in the consistency analysis; furthermore, it was not possible to test them in Madrid, as their dimensions exceeded the maximum size of the hot plate available.

3. Results and discussion

Since the various apparatuses did not allow to test the samples at the same average temperature of the protocol, the correction proposed by ISO 10456 [5] was used for the materials considered in such standard to take into account of the dependence of thermal conductivity on temperature. More specifically, the thermal conductivity λ of each sample was modified from that retrieved at the average measurement temperature T_m to that at the cross comparison reference temperature $T_r = 37.5^\circ\text{C}$, according to the following equation:

$$\lambda(T_r) = \lambda(T_m)e^{f_T(T_r-T_m)} \quad (12)$$

The correction coefficients used for the various materials are indicated in table 2.

Table 2 – Temperature correction coefficients for the materials tested.

Material	Correction coefficient [K^{-1}]	Reference of the ISO 10456
Aerogel	0.0043	Cellular glass
Polystyrene	0.0030	Expanded polystyrene
VIP	0.0030	Calcium silicate
Birch wood	0.0040	Wood wool boards

The results of the tests conducted in the laboratories are reported in table 3 and figure 7.

Table 3 – Results of all tests conducted in the RRT.

Material	Sample	Warm side - Temperature [°C]						Cold side - Temperature [°C]						Rough thermal conductivity [W/m K]						Corrected thermal conductivity [W/m K]						
		Bologna	Latvia	Madrid	Modena	Perugia	Torino	Bologna	Latvia	Madrid	Modena	Perugia	Torino	Bologna	Latvia	Madrid	Modena	Perugia	Torino	Sample	Bologna	Latvia	Madrid	Modena	Perugia	Torino
Aerogel	A1	50.07	-	52.00	19.79	59.31	60.02	24.04	-	22.00	-1.06	15.50	15.02	0.021	-	0.0263	0.0191	0.019	0.0208	A1	0.0210	-	0.0264	0.0216	0.0190	0.0208
	A2		-	52.00		59.36	60.02		-	22.00		15.32	15.01		-	0.0213		0.018	0.0195	A2		-	0.0213		0.0180	0.0195
	A3		-	52.00	19.87	59.48	60.02		-	22.00	-1.07	16.28	15.02		-	0.0229	0.0198	0.019	0.0201	A3		-	0.0230	0.0223	0.0190	0.0201
	A4		-	52.00		59.30	60.02		-	22.00		16.50	15.01		-	0.0233		0.019	0.0197	A4		-	0.0233		0.0190	0.0196
																				Average	0.0210	-	0.0235	0.0219	0.0187	0.0200
																				SD	0.0000	-	0.0021	0.0006	0.0005	0.0006
																				Relative SD	0.0%	-	9.0%	2.5%	2.6%	3.0%
	Polystyrene	P1	49.37	45.90	52.00	20.27	59.56	60.03	24.25	15.7	22.00	-1.17	17.92	15.01	0.0370	0.0358	0.0373	0.0324	0.0360	0.0368	P1	0.0371	0.0365	0.0373	0.0352	0.0359
P2		49.37	46.50	52.00	59.65		60.02	24.26	15.7	22.00	16.93		15.01	0.0370	0.0358	0.0375	0.0370		0.0364	P2	0.0371	0.0365	0.0375	0.0369		0.0364
P3		49.29	46.80	52.00	19.88	59.68	60.02	24.27	15.6	22.00	-1.18	16.93	15.01	0.0360	0.0357	0.0372	0.0324	0.0370	0.0366	P3	0.0361	0.0364	0.0372	0.0353	0.0369	0.0366
P4		49.16	-	52.00		59.67	60.02	24.24	-	22.00		16.85	15.01	0.0370	-	0.0372		0.0370	0.0367	P4	0.0371	-	0.0372		0.0369	0.0367
																				Average	0.0368	0.0365	0.0373	0.0352	0.0367	0.0366
																				SD	0.0005	0.0001	0.0001	0.0000	0.0005	0.0002
																				Relative SD	1.4%	0.2%	0.4%	0.0%	1.4%	0.5%
VIP		V1	50.53	48.1	-	19.22	59.78	60.03	23.42	15.3	-	1.75	15.78	15.02	0.0050	0.0057	-	0.0048	0.0080	0.0049	V1	0.0050	0.0058	-	0.0051	0.0080
	V2	50.76	46.7	-	37.77		60.02	23.43	30.1	-	18.02		15.01	0.0050	0.0051	-	0.0070		0.0045	V2	0.0050	0.0051	-	0.0072		0.0045
																				Average	0.0050	0.0054	-	0.0051	0.0076	0.0047
																				SD	0.0000	0.0005	-	0.0000	0.0006	0.0002
Birch wood	W1	49.51	49.2	52.00	19.62	36.72	60.02	23.50	15.7	22.00	-1.16	18.03	15.01	0.0450	0.0389	0.0426	0.0363	0.0460	0.0392	W1	0.0452	0.0397	0.0427	0.0406	0.0479	0.0391
	W2	49.66	49.2	52.00		36.71	60.02	24.20	15.7	22.00		18.05	15.01	0.0470	0.0382	0.0415		0.0440	0.0389	W2	0.0471	0.0390	0.0416		0.0458	0.0389
	W3	49.67	49.1	52.00	19.97	36.73	60.03	24.23	15.6	22.00	-1.16	18.03	15.01	0.0460	0.0388	0.0415	0.0357	0.0460	0.0392	W3	0.0461	0.0396	0.0415	0.0399	0.0479	0.0392
	W4	49.40	48.5	52.00		36.70	60.02	24.16	15.6	22.00		18.03	15.02	0.0470	0.0389	0.0430		0.0480	0.0388	W4	0.0471	0.0398	0.0430		0.0500	0.0388
																				Average	0.0464	0.0395	0.0422	0.0403	0.0479	0.0390
																				SD	0.0009	0.0004	0.0008	0.0005	0.0017	0.0002
																				Relative SD	2.0%	0.9%	1.8%	1.2%	3.6%	0.5%

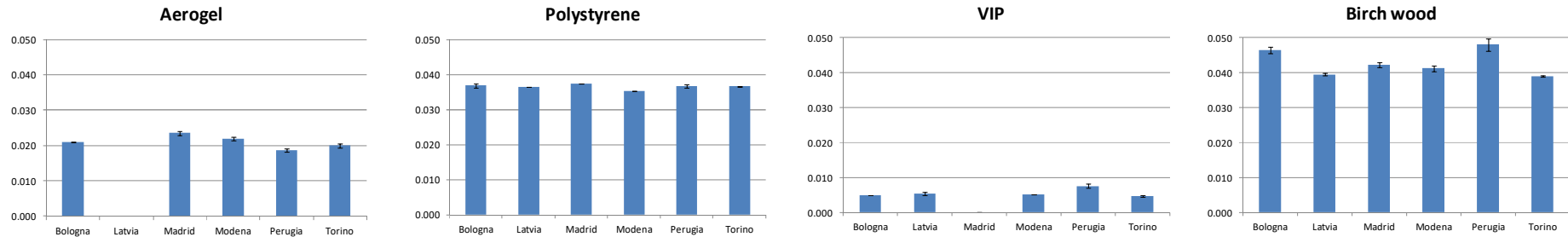


Figure 7 – Corrected thermal conductivity average value [W/m·K] and standard deviation for each laboratory.

All values retrieved appear aligned to what expected from the type of materials tested.

The data consistency analysis confirms that all tests may be considered reasonably consistent, as graphs for k and h reported in figures 8 and 9 demonstrate.

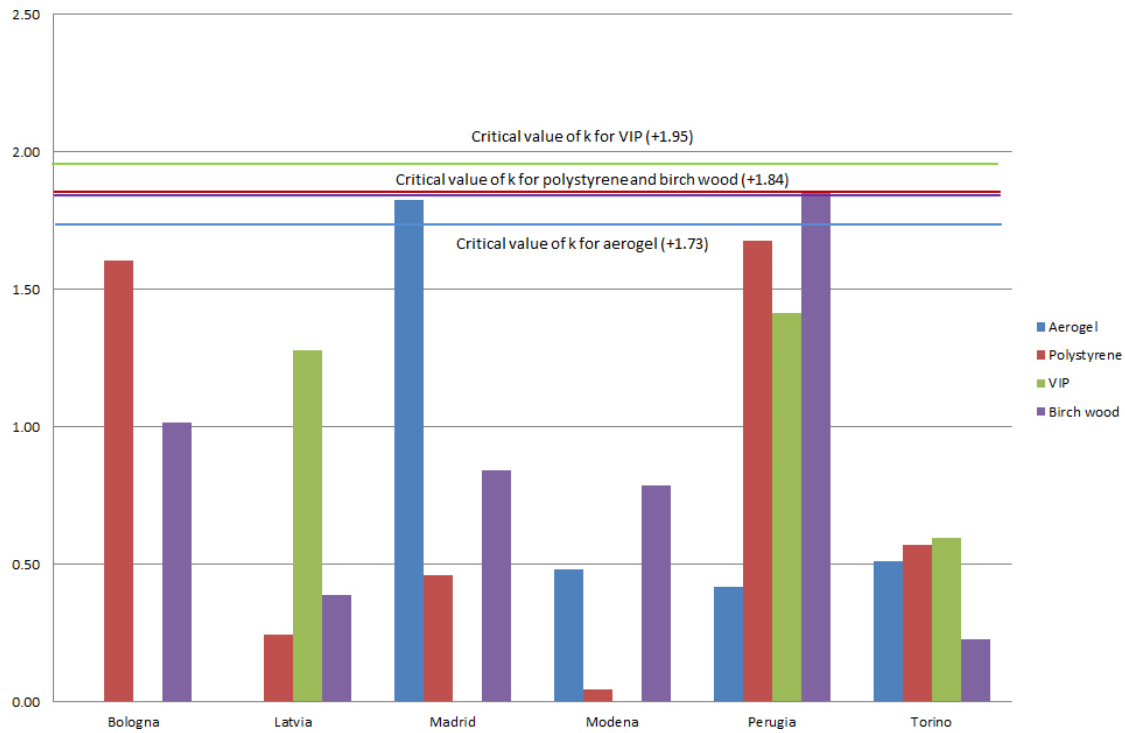


Figure 8 - Within-laboratory consistency k .

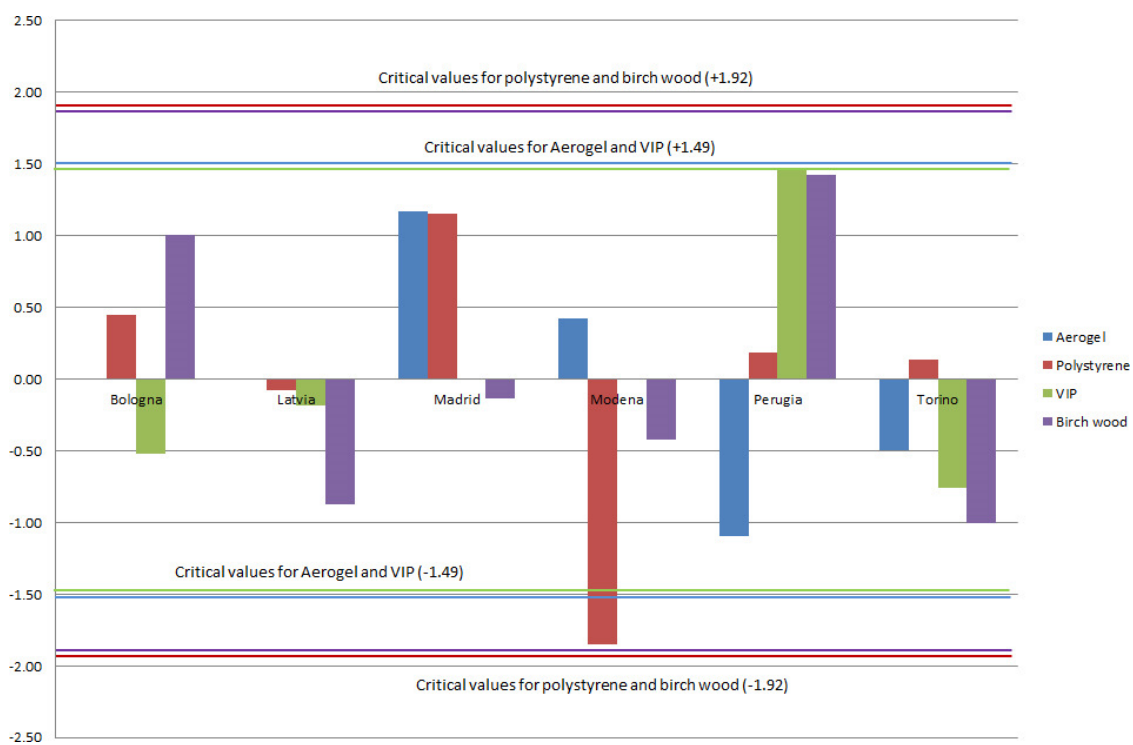


Figure 9 - Between-laboratory consistency h .

Table 4 shows the average of the averages of the thermal conductivity $\bar{\lambda}$ for each material, coupled with the statistical results.

Table 4 – Results of all tests conducted in the RRT (thermal conductivity in W/m K).

	Average of the cell averages	Standard deviation of the cell averages	Relative standard deviation of the cell averages	Repeatability standard deviation	Relative repeatability standard deviation	Reproducibility standard deviation	Relative reproducibility standard deviation	95% Repeatability	Relative 95% repeatability	95% Reproducibility	Relative 95% reproducibility
Aerogel	0.0211	0.00211	10.0%	0.00115	5.5%	0.00233	11.1%	0.00323	15.4%	0.00653	31.0%
Polystyrene	0.0365	0.00069	1.9%	0.00031	0.9%	0.00074	2.0%	0.00087	2.4%	0.00208	5.7%
VIP	0.0057	0.00131	23.1%	0.00039	6.9%	0.00134	23.6%	0.00110	19.4%	0.00376	66.1%
Wood chips	0.0427	0.00366	8.6%	0.00092	2.2%	0.00375	8.8%	0.00257	6.0%	0.01050	24.6%

From the analysis of parameter k the accuracy of the single instrumentation of each material can be evaluated. Each measurement performed by each laboratory should be expressed with his level of uncertainty usually defined by instrumentation accuracy or calculated by means the error propagation theory [32] taking into account the measurement chain. Table 5 reports the uncertainty for each device used in the tests.

Table 5 – Uncertainties declared for the instrumentations used in the tests.

Partner	Device manufacturer and model	Uncertainty
University of Perugia	University of Perugia and University of Roma "La Sapienza" – Own built	Around 5%
University of Latvia	Taurus - TCA 500-P	Around 5%
University of Bologna	University of Bologna	Around 4%
Politecnico di Torino	LaserComp FOX600	Around 2%
University of Modena and Reggio Emilia	University of Modena and Reggio Emilia – Own built	The highest of $\pm 0.002 \text{ W}/(\text{m} \cdot \text{K})$ and 2%
Universidad Politecnica de Madrid	Netzsch - Heat Flow Metter HFM436/3 Lambda Model	Around 3%

However, the statistical analysis for the interlaboratory study has been done according to the Standard ASTM E691 [25], where the standard deviation and the within-laboratory analysis represent the quality of the measurement for each laboratory.

It is showed that, the repeatability inside each laboratory is quite satisfying for all materials (relative repeatability standard deviation ranging from less than 1% up to a maximum of around 7%), confirming results obtained from another interlaboratory study [33]. As regards reproducibility, the RRT outcomes show a relative reproducibility standard deviation not far from 10% for all the samples, except for VIPs, which are characterised by a value of around 25% and the correspondent relative 95% reproducibility of around 70%. The reasons for the low performance of reproducibility for VIPs has to be searched firstly in the very low value of its apparent thermal conductivity, making more influent the absolute instrument uncertainties on the

final values. Secondly, the samples available were only two, instead of the four tested for the other materials, and one laboratory could not perform the measurements because of dimensions issues: all these circumstance limited the number of tests executed. Finally, as showed in the work of Fantucci et al. [34], the VIPs thermal performance is strongly affected by ageing even of a few years and as the RRT lasted more than two years, also the latter may constitute a reason for the VIPs low reproducibility.

4. Conclusions

The Round Robin Test executed for the measurement of innovative insulating materials thermal conductivity involved six laboratories all over Europe. Four different materials (aerogel, vacuum insulation panels, birch wood fibre insulation boards and polystyrene) were chosen, for a total of fourteen samples. A measurement protocol procedure was shared among the partners, at the aim of limiting the possible physical sources of differences in results.

The statistical analyses showed that all laboratories performed adequately both in terms of within-laboratory consistency and between-laboratory consistency, once the correction for different measurement average temperatures is implemented.

The results demonstrated that the hot plate devices, despite the different configurations used in the Round Robin Test, reveal themselves all as useful tools to assess the thermal conductivity of insulating innovative materials. In particular, the repeatability values indicate good performance for all the laboratories and sample, with a relative repeatability standard deviation not higher than 7% in the worst case.

As regards the reproducibility, aerogel, birch wood fibre insulation boards panels and polystyrene registered a satisfactory upper limit of around 10% for the relative reproducibility standard deviation, while vacuum insulation panels, the most insulating samples, present a value close to 25%, which is quite high, at least in relative terms.

Although this low reproducibility could be partly due to the limited number of samples available and to the lack of the results of one lab, it comes to light that data for materials with low thermal conductivities are subjected to higher deviations respect to less insulating ones.

Therefore, designers must handle with particular care these products, as the data available may be subjected to significant uncertainty.

The procedure used in this work appears reasonably robust and suitable to be extended to other materials, or other testing conditions (average temperature, humidity content, pressure on the sample), with the purpose of better covering the possible conditions of use of insulating materials.

References

- [1] A. Ioannou, L. C. M. Itard, Energy performance and comfort in residential buildings: Sensitivity for building parameters and occupancy, *Energy and Buildings* 92 (2015) 216-233.
- [2] F. Asdrubali, F. D'Alessandro, S. Schiavoni, A review of unconventional sustainable building insulation materials, *Sustainable Materials and Technologies* 4 (2015) 1-17.
- [3] S. T. Nguyen, A. D. Tran-Le, M. N. Vu, Q. D. To, O. Douzane, T. Langlet, Modeling thermal conductivity of hemp insulation material: A multi-scale homogenization approach, *Building and Environment* 107, (2016) 127-134.
- [4] Heat, air and moisture control in building assemblies – material properties, in: *ASHRAE Handbook of Fundamentals*, American Society of Heating, Refrigerating and Air Conditioning Engineers, Atlanta, 2009 (Chapter 26).
- [5] ISO 10456, Building Materials And Products – Tabulated Design Values and Procedures for Determining Declared and Design Thermal Values, International Organization for Standardisation, Geneva, Switzerland: International Organization for Standardization; 2007.
- [6] F. F. Domínguez-Muñoz, B. Anderson, J. M. Cejudo-López, A. Carrillo-Andrés, Uncertainty in the thermal conductivity of insulation materials, *Energy and Buildings* 42 (2010) 2159-2168.
- [7] A. A. Abdou, I. M. Budaiwi, The variation of thermal conductivity of fibrous insulation materials under different levels of moisture content, *Construction and Building Materials* 43 (2013) 533–544.
- [8] F. D'Alessandro, G. Baldinelli, F. Bianchi, S. Sambuco, A. Rufini, Experimental assessment of the water content influence on thermo-acoustic performance of building insulation materials, *Construction and Building Materials* 158 (2018) 264–274.
- [9] R. Ji, Z. Zhang, L. Liu, X. Wang, Development of the random simulation model for estimating the effective thermal conductivity of insulation materials, *Building and Environment* 80 (2014) 221-227.

- [10] K. S. Reddy, S. Jayachandran, Investigations on design and construction of a square guarded hot plate (SGHP) apparatus for thermal conductivity measurement of insulation materials, *International Journal of Thermal Sciences* 120 (2017) 136-147.
- [11] ISO 8302, Thermal insulation: determination of steady state thermal resistance and related properties: guarded hot plate apparatus. Geneva, Switzerland: International Organization for Standardization; 1991.
- [12] ASTM 2013 C177-13, Standard test method for steady-state heat flux measurements and thermal transmission properties by means of the guarded hot plate apparatus, *Annual Book of ASTM Standards*, West Conshohocken, Pennsylvania.
- [13] C. S. Sanjaya, T. H. Wee, T. Tamilselvan, Regression analysis estimation of thermal conductivity using guarded-hot-plate apparatus, *Applied Thermal Engineering* 31 (2011) 1566-1575.
- [14] J. Xamán, L. Lira, J. Arce, Analysis of the temperature distribution in a guarded hot plate apparatus for measuring thermal conductivity, *Applied Thermal Engineering* 29 (2009) 617-623.
- [15] C. J. Dey, A. J. Read, R. E. Collins, M. Brunotte, A guarded cold plate apparatus for absolute measurement of heat flow, *International Journal of Heat and Mass Transfer* 41 (2017) 3099-3108.
- [16] EN 12664, Thermal performance of building materials and products – Determination of thermal resistance by means of guarded hot plate and heat flow meter methods - Dry and moist products of medium and low thermal resistance, European Committee for Standardization, Brussels, Belgium, 2001.
- [17] EN 12667, Thermal performance of building materials and products - Determination of thermal resistance by means of guarded hot plate and heat flow meter methods - Products of high and medium thermal resistance, European Committee for Standardization, Brussels, Belgium, 2001.
- [18] M. Lachheb, Z. Younsi, H. Naji, M. Karkri, S. Ben Nasrallah, Thermal behavior of a hybrid PCM/plaster: A numerical and experimental investigation, *Applied Thermal Engineering* 111 (2017) 49-59.
- [19] R. E. Collins, C. A. Davis, C. J. Dey, S. J. Robinson, J. -Z Tang, G. M. Turner, Measurement of local heat flow in flat evacuated glazing, *International Journal of Heat and Mass Transfer* 36 (1993) 2553-2563.
- [20] E. Rasmussen, C. Stacey, A. Koenen, E. L. W. Kurkinen, Comparative testing of thermal conductivity for thermal insulation products – the European Keymark experience for more than 50 equipment at 25

test institutes, 32nd International Thermal Conductivity Conference 20th International Thermal Expansion Symposium April 27– May 1, 2014 Purdue University, West Lafayette, Indiana, USA.

- [21] H. P. Ebert, F. Hemberger, Intercomparison of thermal conductivity measurements on a calcium silicate insulation material, *International Journal of Thermal Sciences* 50 (2011) 1838-1844.
- [22] R. Schreiner, E. G. Hencke, H. P. Ebert, Intercomparison of thermal conductivity measurements on an expanded glass granulate in a wide temperature range *International Journal of Thermal Sciences* 95 (2015) 99-105.
- [23] IEA-ECBCS ANNEX 65 – Long-Term Performance of Super-Insulating Materials in Building Components and Systems - Subtask 2 Report: Characterisation of materials & components - Laboratory Scale (2017). Authors: C. Sprengard, S. Treml, A. Lorenzati, C. Cucchi. Editors: A. Holm, C. Sprengard (In-Press).
- [24] ASTM 2000 C1484-00, Standard Specification for Vacuum Insulation Panels, *Annual book of ASTM Standards*, Vol. 04.06, West Conshohocken, Pennsylvania.
- [25] ASTM 1999 E691-99. Standard practice for conducting an interlaboratory study to determine the precision of a test method, *Annual Book of ASTM Standards*, West Conshohocken, Pennsylvania.
- [26] <http://www.aktarusgroup.com/en/nanotechnology/aerogel/> (retrieved on December 2017).
- [27] <http://www.porextherm.com/en/products/vacupor/vacupor-xps-b2-s.html> (retrieved on December 2017).
- [28] <http://www.finieris.com/en/about-latvijas-finieris> (retrieved on October 2017).
- [29] ASTM C518-17, Standard Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus, *Annual Book of ASTM Standards*, West Conshohocken, Pennsylvania.
- [30] ASTM C1784-14, Standard Test Method for Using a Heat Flow Meter Apparatus for Measuring Thermal Storage Properties of Phase Change Materials and Products, *Annual Book of ASTM Standards*, West Conshohocken, Pennsylvania.
- [31] ASTM E177-04, Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods, *Annual Book of ASTM Standards*, West Conshohocken, Pennsylvania.
- [32] R. J. Moffat, Describing the Uncertainties in Experimental results, *Experimental Thermal and Fluid Science* 1 (1988) 3-17.

[33] H. P. Ebert, F. Hemberger, Intercomparison of thermal conductivity measurements on a calcium silicate insulation material, *International Journal of Thermal Sciences* Volume 50 (2011) 1838-1844.

[34] S. Fantucci, A. Lorenzati, A. Capozzoli, M. Perino, Analysis of the temperature dependence of the thermal conductivity in Vacuum Insulation Panels, *Energy and Buildings* 183 (2019) 64-74.