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IRMM information
REFERENCE MATERIALS

Certification of a Resin-Bonded Glass Fibre Board for Thermal Conductivity between – 10 °C and + 50 °C

IRMM-440

S. QUIN[(1)], G. VENUTI[1)], F. DE PONTE[(2)], A. LAMBERTY[(3)]

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B-2440 Geel, Belgium

Directorate-General
Joint Research Centre

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Mission

The mission of IRMM is to promote a common European measurement system in support of EU policies, especially internal market, environment, health and consumer protection standards.

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ABSTRACT

In order to replace BCR CRM 064, the Institute for Reference Materials and Measurements (IRMM) has proposed to create a new European certified reference material for the thermal conductivity measurements IRMM-440.
A specific production batch of high density (72.5 kg/m^3) resin-bonded glass fibre boards was manufactured. Measurements were performed on the glass fibre boards of 1.200 m x 1.200 m x 35 mm nominal dimensions in order to assess the homogeneity and the stability of the batch.

Before performing the certification measurements, the six selected laboratories were requested to assess their Guarded Hot Plate apparatus.
Each participant carried out the thermal conductivity measurements for the certification of the batch, according to ISO 8302 on two samples from the batch of glass fibre boards with different levels of density (low and high).

The statistical and technical analyses of the thermal conductivity measurements show that the best fit of the thermal conductivity measurements λ versus the mean test temperature θ is not a straight line (as former BCR-064) but a polynomial with 2nd or 3rd order temperature terms. The analyses show that the certification measurements from all the participating laboratories (except DFT for an outlying density) are acceptable. However, before computing the certified thermal conductivity values, all the measurements have been corrected from the identified systematic errors in order to reduce the statistical significant differences between the participants.

ABBREVIATIONS AND SYMBOLS

CRIR	Centre de Recherches Industrielles de Rantigny
CRM	Certified Reference Material
DFT	Dipartimento di Fisica Tecnica
EMPA	Eidgenössische Materialprüfungs- und Forschungsanstalt
FIW	Forschungs-Institut für Wärmeschutz
GHP	Guarded Hot Plate
HFM	Heat Flow Meter
IRMM	Institute for Reference Materials and Measurements
LNE	Laboratoire National d'Essais
NPL	National Physical Laboratory
SP	Sveriges Provnings- och Forskningsinstitut

d	specimen thickness [mm or m]
λ	thermal conductivity [mW/(m.K) or W/(m.K)]
θ	mean test temperature [°C]
ρ_{spec}	specimen density [kg/m^3]

CONTENTS

1. INTRODUCTION

The certified reference material BCR-064 (resin bonded glass fibre board), which certified property is the thermal conductivity [1], is now sold out.

BCR-064 was manufactured by ISOVER SAINT GOBAIN through the TEL® process which was substituted by an improved one, TELSTAR®, with different process parameters and product properties. Therefore, it was impossible to manufacture a new batch of BCR-064 with the same material specifications and the decision was taken to replace BCR-064 by a new one IRMM-440.

2. CERTIFICATION PROCEDURE

In order to replace BCR-064, the Institute for Reference Materials and Measurements (IRMM) initiated a certification project involving six internationally recognized experts in the field of thermal conductivity measurements and are all members of the CEN-TC 89W68. This project was to characterize a new batch of glass fibre boards by thermal conductivity measurements using a Guarded Hot Plate (GHP) apparatus.

On behalf of IRMM, the requirements and the specifications for the production of a new reference material were studied by the Laboratoire National d'Essais (LNE).
A specific production run was made by ISOVER SAINT GOBAIN and a preliminary analysis was made by the producer in order to verify the conformity of the boards with thickness and density specifications.
Then, the assessment of the batch was performed by LNE with the help of the Dipartimento di Fisica Tecnica (DFT) of the University of Padova (Italy).

Before performing the certification measurements, the participating laboratories were requested by IRMM to assess their Guarded Hot Plate apparatus according to Draft CEN/TC 89/WG8 N112 [2].
Subsequently, each participant carried out the thermal conductivity measurements for the certification of the batch on two samples from the batch of glass fibre boards with different levels of density (low and high).
All the participants provided a technical report on the assessment of their GHP equipment and a measurement report on the thermal conductivity measurements. These reports were sent to LNE for assessment.

A first statistical analysis of the results from all the participating laboratories shows that the best fit of the thermal conductivity measurements λ versus the mean test temperature θ is not a straight line (as former BCR-064) but a polynomial with 2nd or 3rd order temperature terms.
These non-linearities on the curve λ versus θ can be explained by:

- measurement errors resulting from GHP apparatus,
- physical phenomena, especially moisture in the fibrous material during the test.

In order to check the validity of the second hypothesis, theoretical calculations, using interpolation physical models, were performed by LNE and DFT.

3. PARTICIPANTS

Six international laboratories participated in the certification project. These laboratories and the name of the persons in charge of the work are as follows:

Dipartimento di Fisica Tecnica (DFT)
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All the participating laboratories performed the certification measurements.
On behalf of IRMM, LNE was in charge of the co-ordination of the project with the help of IRMM, including the material assessment (homogeneity and stability testing) and the data analysis. DFT took part in the homogeneity study and the results analysis through interpolation models.

4. MATERIAL

4.1. Definition of the reference material

4.1.1. Choice of the reference material

In order to manufacture a batch of material intended to become a Certified Reference Material (CRM), the requirements for the new reference material related to thermal properties were defined as follows:

- long-term stability and sufficient homogeneity of the material;
- limited "thickness effect" (i.e. increase of measured thermal conductivity with thickness due to contribution of radiation to the total heat transfer),
- limited change of the thermal conductivity versus the density,
- improved thickness uniformity over one single specimen to be in conformity with the criteria of the International Standards: ISO 8301 [3] and ISO 8302 [4],
- sufficient amount of material to cover the expected needs for the next ten years.

As the long-term stability of glass fibre boards is proven to be excellent, and the TELSTAR® manufacturing process has improved capabilities (fibre diameter and length, density and thickness distributions), it was suggested to replace BCR-064 by a new batch of the same kind of material (i.e. high density glass fibre boards), manufactured by ISOVER SAINT GOBAIN.
Concerning the amount of material, over the past years an average of 15 specimens of BCR-064 were annually sold with a maximum not exceeding 30 specimens. Assuming that a minimum of 2 specimens can be obtained in each board, then a batch of approximately 150 / 200 boards will cover the needs over the next 10 years.

4.1.2. Specifications for production

According to the optimum capability of the manufacturing process and the requirements for the new material, the specifications for the production of the batch of the reference material were defined as follows:

- size of boards: 1.200 m x 1.200 m
- number of boards: 270
- nominal thickness: 35 mm
- flatness tolerances: 0.4 mm
- maximum deviation from parallelism for specimen surfaces: 2 % of specimen thickness
- nominal density: 70 kg/m^3
- range around nominal density ± 5 kg/m^3 between boards
- fineness index (*): 3.6 / 5 g
- maximum thickness effect: 0.5 % of thermal conductivity value
- maximum reproducibility of thermal conductivity at given density: ± 1 %.

4.2. Manufacturing of the reference material

A specific production batch of high density resin bonded glass fibre boards was manufactured by ISOVER SAINT GOBAIN in May 1996 on the 2.4 m wide production line through the TELSTAR® process.

(*) The fineness index is defined from particular air-flow permeability measurement on randomly compacted glass fibre specimen with a given mass introduced in rigid cylindrical cell of fixed dimensions. It is a reference figure without any physical meaning [5]

4.2.1. Controls on the production line

Density and fineness index measurements were carried out on the material in production to eliminate the boards which were outlying from specifications.
Then, the boards were cut into 1.200 m x 1.200 m nominal dimensions and were sandpapered on both faces to eliminate the marks made by the oven conveyor belt in order to minimize the flatness defaults.

4.2.2. Controls performed by CRIR

The research centre of ISOVER SAINT GOBAIN (CRIR) carried out physical and thermal measurements on 9 boards from the batch.

a) *Thickness measurements*
The measurements were performed on 18 specimens of 0.600 m x 0.600 m nominal dimensions.
The specimen thickness was measured at 9 equidistant locations under a pressure of 765 Pa with a 49.7 mm diameter measuring plate.
The average thickness of the 18 specimens is 35.1 mm and the thickness range on individual specimens is between 0.25 mm and 0.75 mm.

b) *Density distribution on the specimens*
The heterogeneity in density within individual 0.600 m x 0.600 m specimens was estimated from measurements on the 18 specimens cut into 0.200 m x 0.200 m small samples.
The density measurements were performed on the 9 small samples from each specimen.
The average density of the 18 specimens is 73 kg/m^3 and the density range on individual specimens is between 10.4 kg/m^3 and 23.3 kg/m^3.

c) *Fibre diameter measurements*
The fibre diameter measurements were performed with a scanning electron microscope.
The average fibre diameter is 5.4 μm and the standard deviation is 3.9 μm.

d) *Binder content measurements*
The binder content measurements were carried out on 5 specimens.
The average binder content is 8.3 % in mass.

e) *Air flow permeability measurements*
The air flow permeability measurements were performed using a 0.300 m diameter perpendicular air flow permeability apparatus with a filtration speed of 44.10^{-3} $m.s^{-1}$ and an air flow of 186.7 $l.mn^{-1}$.
The average specific resistance is 56.9 Rayl/cm, i.e. the air flow permeability is $3.2.10^{-10}$ m^2.

f) *Thermal conductivity measurements*
The measurements were performed on 5 specimens of 0.600 m x 0.600 m nominal dimensions.
The thermal conductivity measurements were carried out at 10 °C according to ISO 8301 using a Heat Flow Meter (HFM) apparatus (with a 0.305 m x 0.305 m metering area).

The average thermal conductivity of the 5 specimens (of apparent density within the range [69 kg/m^3 - 77 kg/m^3]) is 31.0 mW/(m.K) and the corresponding range is 0.3 mW/(m.K).

g) *Thickness effect*
CRIR investigated the thickness effect due to radiation for this type of material (3.6/5 g fineness index and 70 kg/m^3 density). The results analysis shows that the thickness effect is not relevant for 35 mm thickness boards.

4.3. Testing of the batch

A total of 282 glass fibre boards was supplied. The boards were packaged by 6 in boxes and delivered in containers of 6 boxes.
Each board of the batch is identified with the following code:

- a letter " A " to " I " corresponding to a given container,
- a 1st serial number " 1 " to " 6 " corresponding to a given box,
- a 2nd serial number " 1 " to " 6 " corresponding to a board in a given box.

4.3.1. Preliminary assessment

A preliminary characterization of the batch was made by LNE in order to verify if the reference material meets the specifications.

a) Characterization of the batch by thickness and density measurements

The characterization was performed on the boards without any specific conditioning before testing.

- Thickness measurements

The thickness of the boards was measured under a pressure of 250 Pa with a 0.200 m square measuring plate. On each board, the measurements were taken at 4 equidistant locations.
The histogram of thickness distribution of the boards is given in Annex 1 (Figure A1a).
The average thickness of the whole batch is 35.18 mm and the corresponding standard deviation is 0.08 mm.
The range of thicknesses on individual boards was also defined. The corresponding histogram of thickness range distribution is given in Annex 1 (Figure A1b). The average value is 0.21 mm and the corresponding standard deviation is 0.10 mm.

- Deviation from flatness

The deviation from flatness was estimated on a set of 10 boards randomly selected in the batch. On each board, the measurements were taken at 36 equidistant locations under a pressure of 2800 Pa with a 48 mm diameter measuring plate.
The results analysis shows that the maximum deviation from flatness (high or low value) is mostly found on the edges of the boards. This deviation is much lower when the measurements on the edges of the boards are not taken into account:

- the maximum deviation over the total area of the board is within 0.5 mm - 0.7 mm
- the maximum deviation without the edges is within 0.3 mm - 0.4 mm.

- Density measurements

The apparent density of the boards were calculated from the measurements of mass, length and width of the boards and from the thickness measurements as described previously.
The histogram of density distribution of the boards is given in Annex 1 (Figure A1c).
The average apparent density of the whole batch is 71 kg/m^3 and the corresponding standard deviation is 1.36 kg/m^3. The density range between boards is 8.85 kg/m^3.

b) Density distribution on the boards

The heterogeneity in density within individual boards was estimated from measurements on 3 different boards cut into 0.200 m x 0.200 m small samples.
The results analysis shows that the density range on individual boards is between 13.1 kg/m^3 and 22.5 kg/m^3.

c) Thermal conductivity measurements

The measurements were performed on 16 specimens of 0.600 m x 0.600 m nominal dimensions at different densities and different mean temperatures. The thermal conductivity measurements were carried out on the one hand according to ISO 8301 using a single specimen HFM apparatus, and on the other hand according to ISO 8302 using a two specimen GHP apparatus.
The results are given in Annex 1 (Table A1). The first analysis of thermal conductivity results versus density do not show any dependence of thermal conductivity with density.

d) Outcome of the preliminary assessment

The results analysis shows that the batch of material produced by ISOVER meets the specifications.

4.3.2. Homogeneity assessment

Specific measurements were performed by DFT and LNE on specimens from the batch in order to assess the degree of homogeneity of the reference material.

a) Sampling

A sample of 24 glass fibre boards was systematically selected from the batch. In order to cover the density range and the whole boards from the batch, the selection was based on two criteria: the density level and the identification code of the boards.
The selected boards were divided up among both laboratories as follows:
- DFT: 4 boards cut into 16 specimens of 0.600 m x 0.600 m nominal dimensions,
- LNE: 20 boards cut into 40 specimens of 0.600 m x 0.600 m nominal dimensions.

b) Specimen thickness distribution

The thickness measurements were performed by both laboratories under different pressures not exceeding 2000 Pa.

• DFT measurements

The thickness of the 16 specimens was measured under two pressures (164 Pa and 1170 Pa respectively) with a 0.500 m x 0.500 m measuring plate. On each specimen, the measurements were taken at the 4 corners of the measuring plate.

The histograms of thickness distribution of the 16 specimens for both pressures are given in Annex 2 (Figure A2a and Figure A2b). The average thickness is 35.20 mm and 34.65 mm respectively (at 164 Pa and 1170 Pa respectively) and the corresponding standard deviation is 0.13 mm and 0.075 mm respectively.

The thickness range on individual specimens was also defined. The corresponding histogram of thickness range distribution for both pressures is given in Annex 2 (Figure A2c). The average value is 0.24 mm and 0.16 mm respectively and the corresponding standard deviation is 0.10 mm and 0.08 mm respectively.

• LNE measurements

The thickness of the 40 specimens was measured under a pressure of 1500 Pa with a 0.610 m x 0.610 m measuring plate. On each specimen, the measurements were taken at the 4 corners of the measuring plate.

The histogram of thickness distribution of the 40 specimens is given in Annex 2 (Figure A2d).The average value is 34.44 mm and the standard deviation is 0.05 mm.

The thickness range on individual specimens is represented by the histogram given in Annex 2 (Figure A2e). The average value is 0.28 mm and the corresponding standard deviation is 0.10 mm.

• Analysis of results

The results analysis of the thickness measurements shows that a pressure change from 164 Pa to 1500 Pa introduces a thickness change of 2 % due to the compression of the material.

The specimen thickness distribution shows that the thickness is very consistent from specimen to specimen with a variability lower than 0.2 % when measuring under 1000 Pa and 1500 Pa pressures (0.4 % under a pressure of 164 Pa).

Moreover, for the certification measurements, it was decided to measure the specimen thickness under a pressure corresponding to the one which is applied in the apparatus during the thermal conductivity measurements, i.e. between 1000 Pa and 2000 Pa. This pressure range ensures adequately uniform contact between the specimens and the cold and hot plates of the apparatus.

In addition, in order to avoid the thickness change during the measurements, the thermal conductivity measurements had to be carried out with spacers fixing the specimen thickness.

c) Specimen density distribution

The apparent density measurements were carried out by both laboratories on the specimens. The apparent density results were calculated from the measurements of the mass, length and width of specimens and from the thickness measurements performed under 1170 Pa and 1500 Pa pressures.

• DFT measurements

The histogram of apparent density distribution of the 16 specimens for a pressure of 1170 Pa is given in Annex 2 (Figure A2f). The average density is 71.9 kg/m^3 and the corresponding standard deviation is 2.6 kg/m^3. The density range between specimens is 8.5 kg/m^3.

• <u>LNE measurements</u>
The histogram of apparent density distribution of the 40 specimens for a pressure of 1500 Pa is given in Annex 2 (Figure A2g). The average density is 72.5 kg/m^3 and the corresponding standard deviation is 3.2 kg/m^3. The density range between specimens is 14.8 kg/m^3.

d) Thermal conductivity distribution

The thermal conductivity measurements were carried out by both laboratories according to ISO 8301 using a single specimen HFM apparatus.
The long-term stability of both HFM apparatuses, used by DFT and LNE, has been proven by periodic measurements performed on reference material specimens. The reproducibility of thermal conductivity measurements of both HFM apparatuses is lower than 0.2 % (relative standard deviation: 0.17 % for DFT and 0.15 % for LNE).
All the thermal conductivity measurements were performed at 20 °C with thicknesses fixed by spacers, which were set between the hot and cold plates of the apparatus.

• <u>DFT measurements</u>
The histogram of thermal conductivity distribution of the 16 specimens is given in Annex 2 (Figure A2h). The average thermal conductivity is 32.02 mW/(m.K) and the corresponding standard deviation is 0.13 mW/(m.K). The thermal conductivity range between specimens is 0.52 mW/(m.K).

• <u>LNE measurements</u>
The histogram of thermal conductivity distribution of the 40 specimens is given in Annex 2 (Figure A2i). The average thermal conductivity is 31.47 mW/(m.K) and the corresponding standard deviation is 0.13 mW/(m.K). The thermal conductivity range between specimens is 0.51 mW/(m.K).

e) Outcome of the homogeneity study

The plots of the thermal conductivity measurements versus the specimen density, as performed by DFT and LNE, are given in Annex 2 (Figure A2j and Figure A2k). The results do not show any remarkable dependence of thermal conductivity with density.

The results of the homogeneity study performed by DFT and LNE are summarized in Annex 2 (Table A2). The observed variability between specimens is:
- within 0.2 % for the thickness,
- within 4 % for the density,
- within 0.4 % for the thermal conductivity, compared to the reproducibility of HFM apparatuses which is lower than 0.2 %.

These results show that slight discrepancies are detected as regards to the certified property (thermal conductivity), and the homogeneity is better than excepted.

4.3.3. Stability assessment

In order to assess the long-term stability of the thermal conductivity of the reference material with time, LNE has carried out periodical measurements on specimens from the batch.

a) Sampling
A sample of 5 glass fibre boards was randomly selected from the batch.

The selected boards were cut into 6 specimens of 0.600 m x 0.600 m nominal dimensions.

b) Measurement method

The thermal conductivity measurements were performed at 20 °C according to ISO 8301 using the LNE single specimen HFM apparatus which has a reproducibility within 0.15 % (relative standard deviation: 0.15 % - range: 0.21 mW/(m.K)).
The thermal conductivity measurements were regularly carried out on the 6 specimens over a period of 36 months from the manufacturing.

c) Outcome of the stability study

The plot of the thermal conductivity measurements versus the age of the reference material is given in Annex 3 (Figure A3).
The results of the stability study are summarized in Annex 3 (Table A3).
The observed variability of the thermal conductivity measurements with time is due to the HFM variability. The results do not show any variation of thermal conductivity with time, ensuring thus the long-term stability of the certified property.

4.3.4. Synthesis of the assessments

The lower observed thermal conductivity variability between specimens from the batch and the independence of the certified property with specimen density and with time show that the batch of reference material is really suitable for the replacement of BCR-064: the thermal conductivity will only depend on the temperature.

5. MEASUREMENT PROCEDURES

The following measurement procedures were adopted by all the participating laboratories during the certification measurements.

5.1. Specimen thickness measurements

Each specimen was put on a reference table and was gently covered by a measuring plate. The flatness default of the table and the measuring plate should be lower than 0.05 mm.
The nominal dimensions of the measuring plate was equal or slightly larger than the specimen itself and its mass was such as the pressure applied on the specimen was the same as the one in the apparatus during the thermal conductivity measurements.
The current working pressure for GHP or HFM apparatuses is from 1000 Pa up to 2000 Pa. This pressure range ensures adequately uniform contact between the specimen and the cold and hot plates of the apparatus.
The thickness of each specimen was measured at the four corners of the measuring plate using gauge comparators or micrometers.

5.2. Specimen density measurements

The specimen apparent density was calculated from the measurements of the mass, length and width of specimens and from the thickness measurements as described previously. These physical characteristics were measured after the conditioning of the specimens in the surrounding environment (standard laboratory atmosphere) to reach equilibrium with the environment (constant mass).
The specimen apparent density is given by dividing the mass by its volume (length x width x thickness).

5.3. Thermal conductivity measurements

The thermal conductivity measurements were carried out according to the International Standard ISO 8302 using a Guarded Hot Plate (GHP) Apparatus.
In order to avoid thickness change during the measurements (due to the compressibility of the material), the specimen thickness was fixed using spacers or plate separation.
The principle of thermal conductivity measurements (using the example of a two specimen GHP apparatus) and the expression of results are given in Annexe 4.

6. CERTIFICATION MEASUREMENTS

6.1. Measurement protocol

Before performing the thermal conductivity measurements for the certification of the batch, the participating laboratories were requested by IRMM to carry out the metrological assessment of their Guarded Hot Plate apparatus.

6.1.1. GHP apparatus assessment

All the participating laboratories had assessed their Guarded Hot Plate Apparatus according to Draft CEN/TC 89/WG8 N112.
This document gives specific technical criteria for the assessment of laboratories to undertake steady state heat transfer property measurements by the Guarded hot Plate method. It specifies contents of the documentation required by EN 45001 [5] (equipment manual, calibration and maintenance files, measurement procedure document). It provides information on mandatory equipment performance specifications, equipment description and on calculations for the equipment design and error analysis. Then, it gives information on experimental procedures suitable for the assessment of instrument accuracy and on performance check.
Each participant provided a technical report on the assessment of their GHP equipment. A copy of these reports is given in Annex 8 and a summary of the technical information on GHP equipment of each participating laboratory is given in Annex 5 (Table A5a).

6.1.2. Thermal conductivity measurements

Each participant had to perform the certification measurements on two samples from the batch of glass fibre boards with different levels of density (low and high). Each sample consisted of one or two specimens of 0.035 m thickness. The specimen number per sample and the specimen nominal dimensions depended on the type and the dimensions of the equipment of each participating laboratory.

The thermal conductivity measurements had to be carried out, using the measurement procedures as described previously, at five (four for NPL) different mean test temperatures, as a minimum, within the range [- 10 °C / + 50 °C]. As requested by IRMM, three thermal conductivity measurements had to be carried out on each sample at the following mean test temperatures: - 10 °C / 0 °C / + 20 °C, except NPL who performed the measurements especially at 0 °C and + 20 °C ; the two remaining measurements, performed on each sample, had to be chosen within the range [- 10 °C / + 50 °C].

6.2. Presentation of the thermal conductivity results

Each participant provided a measurement report on the thermal conductivity measurements performed on both samples. A copy of these documents is given in Annex 8.

The thermal conductivity measurements were performed on the samples not enclosed in a vapour tight envelope. The measurements were carried out under a temperature difference from 10 K up to 20 K over the range [- 10 °C / + 50 °C], except DFT who performed the measurements over the range [- 170 °C / + 30 °C].

A summary of the experimental test conditions of the certification measurements performed by the participating laboratories is given in Annex 5 (Table A5b) and a synthesis of the thermal conductivity results reported by the participating laboratories is given in Annex 5 (Table A5c).
The plots of the thermal conductivity measurements from all the participating laboratories versus the mean test temperature are given in Annex 5 (Figure A5a and Figure A5b) over the temperature ranges [-170 °C / + 50 °C] and [-10 °C / + 50 °C].

7. EVALUATION OF THE THERMAL CONDUCTIVITY RESULTS

Before performing the technical scrutiny of the data, a preliminary statistical analysis of thermal conductivity results was carried out in order to assess the consistency and the quality of the data.

7.1. Preliminary statistical analysis of the thermal conductivity results

7.1.1. Comparison of the results

In order to compare the thermal conductivity results between the participating laboratories at the same temperature conditions, it was decided as a first hypothesis to interpolate the data to the exact temperature level using a linear interpolation.

The comparison of the thermal conductivity results on low and high density samples was performed at the following temperature levels: - 10 °C / 0 °C / + 10 °C / + 20 °C / + 30 °C / + 40 °C / + 50 °C. The following statistical calculations were performed to ascertain the compatibility between the various sets of data:

- the average and the standard deviation at the 95 % confidence interval of each set of data,
- the Cochran test to identify outlying variances,
- the Nalimov t-test to identify outlying data set averages,
- the Scheffe multiple t-test at the 0.05 and 0.01 significance levels to check whether any two sets of data could be considered as samples from the same population,
- the one-way analysis of variance to calculate the within- and between-laboratories standard deviations,
- the Snedecor F-test to check whether the between-laboratories standard deviation was significantly different from zero,
- the Bartlett test to check whether the differences between the variances were significant.

A summary of the statistical analysis results is given in Annex 6 (Table A6a). The results of the statistical analysis show that there are significant differences between sets of data at the 0.05 significance level, and for some temperature levels at the 0.01 significance level. The data sets are divided into two groups: DFT, FIW and SP on one side and EMPA, LNE and NPL on the other side, particularly at the extreme temperature levels.

7.1.2. Individual analysis of the results

A statistical analysis of the thermal conductivity λ versus the mean test temperature θ on low and high density samples was performed by least squares regression for each participating laboratory. For each series, the best fit was determined through the analysis of statistical parameters (standard error, correlation coefficients, residuals and trends analysis).
The results of the statistical analysis are given in Annex 6 (Table A6b). These results show that the best relationship between λ and θ over the temperature range [-10 °C / + 30 °C up to + 50 °C] is not a straight line (contrary to the best fit determined for former BCR-064) but is a polynomial with second or third order temperature terms.
These non-linearities on the curve λ versus θ can be explained by:

- measurement errors resulting from the GHP apparatus,
- physical phenomena, especially moisture in the fibrous material during the test.

The first hypothesis was evaluated through the analysis of the GHP assessment report of each participant and through a critical technical discussion between the participating laboratories on possible sources of errors in the thermal conductivity measurements.
To verify the validity of the second hypothesis, theoretical calculations, using interpolation physical models, were performed by LNE and DFT in order to assess the influence of the relative humidity on the thermal conductivity results.

7.2. Technical discussion

All the laboratories participated in a technical discussion. During this meeting, the certification measurements including experimental test conditions were inspected and the metrological assessment of each GHP equipment was discussed. Moreover, the results of the theoretical calculations using interpolation physical models were presented.

7.2.1. Technical scrutiny of data

Although the preliminary statistical analysis of the thermal conductivity measurements showed some significant differences between data sets, the acceptance of data sets to be used to calculate the certified values was decided on technical grounds.

a) Rejection of a data set on an outlying high density sample

When inspecting very closely the density levels of each measured sample, it appeared that DFT carried out the certification measurements on a 81 kg/m^{3}density sample. The density of this sample was far from the other high density samples, which were close to 75 kg/m^3.
Moreover, the slopes of the thermal conductivity results were compared. The plot of these slopes versus the density sample is given in Annex 6 (Figure A6a). This figure

shows that the slope of the 81 kg/m^3 density sample differs from all the other slopes, including the DFT slope on low density sample.
Considering the sample density ranges from 63.5 kg/m^3 to 78.5 kg/m^3, as given by the homogeneity testing, it was decided to discard from the batch the samples with densities greater than 80 kg/m^3. Thus, the thermal conductivity measurements performed by DFT on the high density sample have been rejected for the calculation of the certified values.

b) Rejection of a suspect measurement

When inspecting very closely the thermal conductivity measurements performed by LNE at - 10 °C mean test temperature on both samples, it appeared that the discrepancies between all the data were within 0.1 % except for one data. The observed relative difference between this suspect data and the other measurements (0.35 %) was higher than the reproducibility of LNE measurements (0.2 %). So, this data has been rejected for the calculation of the certified values.

c) Measurement errors

The reliability of individual sets of data from each participating laboratory was investigated through a critical review of the assessment of the GHP equipment and of the experimental test conditions.
It appeared that the discrepancies between the participants could be due to systematic measurements errors resulting from the linearity test analysis and from thickness measurements.
Therefore, it was decided to correct the thermal conductivity measurements (as described in section 8.1.) before calculating the certified values and their uncertainties.

7.2.2. Interpolation physical models

Theoretical calculations using interpolation physical models were carried out by LNE and DFT in order to validate the non-linearities on the curve λ versus θ.
Interpolation models of the heat transfer through dry mineral fibres insulation are developed in the CEN document prEN 12939 [6]. They are based on physical phenomena: conduction through solid matrix (fibres) and through dry air and radiation in the material. The resulting expression of the heat transfer is as follows:

$$\lambda_{mod} = \lambda_{cd} + \lambda_{rad}$$

where: – λ_{cd} is the combined thermal conductivity of dry air and fibres.
– λ_{rad} is the thermal conductivity due to radiation heat transfer.

The conductive component λ_{cd} depends on sample and glass density, glass and dry air thermal conductivity.
The radiative component λ_{rad} depends on testing conditions (temperature difference, apparatus emissivity), sample density and a specific scattering (or extinction) parameter (depending on temperature and fibre diameter).

a) LNE study

LNE developed a heat transfer model through moist fibrous insulation, derived from the previous basic models. This model takes into account a supplementary parameter: the moisture of the fibrous insulation. It assumes that the heat transfer is due to two mechanisms:

- conduction through fibres and through moist air,
- radiation in the material.

In this case, the conductive component λ_{cd} depends on sample and glass density, glass thermal conductivity and on moist air thermal conductivity. The moist air thermal conductivity is a combination of dry air thermal conductivity and water vapour thermal conductivity, depending on relative humidity and partial pressure of both gases.

Calculations were performed in order to determine the influence parameters on the model results. The preponderant parameters are on the one hand related to the sample characteristics: specific scattering parameter and sample density and on the other hand related to the experimental test conditions: relative humidity.

The heat transfer model was applied to the specific data of sample characteristics and of experimental test conditions for the certification measurements. The model results show that the best relationship between λ and θ is a third order temperature terms polynomial.
A copy of the synthesis document dealing with this model is given in Annex 8.

b) DFT study

DFT developed a similar heat transfer model through moist fibrous insulation (as described previously), but introduced a supplementary coefficient taking into account the effect of relative humidity on the conduction through the solid matrix (fibres).

The analysis of the certification measurements through the heat transfer model shows that the relative humidity of the air enclosed within the fibres affects the thermal conductivity of both moist air and solid matrix. But, both effects have opposite signs and partially compensate. However, the calculations prove that the thermal conductivity measurements performed by the participating laboratories are accurate and cannot be interpolated by a straight line.

Both laboratories confirm using interpolation physical models that the thermal conductivity versus the mean test temperature is not a straight line over the temperature range [-10 °C / + 50 °C] for the new reference material.

8. CERTIFIED THERMAL CONDUCTIVITY

Before calculating the certified values and their uncertainties, the thermal conductivity results were corrected taking into account both measurement errors (from linearity test analysis and from thickness measurements).

8.1. Corrected thermal conductivity results

8.1.1. Error resulting from the linearity test analysis

The corresponding error was identified through the analysis of the linearity tests which had been performed by the participating laboratories for the assessment of their GHP apparatus.

The linearity tests consist in performing thermal conductivity measurements at some relevant mean test temperatures under widely different temperature differences. In the best case, the thermal conductivity results are independent of the temperature difference, and the relationship between the electrical power dissipated in the metering area and the temperature difference is a straight line passing through the origin.
However, for most of the participants, it appeared that the thermal conductivity results depended on the temperature difference. The corresponding error could be due to combined effects of edge heat losses, bad setting of the imbalance sensors and temperature measurement errors.
This error was evaluated from the expression of the electrical power versus the temperature difference, determined by least square regression, given as follows:

$$P_{meas} = a \times \Delta T + b = P + \delta P$$

where: – P_{meas} is the measured electrical power.
- a, b are the linear regression coefficients (slope and intercept).
- ΔT is the temperature difference across the specimen.
- P (or $a \times \Delta T$) is the actual electrical power (= heat flow rate through specimens in the ideal conditions).
- δP (or b) is the resulting absolute error.

Note: the error analysis could also be performed using the similar expression with the thermal conductivity and the temperature difference:

$$\lambda_{meas} \times \Delta T = \lambda \times \Delta T + \delta\lambda$$

where: – λ_{meas} is the measured thermal conductivity.
- λ is the actual thermal conductivity (in the ideal conditions).
- $\delta\lambda$ is the resulting absolute error.

Thus, the absolute error (δP or $\delta\lambda$) was determined as described previously from the linearity test results given by each participating laboratory.

The relative errors on the thermal conductivity measurements, corresponding to the operating temperature difference during the certification measurements, are given in Annex 7 (Table A7a). Moreover, in order to correct the thermal conductivity results at each mean test temperature over the measurement range, the relative errors have been linearly interpolated, for each mean test temperature, from the previous results. These values are also given in Annex 7 (Table A7a).

8.1.2. Error resulting from the thickness measurements

The error in the specimen thickness measurement was identified through the analysis of the experimental test conditions, particularly the eventual correction for the thermal expansion of the spacers over the temperature range [-10 °C / + 50 °C].

For most of the participants, the thickness of the spacers, fixing specimen thickness, was only measured at the ambient temperature.
However, the thickness of the spacers could change over the temperature range [-10 °C / + 50 °C], depending on the nature of the material used for spacers.

So, it was decided to correct the thickness taking into account the spacers thermal expansion over the mean test temperature range. The spacer thickness was computed over the measurement range from the following expression:

$$d_\theta = \alpha \times d_{20°C} \times (\theta - 20) + d_{20°C}$$

where: – d_θ is the spacer thickness at the temperature θ.
– $d_{20°C}$ is the spacer thickness at the ambient temperature.
– α is the thermal expansion coefficient of spacers.

The values of specimen (or spacer) thicknesses are given in Annex 7 (Table A7b) for each participating laboratory.

The thermal conductivity results, corrected from both errors as described previously, are given in Annex 7 (Table A7b).

8.2. Statistical analysis of the corrected thermal conductivity results

In order to compare the thermal conductivity results between the participating laboratories at the same temperature conditions, the data were interpolated to the exact temperature level using the best fit of the thermal conductivity versus the mean test temperature.
Thus, for each series, the best fit was determined by least squares regression through the analysis of statistical parameters (standard error, correlation coefficients, residuals and trends analysis). The polynomials, which were determined for each laboratory, are given in Annex 7 (Table A7c).

The comparison of the thermal conductivity results on low and high density samples was performed at the following temperature levels: - 10 °C / 0 °C / + 10 °C / + 20 °C / + 30 °C / + 40 °C / + 50 °C.

Firstly, in order to verify the compatibility between all various sets of data, and hence to treat them as one single set of data (i.e. to pool all individual measurement results), the following statistical calculations were performed at the 0.05 and 0.01 significance levels:

- the Cochran test to identify outlying variances,
- the Dixon test, Nalimov t-test and Grubbs test to identify outlying data set averages,
- the Scheffe multiple t-test taking into account the combined standard uncertainty (quadratic sum of standard deviation and standard uncertainty) of each set of data ; this test was performed to check whether any two sets of data could be considered as samples from the same population,
- the one-way analysis of variance to calculate the within- and between-laboratories standard deviations,
- the Snedecor F-test to check whether the between-laboratories combined standard uncertainties were significantly different from zero,
- the Bartlett test taking into account the combined standard uncertainty of each set of data to check whether the differences between the variances were significant.

Secondly, in order to verify if the average and the confidence interval could be calculated assuming that the considered population of data is normally distributed, the

Lilliefors version of the Kolmogorov-Smirnov test was performed at the 0.05 and 0.01 significance levels.

The individual measurement results, the average value, standard deviation and standard uncertainty of each data set are given for each temperature level in Annex 7 (Table A7d to Table A7j). The diagrams of averages and their expanded combined uncertainties at the 95 % confidence interval are also given in these tables for each temperature level (Table A7d to Table A7j).

The results of the analysis of variance and the conclusions of the tests are given in Annex 7 (Table A7k). The results of the statistical analyses show that the pooling of all data sets is allowed for each temperature level, and the option for "pooling" has been selected.
The average, the standard deviation and the half-width of the 95 % tolerance interval of all individual values and the half-width of the 95 % confidence interval of the average, including the conclusion of the normality tests, are given in Table A7k.

8.3. Certified values and uncertainties

The certified values of the thermal conductivity over the range of mean test temperature [-10 °C / + 50 °C] were calculated from all the individual values retained for the process of certification (as the pooling was allowed for each temperature level).

The relationship between the thermal conductivity λ and the mean test temperature θ was determined by weighted least squares regression. The standard uncertainty of each individual measurement was included in the regression as a weight for each value. The best fit was selected through the analysis of statistical parameters (standard error, correlation coefficients, residuals and trends analysis). The curve λ versus θ over the range [-10 °C / + 50 °C] is a polynomial with second order temperature terms, given as follows:

$$\lambda\,[\mathbf{W/(m.K)}] = \mathbf{0.0293949} + \mathbf{0.0001060} \times \theta\,[^\circ\mathbf{C}] + \mathbf{2.047} \times \mathbf{10^{-7}} \times \theta^2\,[(^\circ\mathbf{C})^2]$$

The combined uncertainty of the λ certified values was determined from both contributions: uncertainty of the thermal conductivity measurements and uncertainty due to the fit λ versus θ. The uncertainty due to non-homogeneity of the reference material was negligible compared to the others sources of uncertainty (slight discrepancies were detected as regards to the thermal conductivity as described in section 4.3.2.).

The certified values, calculated using the above equation at 5 °C intervals within the range [-10 °C / + 50 °C], and their relative combined uncertainties at the 95 % confidence interval are given in Table 1.

Mean test temperature [°C]	λ certified values [W/(m.K)]	λ relative uncertainty [%] at the 95 % confidence interval
- 10	0.02836	1.0
- 5	0.02887	0.9
0	0.02940	0.9
5	0.02993	0.9
10	0.03048	0.9
15	0.03103	0.8
20	0.03160	0.8
25	0.03217	0.8
30	0.03276	0.8
35	0.03336	0.8
40	0.03396	0.8
45	0.03458	0.8
50	0.03521	0.8

Table 1: Certified values for IRMM-440 and their limit of uncertainty

The overall relative uncertainty of the λ certified values is within [0.8 % - 1 %], which corresponds to a constant limit of uncertainty at the 95 % confidence level of ± 0.00028 W/(m.K) over the measurement range [-10 °C / + 50 °C].

The plots of all the thermal conductivity measurements (retained for the process of certification) and the certified values (with their limits of uncertainty at the 95 % confidence interval) versus the mean test temperature over the range [-10 °C / + 50 °C], are given in Figure 1.

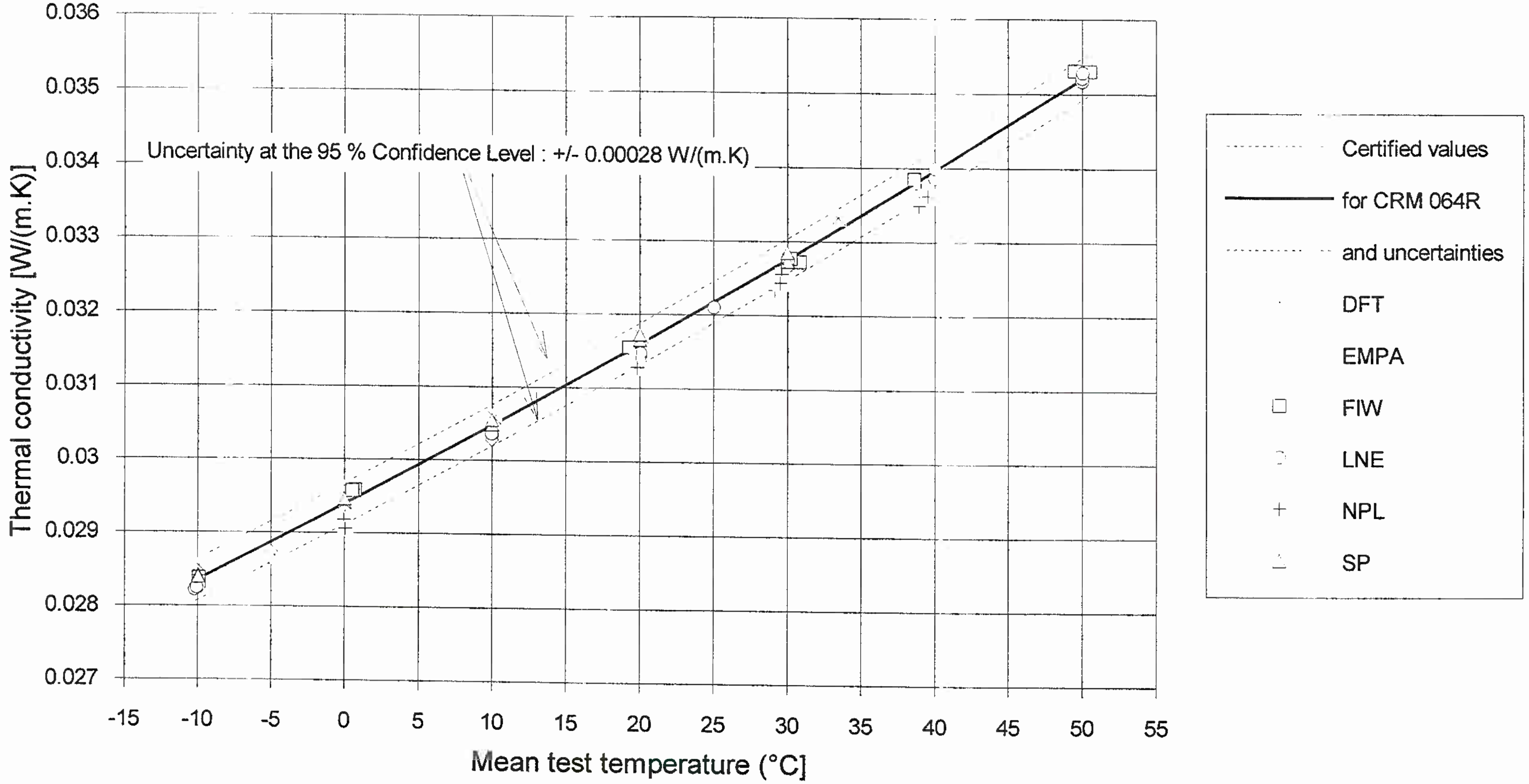

Figure 1: Certified values for IRMM-440 versus temperature over the range [-10 °C / + 50 °C]

9. INSTRUCTIONS FOR USE

The reference material is intended for the proven performance check of an equipment for absolute measurements (i.e. Guarded Hot Plate equipment, Guarded Hot Box equipment...), or for the calibration of instruments based on relative measuring methods (i.e. Heat Flow Meter equipment...). In the latter case (reference material used for calibration), it is particularly important to follow the instructions for use of the reference material to avoid serious measurement errors.

To protect the reference material from accidental damage, it is recommended to keep it in a strong protective box.

The thickness of the reference material sample shall not be modified by slicing to suit the requirements of a particular equipment.
The cutting of a reference material sample into small samples is possible. But, it is imperative to verify that the density of each small sample is in the certification range [64 kg/m^3 - 78 kg/m^3].

9.1. Preparation and conditioning before measurements

Before performing the thermal conductivity measurements, the reference material shall be dried in an oven at a temperature ranging from 70 °C to 100 °C until a constant mass is obtained.
Then, the reference material shall be conditioned in the standard atmosphere of the laboratory (usually temperature from 20 °C up to 23 °C - relative humidity from 40 % RH up to 65 % RH) to reach the equilibrium with the environment (i.e. constant mass) ; this is obtained if two successive measurements within a 24 hours interval do not differ by more than 0.5 %.

9.2. Precautions during the thermal conductivity measurements

The thickness and density of the reference material shall be measured as described in section 5.1. and 5.2.

In order to avoid the thickness change (and density change) of the reference material during the thermal conductivity measurements (due to the compressibility of the material), the sample thickness shall be fixed with adequate spacers or plate separation.
When using spacers, it is recommended to measure the spacers thickness at each mean test temperature, particularly when operating over the range [-10 °C / + 50 °C] in order to avoid thickness measurement errors due to thermal expansion of the spacers. Whenever possible, accurate thickness measurements should be carried out in situ.

When operating below ambient temperature, in order to avoid moist air transfer into the apparatus, causing condensation and/or freezing of water vapour within the material, it is recommended to control the dew point of the atmosphere surrounding the reference material at least 5 K below the cold plate temperature or to place the reference material in a vapour-tight envelope during the thermal conductivity measurements.

After completion of a series of tests, the reference material shall be removed from the apparatus and after reaching equilibrium with the standard laboratory atmosphere, the reference material shall be weighted again. The difference between mass determinations before and after the thermal conductivity measurements should be within 0.5 % to ensure the absence of disturbing moisture transfer into the sample during the thermal conductivity measurements.

10. REFERENCES

[1] ZIEBLAND (H.) - *Certification report on a reference material for the thermal conductivity of insulating materials between 170 K and 370 K. Resin-bonded glass fibre board (BCR No 64) (*1982)

[2] Draft CEN/TC 89/WG8 N112 - *Building products and components. Specific criteria for the assessment of laboratories performing heat transfer property measurements. Part 2: Measurements by the Guarded Hot Plate method* (1998)

[3] ISO 8301 - *Thermal insulation. Determination of steady-state thermal resistance and related properties - Heat Flow Meter apparatus* (1991).

[4] ISO 8302 - *Thermal insulation. Determination of steady-state thermal resistance and related properties - Guarded Hot Plate apparatus* (1991).

[5] EN 45001 - *General criteria for the operation of testing laboratories* (1989).

[6] prEN 12939 - *Thermal performance of building materials. Determination of thermal resistance by means of Guarded Hot Plate and Heat Flow Meter methods. Thick products of high and medium thermal resistance* (1998).

ANNEX 1

PRELIMINARY ASSESSMENT OF THE BATCH

- **Board thickness measurements**

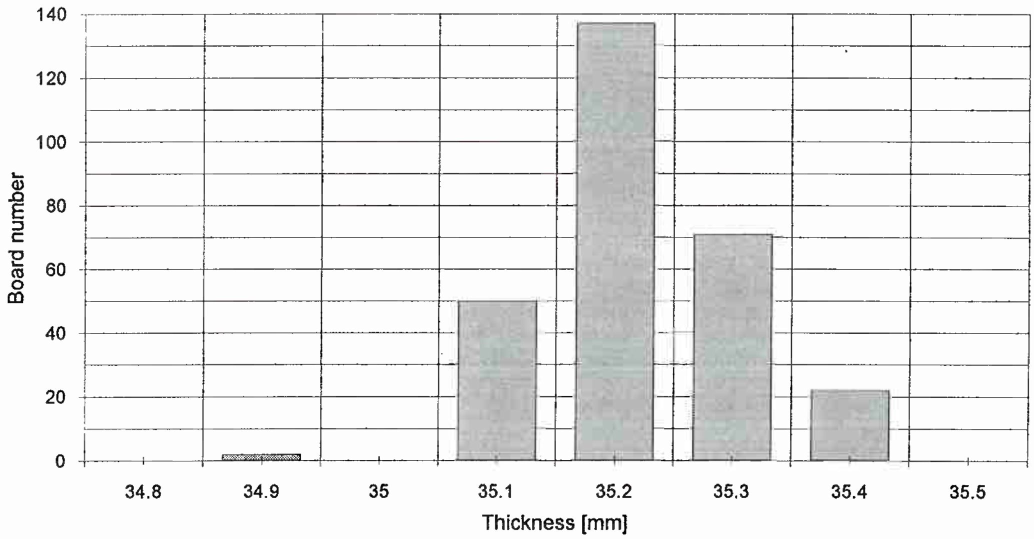

Figure A1a: Histogram of board thickness distribution

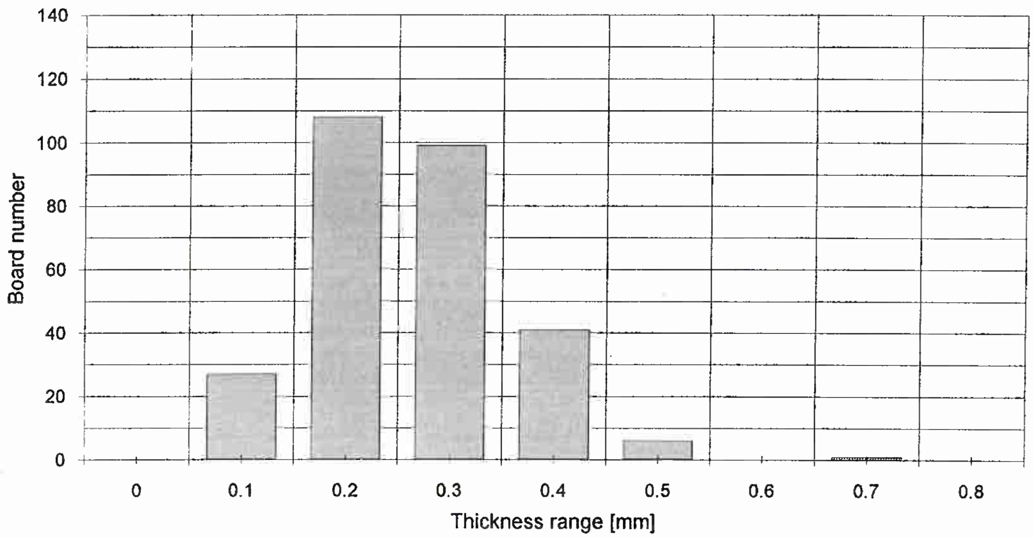

Figure A1b: Histogram of thickness range distribution of the boards

- **Board apparent density measurements**

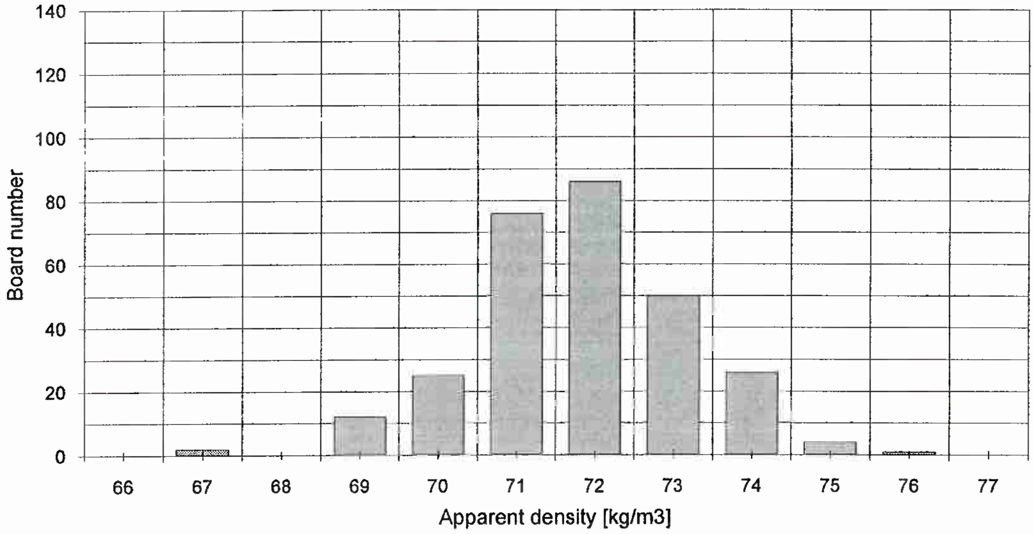

Figure A1c: Histogram of board density distribution

- **Thermal conductivity measurements of 0.600 m x 0.600 m specimens**

Identification number and physical characteristics									Thermal conductivity measurements using two specimen GHP and single specimen HFM apparatuses				
1.200 m x 1.200 m Boards			0.600 m x 0.600 m Specimens (overall size GHP and HFM)			0.300 m x 0.300 m Specimens (metering area GHP and HFM)			Test characteristics		GHP		HFM
ident. number	thickness [mm]	density [kg/m³]	ident. number	thickness [mm]	density [kg/m³]	ident. number	thickness [mm]	density [kg/m³]	spacer thickness [mm]	density [kg/m³]	λ at 10°C [mW/(m.K)]	λ at 20°C [mW/(m.K)]	λ at 20°C [mW/(m.K)]
I11	35.10	72.15	I11a	35.21	70.42	I11a1	34.60	69.92	35.12	72.05	30.38	31.60	31.80
			I11b	35.17	73.68	I11b1	34.59	78.18					31.34
			I11c	35.18	71.51	I11c1	34.70	74.92	35.12	71.94	30.51	31.61	-
			I11d	35.19	72.38	I11d1	34.84	75.06					31.29
D24	35.10	72.85	D24a	35.01	71.53	D24a1	34.87	70.63	34.90	72.32	-	31.46	31.57
			D24c	34.93	72.58	D24c1	34.71	75.31					31.58
			D24b	35.02	73.85	D24b1	34.71	73.99	34.18 (*)	75.63 (*)	30.22 (*)	31.36 (*)	-
			D24d	34.88	74.05	D24d1	34.60	77.62					-
B61	35.11	75.32	B61a	34.86	77.76	B61a1	34.72	78.44	34.90	77.22	30.46	31.49	31.52
			B61d	34.88	76.81	B61d1	34.73	81.90					31.87
I13	35.30	66.47	I13a	35.07	65.67	I13a1	34.85	66.44	35.00	65.67	30.23	31.33	31.65
			I13c	35.00	65.33	I13c1	34.95	62.65					31.49
D66	35.07	74.64	D66b	34.97	73.95	-	-	-	34.90	74.02	30.41	31.40	-
			D66c	34.94	73.87	-	-	-					-
F44	35.08	71.31	F44c	34.89	71.65	-	-	-	34.90	70.33	30.36	31.43	-
			F44d	34.89	69.05	-	-	-					-

(*) measurement performed on specimens without spacers (compressed specimens)

Table A1: Thermal conductivity measurements

HOMOGENEITY ASSESSMENT OF THE BATCH

- **Specimen thickness distribution (DFT measurements)**

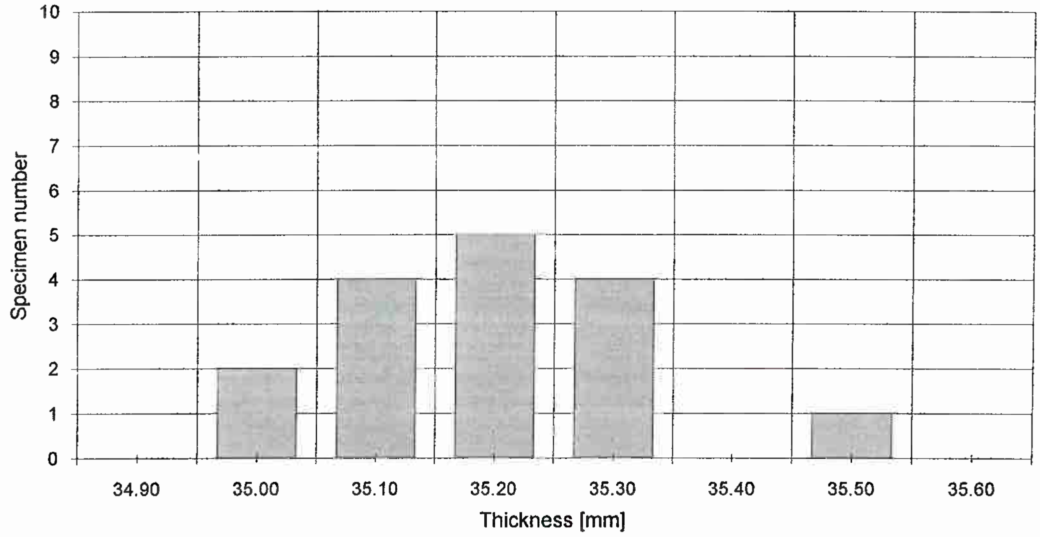

Figure A2a: Histogram of specimen thickness distribution (at 164 Pa)

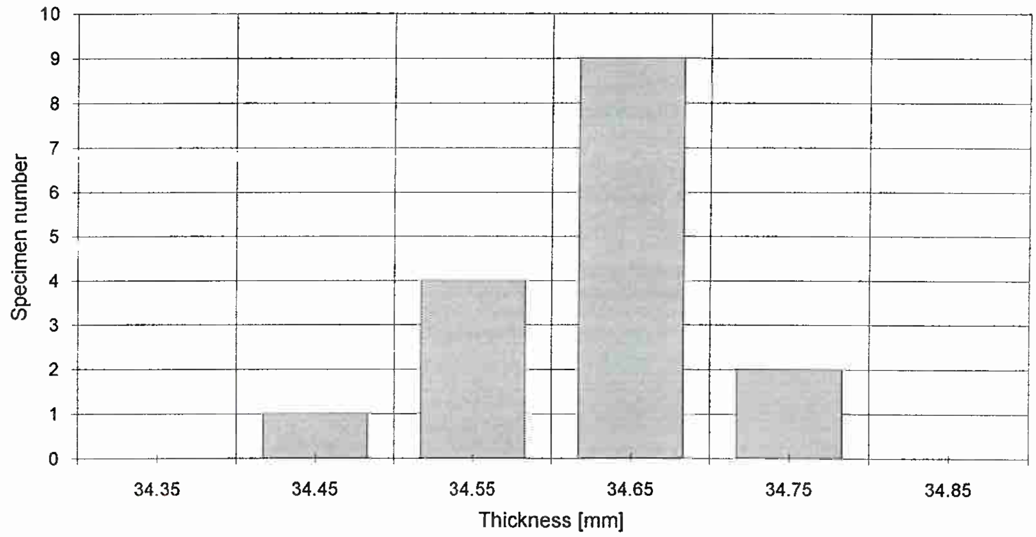

Figure A2b: Histogram of specimen thickness distribution (at 1170 Pa)

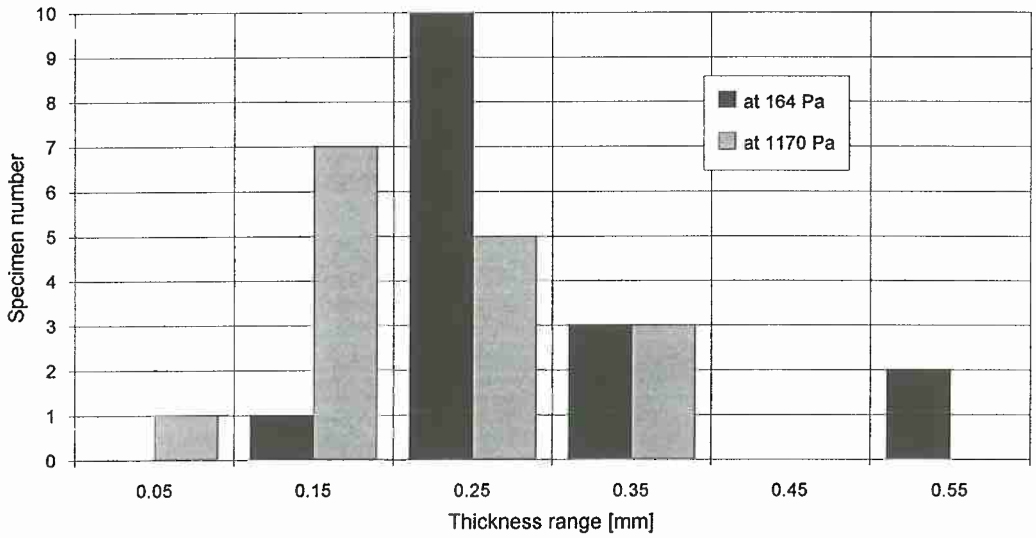

Figure A2c: Histogram of specimen thickness range distribution (at 164 Pa - 1170 Pa)

- **Specimen thickness distribution (LNE measurements)**

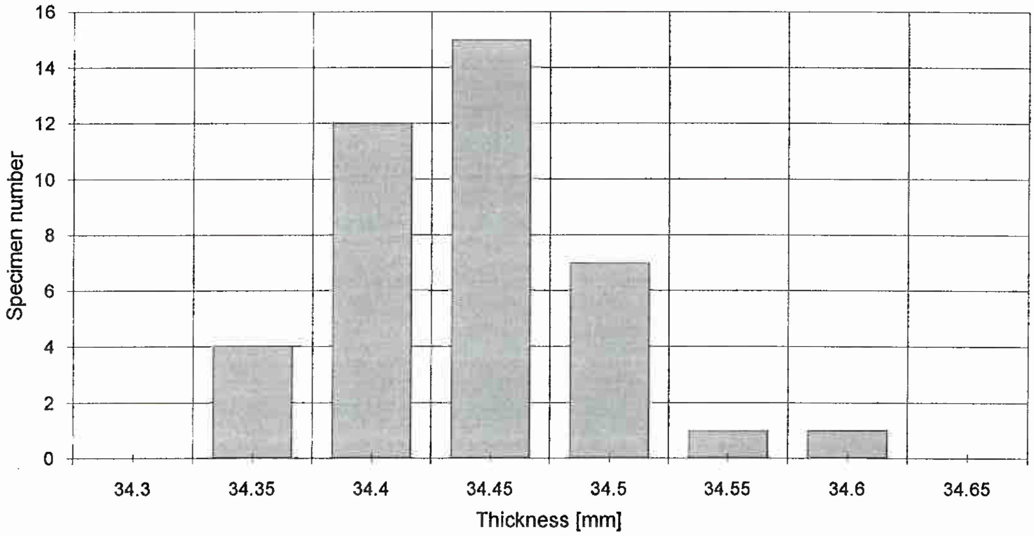

Figure A2d: Histogram of specimen thickness distribution (at 1500 Pa)

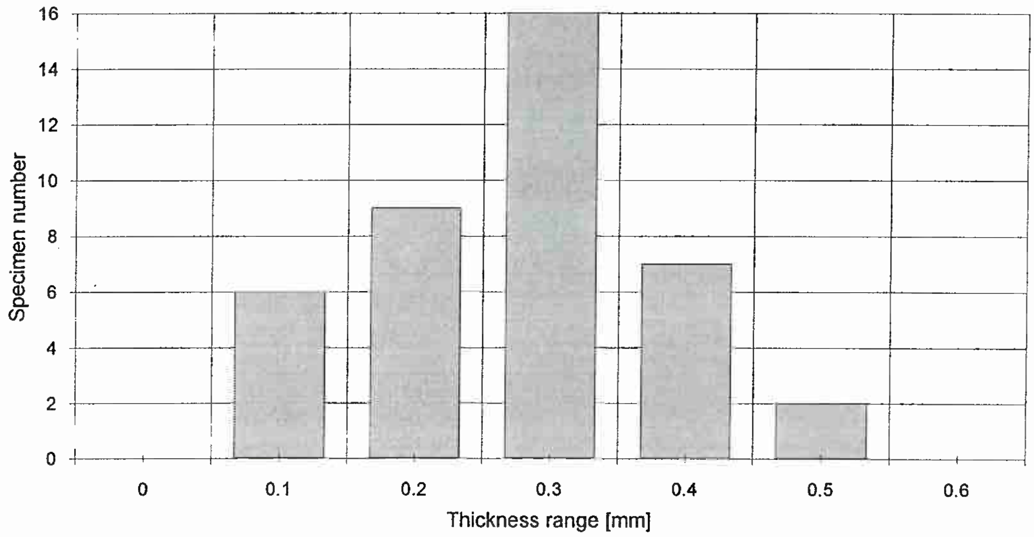

Figure A2e: Histogram of specimen thickness range distribution (at 1500 Pa)

- **Specimen density distribution (DFT measurements)**

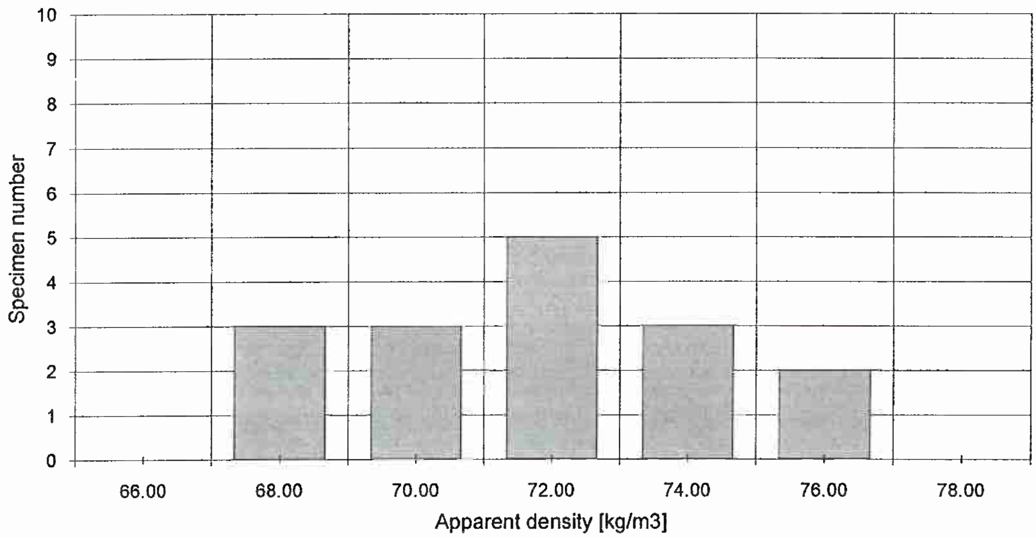

Figure A2f: Histogram of specimen apparent density distribution (at 1170 Pa)

- **Specimen density distribution (LNE measurements)**

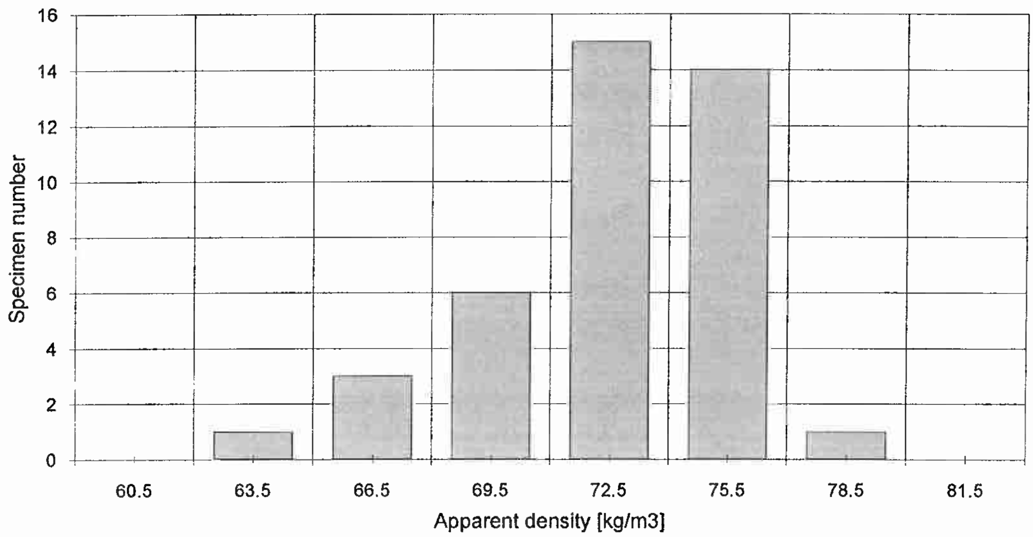

Figure A2g: Histogram of specimen apparent density distribution (at 1500 Pa)

- **Thermal conductivity distribution (DFT measurements)**

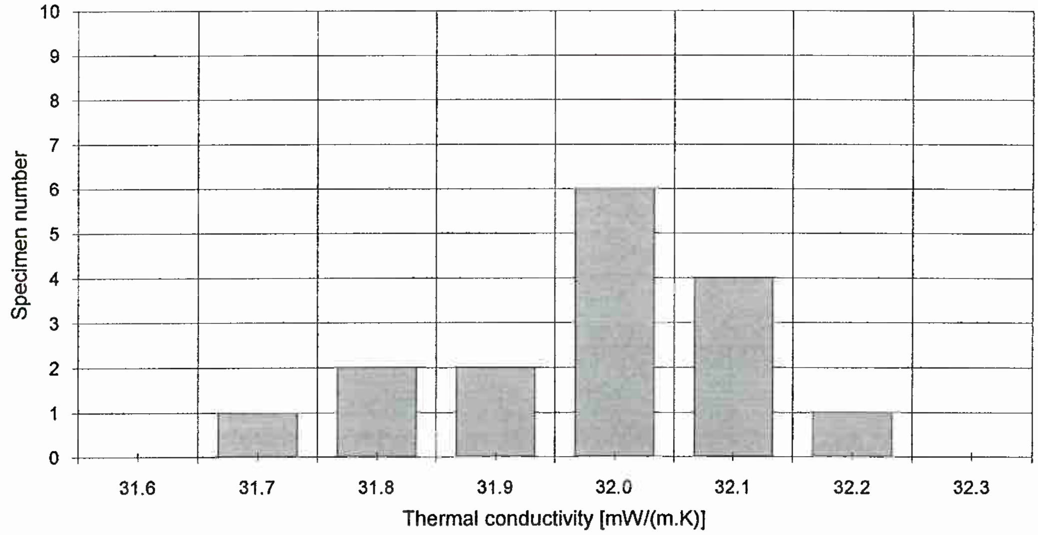

Figure A2h: Histogram of thermal conductivity distribution

- **Thermal conductivity distribution (LNE measurements)**

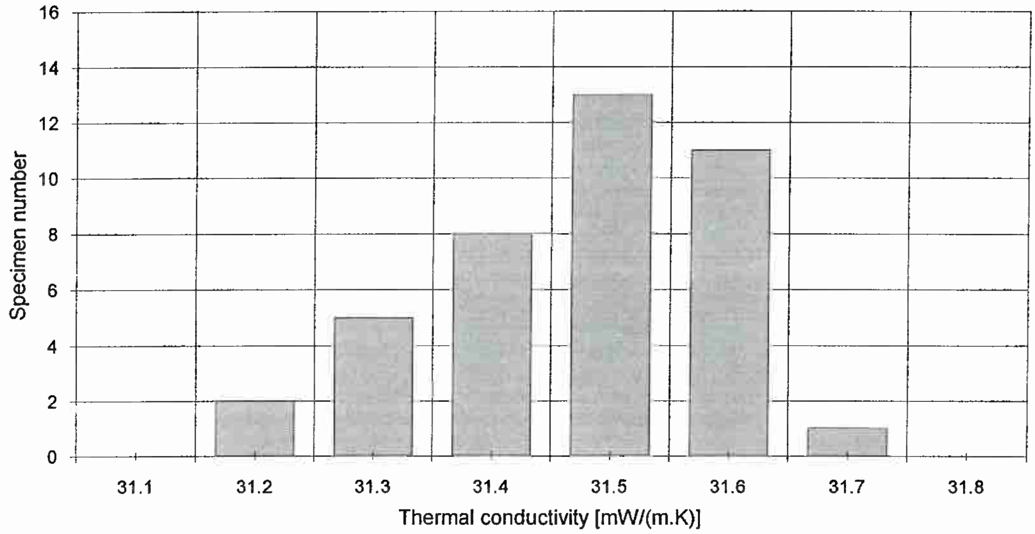

Figure A2i: Histogram of thermal conductivity distribution

- **Thermal conductivity versus density (DFT measurements)**

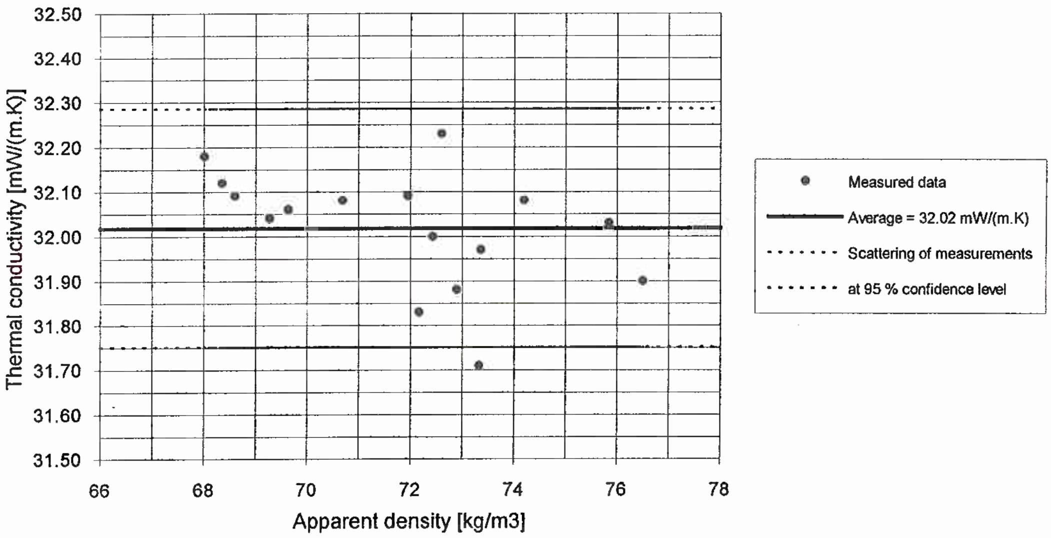

Figure A2j: Thermal conductivity versus specimen density

- **Thermal conductivity versus density (LNE measurements)**

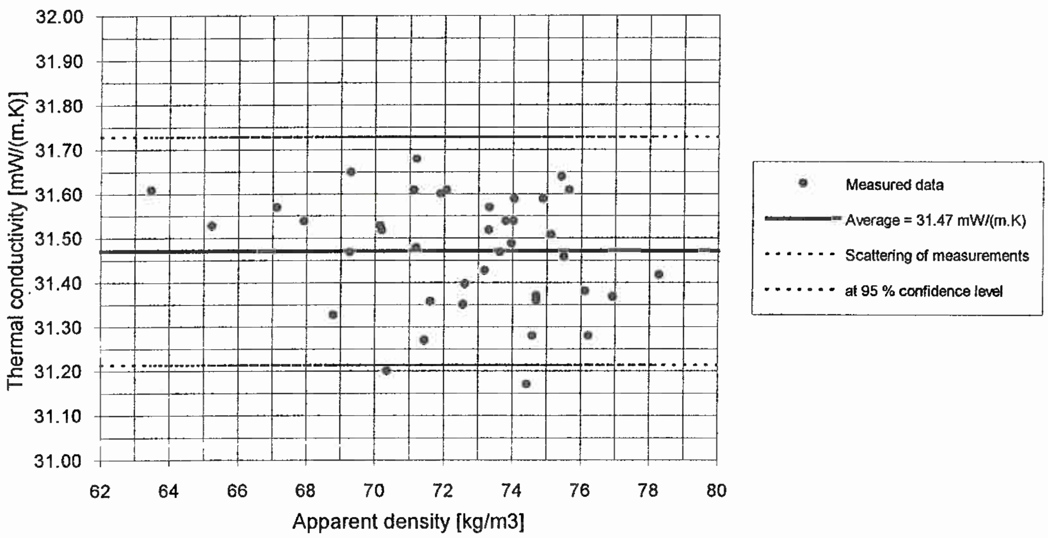

Figure A2k: Thermal conductivity versus specimen density

- **Outcome of the homogeneity study**

Thickness statistical analysis	DFT	LNE
Number of specimens	16	40
Average value [mm]	34.65	34.44
Standard deviation [mm]	0.075	0.05
Relative standard deviation [%]	0.20	0.15
Density statistical analysis	DFT	LNE
Number of specimens	16	40
Average value [kg/m^3]	71.9	72.5
Standard deviation [kg/m^3]	2.6	3.2
Relative standard deviation [%]	3.6	4.4
Thermal conductivity statistical analysis	DFT	LNE
Number of specimens	16	40
Average value (at 20°C) [mW/(m.K)]	32.02	31.47
Standard deviation [mW/(m.K)]	0.13	0.13
Relative standard deviation [%]	0.42	0.41

Table A2: Results of the homogeneity study carried out by DFT and LNE

STABILITY ASSESSMENT OF THE BATCH

- **Thermal conductivity as a function of time**

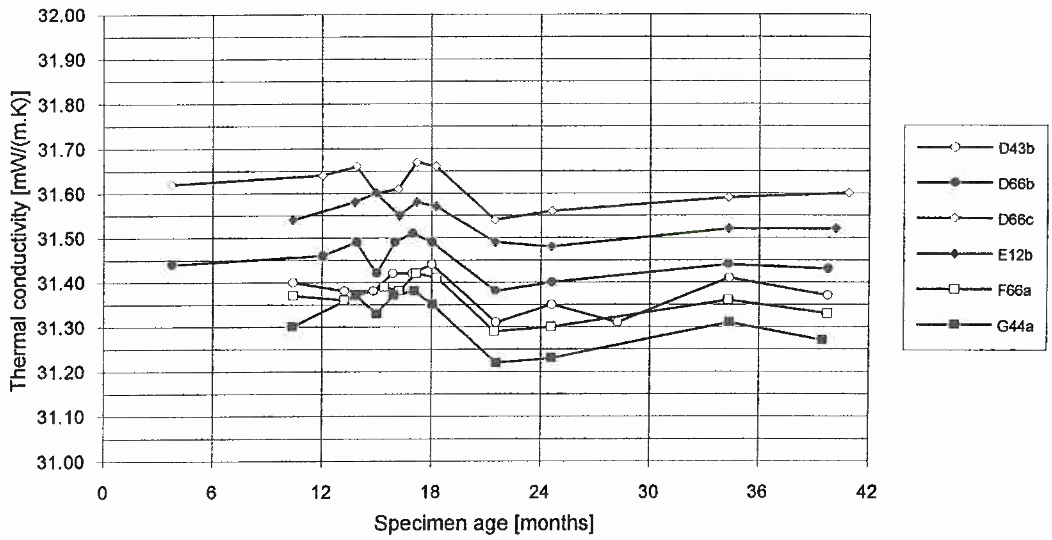

Figure A3: Thermal conductivity versus specimen age

- **Outcome of the stability study**

Identification code of the specimens	Thermal conductivity statistical analysis [mW/(m.K)]		
	Average value	Standard deviation	Range
D43b	31.38	0.04	0.13
D66b	31.45	0.04	0.13
D66c	31.61	0.04	0.13
E12b	31.54	0.04	0.12
F66a	31.36	0.04	0.13
G44a	31.31	0.06	0.16

Table A3: Results of the stability study carried out at 20 °C by LNE

ANNEX 4

THERMAL CONDUCTIVITY
MEASUREMENT PRINCIPLE AND EXPRESSION OF RESULTS

- **Principle of measurement**

The thermal conductivity measurements could be carried out using a symmetrical two specimen Guarded Hot plate (GHP) apparatus.
The symmetrical two specimen GHP apparatus is designed to establish within two flat slabs an unidirectional uniform density of heat flow rate, which would exist in an infinite slab bounded by two flat parallel isothermal surfaces.

A schematic of GHP apparatus principle is given in Figure A4.

Under steady state conditions (uniform and constant density of heat flow rate through the specimens), the following quantities are determined:

- the density of heat flow rate through the specimens from the measurement of the heat flow rate and the metering area.
- the temperature difference across the specimens from the individual measurements of the cold and hot plates temperature.
- the specimen thickness.

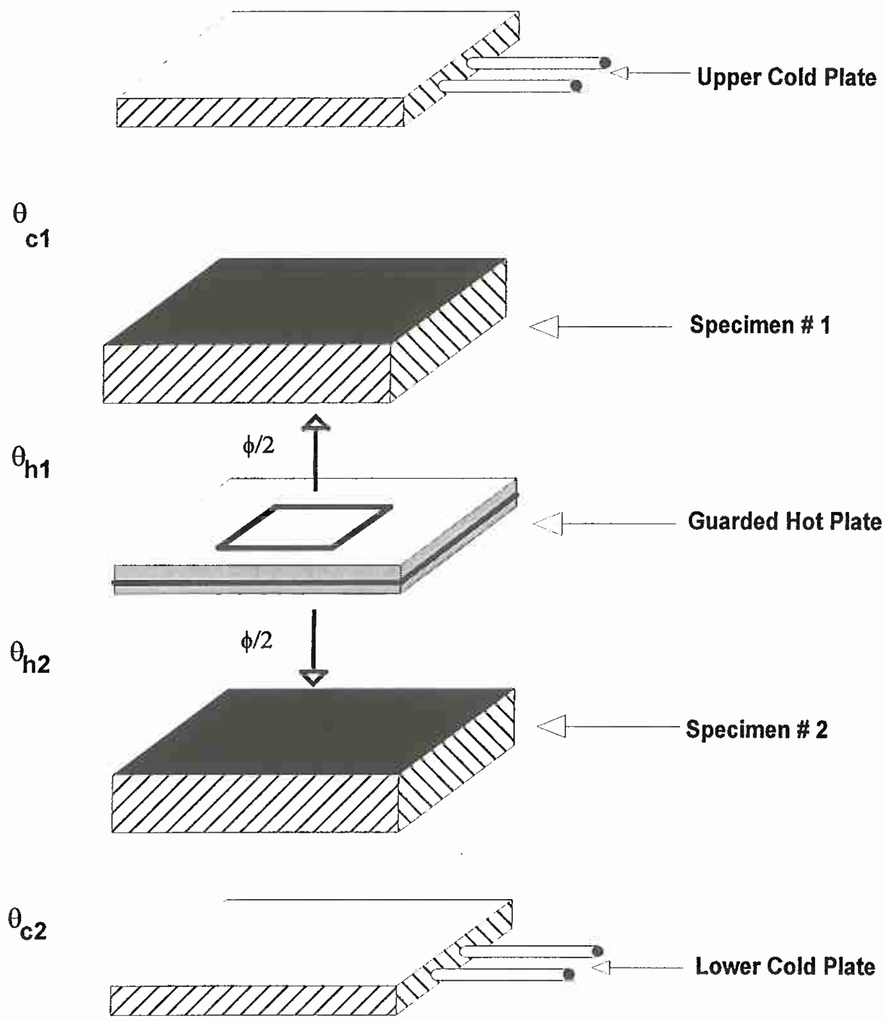

Figure A4: Guarded Hot Plate apparatus principle

- **Expression of results**

The heat transfer properties (thermal resistance and thermal conductivity) are determined after the validation of the attainment of steady state conditions.
The heat transfer properties are calculated from the average of steady state data which are regularly measured over a measurement interval, using the following equation:

$$R_m = \frac{d_m}{\lambda_m} = \frac{1}{n}\sum_{i=1}^{n}\frac{2\,A\,\Delta\theta_m}{\Phi} \quad and \quad \Delta\theta_m = \frac{(\theta_{h1} - \theta_{c1}) + (\theta_{h2} - \theta_{c2})}{4}$$

where:
- R_m : thermal resistance of the specimens [m².K/W]
- λ_m : thermal conductivity of the specimens [W/(m.K)]
- d_m : average thickness of the specimens [m]
- n : number of measurements
- A : metering area [m²]
- Φ : heat flow rate through the specimens [W]
- $\Delta\theta_m$: temperature difference across the specimens [K]
- θ_{h1} : temperature of the upper hot plate [°C]
- θ_{h2} : temperature of the lower hot plate [°C]
- θ_{c1} : temperature of the upper cold plate [°C]
- θ_{c2} : temperature of the lower cold plate [°C]

The heat transfer properties (thermal resistance and thermal conductivity) are defined at the mean test temperature θ_m, as given in the following equation:

$$\theta_m = \frac{(\theta_{h1} + \theta_{c1} + \theta_{h2} + \theta_{c2})}{4}$$

ANNEX 5

CERTIFICATION MEASUREMENTS

• Measurement protocol

Laboratory	DFT	EMPA	FIW	LNE	NPL	SP
Type of equipment	two specimen GHP with edge insulation and secondary T-shaped guard	two specimen GHP with edge insulation	two specimen GHP with independent controlled secondary guard and edge insulation	two specimen GHP with temperature controlled peripheral guard and edge insulation	single specimen GHP with linear temperature gradient edge guards	single specimen GHP with edge insulation
Dimensions of equipment						
• Overall dimensions	• 0.300 m x 0.300 m	• 0.750 m x 0.750 m	• 0.800 m x 0.800 m	• 0.610 m x 0.610 m	• 0.610 m x 0.610 m	• 0.400 m x 0.400 m
• Metering area	• 0.148 m x 0.148 m	• 0.300 m x 0.300 m	• 0.300 m x 0.300 m	• 0.300 m x 0.300 m	• 0.305 m x 0.305 m	• 0.202 m x 0.202 m
• Guard width	• 0.076 m	• 0.225 m	• 0.250 m	• 0.155 m	• 0.150 m	• 0.099 m
• Gap width	• 0.002 m	• 0.002 m	• 0.0015 m	• 0.001 m	• 0.002 m	• 0.003 m
Operating ranges						
• Thickness	• 0.020 m to 0.040 m	• 0.020 m to 0.190 m	• 0.020 m to 0.200 m	• 0.020 m to 0.160 m	• 0.020 m to 0.250 m	• 0.030 m to 0.070 m
• Thermal resistance	• 0.01 m^2.K/W to 4 m^2.K/W	• 0.1 m^2.K/W to 10 m^2.K/W	• 0.01 m^2.K/W to 8 m^2.K/W	• 0.1 m^2.K/W to 10 m^2.K/W	• 0.2 m^2.K/W to 12.5 m^2.K/W	• 0.1 m^2.K/W to 2.5 m^2.K/W
• Temperature difference	• 2 K to 45 K	• 10 K to 40 K	• 5 K to 30 K	• 5 K to 40 K	• 8 K to 30 K	• 10 K to 30 K
• Cooling unit temperature	• - 170 °C to + 35 °C	• - 15 °C to + 40 °C	• - 40 °C to + 60 °C	• - 15 °C to + 55 °C	• - 15 °C to + 40 °C	• - 20 °C to + 40 °C
• Heating unit temperature	• - 170 °C to + 35 °C	• - 5 °C to + 50 °C	• - 40 °C to + 60 °C	• - 5 °C to + 65 °C	• + 5 °C to + 50 °C	• 0 °C to + 60 °C
• Surrounding environment	• T-shaped guard at heating unit temperature / dew point below the cold plate	• 23 ± 2 °C / 50 ± 5 % RH	• 23 ± 8 °C / (5 to 90) % RH	• peripheral guard at mean test temperature	• linear temperature gradient edge guards	• ambient temperature at mean test temperature / dew point 5°C below the cold plate
Performance check						
• Deviation from flatness	• 0.05 mm	• Cooling unit: 0.05 mm Heating unit: 0.15 mm	• < 0.05 mm	• < 0.05 mm	• < 0.15 mm	• < 0.10 mm
• Uncertainty of the temperature difference measurement	• ± 0.65 % at room temp. ± 1.15 % at low temp.	• ± 1.4 % (ΔT= 10 K) ± 1 % (ΔT= 15 K)	• ± 0.11 %	• ± 0.7 %	• ± 0.156 % (ΔT= 18 K)	• ± 0.5 %
• Edge heat losses and imbalance error	• ± 0.02 % at room temperature	• < ± 0.5 %	• ± 0.24 %	• ± 0.3 %	• < ± 0.4 %	• ± 0.3 %
• Emissivity	• 0.89	• 0.92	• 0.93	• 0.86	• 0.89	• 0.904
• Linearity test (max. deviation)	• ± 0.2 % from mean value	• ± 0.2 % from mean value	• ± 0.5 %	• ± 0.2 % from mean value	• ± 0.3 % from mean value	• < 0.3 % from mean value
• Reproducibility	• < 0.1 % from mean value	• ± 0.2 %	• ± 0.3 %	• ± 0.2 % from mean value	• ± 0.3 % from mean value	• < 0.2 % from mean value
• Proven performance check	• BCR CRM 039-064-124	• -	• BCR CRM 039-064-124	• BCR CRM 064	• -	• BCR CRM 064
Maximum uncertainty on λ						
• arithmetic sum	• ± 1.1 %	• ± 2.3 %	• ± 0.95 %	• ± 1.9 %	• ± 2.2 %	• ± 1.9 %
• quadratic sum	• ± 0.7 %	• ± 1 %	• -	• ± 1.05 %	• ± 1.4 %	• ± 0.7 %
• others	• -	• ± 2 % (certificate)	• -	• -	• ± 1.5 % (certificate)	• -

Table A5a: Summary of the technical information on GHP equipment of the participating laboratories

- **Presentation of the thermal conductivity results**

Laboratory	DFT	EMPA	FIW	LNE	NPL	SP
Thickness measurement • Applied pressure • Measurement method • Correction for spacers thermal expansion	• 1000 Pa • measuring plate • no correction	• 1000 Pa • outside GHP equipment • no correction	• 1000 Pa • outside GHP equipment • no correction	• 1500 Pa • measuring plate • corrections	• 1500 Pa • measuring plate • corrections	• 1000 Pa • outside GHP equipment • no correction
Conditioning before test • Drying • Surrounding environment	• no drying • 23 ± 2 °C / 50 ± 5 % RH	• no drying • 23 ± 2 °C / 50 ± 5 % RH	• at 105 °C • 23 ± 5 °C / 50 ± 10 % RH	• at 70 °C • 23 ± 2 °C / 50 ± 5 % RH	• no drying • 21 ± 2 °C / 40 % RH	• no drying • 20 ± 2 °C / 50 ± 5 % RH
Conditions during test • Means for setting thickness • Temperature difference • Humidity control	• PVC foam spacers • 11 K • dew point below the cold plate	• wood spacers • 10 K • no control	• brass spacers • 12 K to 16 K • no control	• polyacetal spacers • 15 K • no control	• plate separation • 17 K • dry air is trickled inside to eliminate condensation when operating below the dew point	• plastics (PMMA) spacers • 20 K • dew point 5 °C below the cold face
Method for the determination of λ results • Duration of test • Steady state condition • Criterion for steady state	• - • minimum 10 hours • validation of criterion on 10 h interval (≤ ± 0.1 %)	• - • minimum 8 hours • 5 measurements	• 24 hours • minimum 5 hours • validation of criteria on 100 mn interval (1 measurement every 20 minutes)	• - • minimum 16 hours • validation of 2 criteria on 16 h interval (based on standard deviation and slope)	• 16 - 24 hours • - • validation of criterion on 10 h interval (≤ ± 0.5 %)	• minimum 48 hours • minimum 24 hours • validation of 2 criteria (based on standard deviation and slope)
Relative expanded uncertainty of λ results (at the 95 % confidence interval)	± 1.2 % at temp. ≤ 0 °C ± 0.7 % at temp. > 0 °C	± 2.0 %	± 1.0 %	± 1.05 %	± 1.5 %	± 0.7 %

Table A5b: Summary of the experimental test conditions of the measurements performed by the participating laboratories

Laboratory	DFT			EMPA			FIW		
LOW DENSITY SAMPLE • Identification • Density	• I13a - F66b • 66.4 - 67.1 kg/m³			• A54a - C55b • 71.2 - 71.0 kg/m³			• B21b - H22b • 68.7 - 69.5 kg/m³		
• Measurements results	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]
	- 169.54	34.71	11.41	- 5.00	34.70	28.51	- 9.96	34.50	28.39
	- 150.98	34.71	13.47	0.00	34.70	28.96	0.52	34.50	29.59
	- 132.07	34.71	15.57	10.00	34.70	30.21	19.29	34.50	31.60
	- 103.25	34.71	18.71	20.00	34.70	31.41	30.22	34.50	32.85
	- 63.22	34.71	22.95	30.00	34.70	32.75	38.60	34.50	33.96
	- 31.77	34.71	26.24				49.48	34.50	35.45
	- 20.76	34.71	27.39						
	- 9.96	34.71	28.58						
	0.08	34.71	29.63						
	0.22	34.71	29.62						
	10.06	34.71	30.63						
	10.13	34.71	30.61						
	20.05	34.71	31.70						
	20.09	34.71	31.71						
	33.41	34.71	33.31						
	33.45	34.71	33.29						
HIGH DENSITY SAMPLE • Identification • Density	• B31c - B61d • 81.4 - 81.5 kg/m³			• E32a - E11b • 73.5 - 74.8 kg/m³			• H45a - B31a • 75.0 - 75.2 kg/m³		
• Measurements results	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]
	- 150.91	34.69	13.67	- 5.00	34.75	28.61	- 9.92	34.50	28.45
	- 132.05	34.69	15.77	0.00	34.75	29.16	0.70	34.50	29.60
	- 103.16	34.69	18.91	10.00	34.75	30.25	19.22	34.50	31.60
	- 62.99	34.69	23.08	20.00	34.75	31.44	30.85	34.50	32.80
	- 31.53	34.69	26.30	30.00	34.75	32.81	50.56	34.50	35.44
	- 9.94	34.69	28.52						
	- 9.78	34.69	28.53						
	0.11	34.69	29.53						
	10.02	34.69	30.45						
	19.98	34.69	31.48						
	33.37	34.69	32.96						

Table A5c: Synthesis of the thermal conductivity measurements reported by the participating laboratories (1/2)

Laboratory	LNE			NPL			SP		
LOW DENSITY SAMPLE • Identification • Density	• E12a - E62b • 69.3 - 70.2 kg/m³			• B15b • 71.5 kg/m³			• C55d • 66.0 kg/m³		
• Measurements results	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]
	- 10.19	34.33	28.11	- 0.05	34.27	29.21	- 10.00	34.70	28.49
	- 10.16	34.33	28.03	0.03	34.15	29.09	0.00	34.70	29.58
	- 10.09	34.33	28.13	19.72	34.28	31.43	10.00	34.70	30.69
	0.00	34.36	29.15	19.83	34.19	31.36	20.00	34.70	31.89
	9.98	34.40	30.30	29.50	34.26	32.51	30.00	34.70	33.03
	20.00	34.43	31.46	29.11	34.21	32.41			
	20.03	34.43	31.48	39.35	34.29	33.62			
	25.05	34.44	32.05	39.53	34.25	33.63			
	30.05	34.45	32.69						
	40.00	34.46	33.89						
	49.98	34.48	35.04						
	50.04	34.48	35.11						
HIGH DENSITY SAMPLE • Identification • Density	• E41a - D61a • 74.0 - 73.8 kg/m³			• E31b • 75.3 kg/m³			• F66e • 75.0 kg/m³		
• Measurements results	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]
	- 9.99	34.34	28.14	0.03	34.42	29.25	- 10.00	34.60	28.50
	- 9.91	34.34	28.16	0.04	33.88	29.25	0.00	34.60	29.58
	- 0.02	34.37	29.19	19.79	34.47	31.51	10.00	34.60	30.63
	19.99	34.44	31.44	19.76	34.39	31.40	20.00	34.60	31.81
	20.00	34.44	31.43	29.57	34.50	32.63	30.00	34.60	32.91
	29.98	34.46	32.62	29.14	34.36	32.41			
	40.00	34.47	33.76	39.07	34.52	33.72			
	49.99	34.49	35.01	38.95	34.43	33.49			
	50.00	34.49	35.00						

Table A5c: Synthesis of the thermal conductivity measurements reported by the participating laboratories (2/2)

- **Thermal conductivity measurements versus mean test temperature**

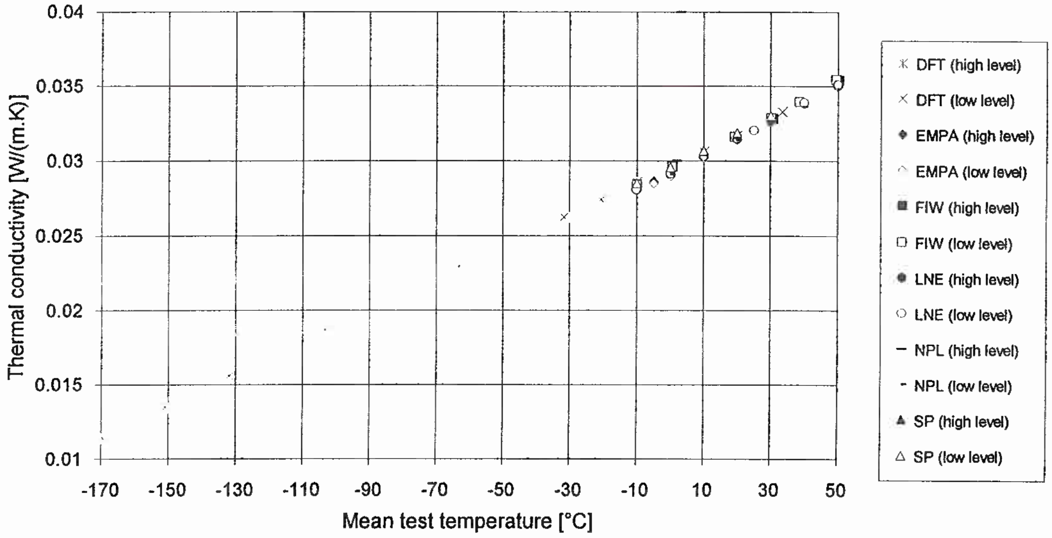

Figure A5a: Thermal conductivity versus temperature over the range [-170 °C / + 50 °C]

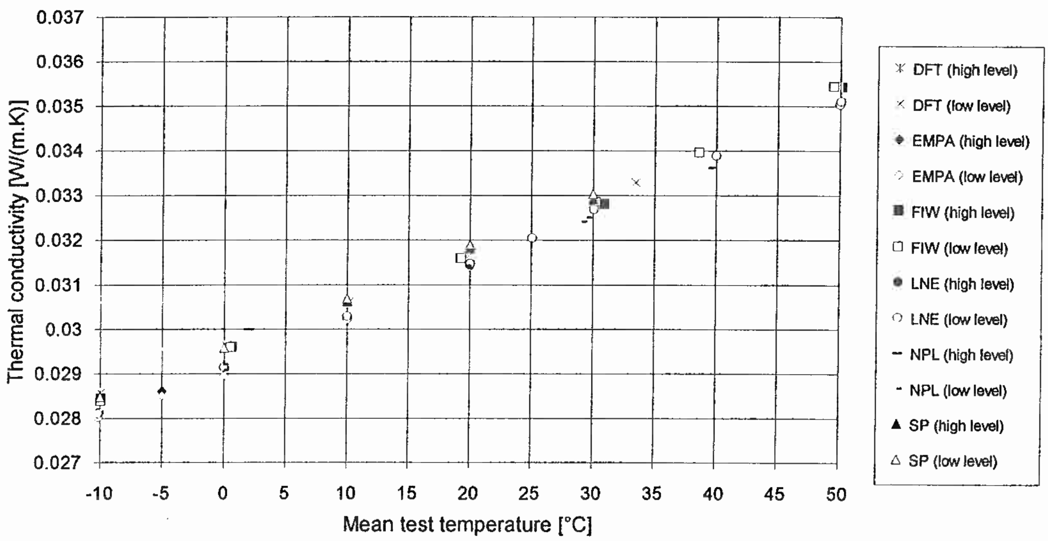

Figure A5b: Thermal conductivity versus temperature over the range [-10 °C / + 50 °C]

- **Statistical analysis results (at the stage of technical scrutiny of the data)**

Thermal conductivity at the temperature level	θ = - 10 °C	θ = 0 °C	θ = + 10 °C	θ = + 20 °C	θ = + 30 °C	θ = + 40 °C	θ = + 50 °C
Number of data sets	4	6	4	6	6	3	2
Number of individual data	12	15	8	17	15	7	6
All data sets compatible two by two ? (Scheffe multiple t-test) /1/	/2/	/3/	/4/	/6/	/7/	/8/	/9/
Outlying data sets? (Nalimov t-test)	no	no	no	no	no	no	no
Outlying variances ? (Cochran test)	no	no	/5/	no	no	no	no
Average of data set averages [W/(m.K)]	0.02839	0.02935	0.03042	0.03158	0.03276	0.03388	0.03524
Within-data sets standard deviation [W/(m.K)]	0.00004	0.00007	0.00006	0.00007	0.00011	0.00009	0.00006
Between-data sets standard deviation [W/(m.K)]	0.00022	0.00022	0.00020	0.00015	0.00013	0.00017	0.00027
Between-data sets standard deviation significantly different from zero? (Snedecor F-test)	yes	yes	yes	yes	yes	no	no
Variance homogeneous ? (Bartlett test)	yes	yes	yes	yes	yes	yes	yes

/1/ The tests are performed at the 0.05 and 0.01 levels of significance.
/2/ LNE data set is significantly different from DFT, FIW and SP data sets at the 0.05 and 0.01 levels of significance.
/3/ Two homogeneous groups are identified: (EMPA, LNE, NPL) and (DFT, FIW, SP) ; both groups of data sets are significantly different two by two at the 0.05 and 0.01 levels of significance.
/4/ EMPA data set is significantly different from DFT and SP data sets at the 0.05 level of significance, but not at the 0.01 level.
/5/ DFT data set is suspected to be an outlier of variance.
/6/ SP data set is significantly different from EMPA, LNE and NPL data sets at the 0.05 and 0.01 levels of significance and FIW data set is significantly different from LNE and NPL data sets at the 0.05 significance level but not at the 0.01 level.
/7/ There are no statistically significant differences between any two sets of data at the 0.05 level of significance.
/8/ FIW data set is significantly different from NPL data sets at the 0.05 significance level but not at the 0.01 level.
/9/ FIW data set is significantly different from LNE data sets at the 0.05 and 0.01 significance levels.

Table A6a: Summary of statistical data (at the stage of technical scrutiny)

- **Individual statistical analysis of the results**

Laboratory	Degree of least squares regression		Temperature range
	LOW DENSITY SAMPLE	HIGH DENSITY SAMPLE	
DFT	3^{rd} order	3^{rd} order without 2^{d} term	[- 170 °C / + 35 °C]
	3^{rd} order without 2^{d} term	3^{rd} order without 2^{d} term	[- 10 °C / + 35 °C]
EMPA	1^{st} order	3^{rd} order without 2^{d} term	[- 5 °C / + 30 °C]
FIW	3^{rd} order without 2^{d} term	3^{rd} order without 2^{d} term	[- 10 °C / + 50 °C]
LNE	2^{d} order	2^{d} order	[- 10 °C / + 50 °C]
NPL	1^{st} order	1^{st} order	[0 °C / + 40 °C]
SP	1^{st} order	1^{st} order	[- 10 °C / + 30 °C]

Table A6b: Best fits of λ versus θ for the participating laboratories

- **Technical scrutiny of data**

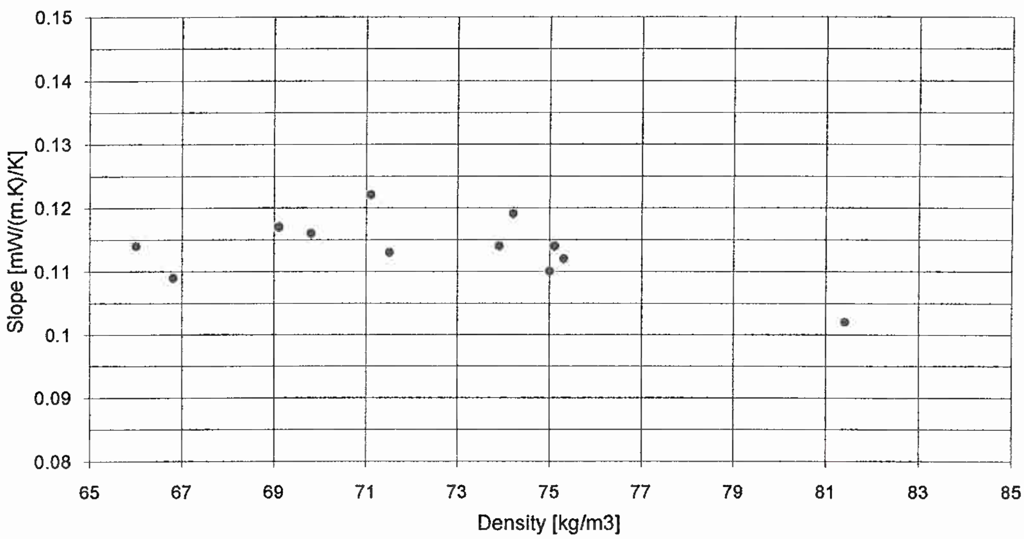

Figure A6a: Slope of thermal conductivity versus density

Laboratory	DFT			EMPA			FIW		
Results obtained from the linearity tests	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(\lambda_{meas}-\lambda)}{\lambda}\right)$ [%]	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(\lambda_{meas}-\lambda)}{\lambda}\right)$ [%]	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(\lambda_{meas}-\lambda)}{\lambda}\right)$ [%]
	- 52 + 25	11 11	- 0.20 0.00	0 + 15 + 30	10 10 10	- 0.40 - 0.20 0.00	- 10 + 5 + 35 + 50	15 12 / 16 13 12	+ 0.20 0.00 + 0.30 + 0.45
Interpolated results	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(\lambda_{meas}-\lambda)}{\lambda}\right)$ [%]	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(\lambda_{meas}-\lambda)}{\lambda}\right)$ [%]	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(\lambda_{meas}-\lambda)}{\lambda}\right)$ [%]
	- 10 0 + 10 + 20 + 30	11 11 11 11 11	- 0.10 - 0.10 0.00 0.00 0.00	- 5 0 + 10 + 20 + 30	10 10 10 10 10	- 0.45 - 0.40 - 0.25 - 0.15 0.00	- 10 0 + 20 + 30 + 40 + 50	15 15 14 13 13 12	+ 0.20 0.00 + 0.15 + 0.25 + 0.35 + 0.45
Laboratory	LNE			NPL			SP		
Results obtained from the linearity tests	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(\lambda_{meas}-\lambda)}{\lambda}\right)$ [%]	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(P_{meas}-P)}{P}\right)$ [%]	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(\lambda_{meas}-\lambda)}{\lambda}\right)$ [%]
	- 10 0 + 20 + 50	15 15 15 15	- 0.40 - 0.45 0.00 - 0.50	+ 12 + 24 + 35	16 16 16	+ 0.20 + 0.30 + 0.10	- 10 + 10 + 30	20 20 20	+ 0.10 + 0.40 + 0.60
Interpolated results	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(\lambda_{meas}-\lambda)}{\lambda}\right)$ [%]	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(P_{meas}-P)}{P}\right)$ [%]	Mean temperature [°C]	Temperature difference [°C]	Relat. error $\left(\frac{(\lambda_{meas}-\lambda)}{\lambda}\right)$ [%]
	- 10 0 + 10 + 20 + 25 + 30 + 40 + 50	15 15 15 15 15 15 15 15	- 0.40 - 0.45 - 0.20 0.00 - 0.15 - 0.15 - 0.30 - 0.50	0 + 20 + 30 + 40	16 16 16 16	+ 0.10 + 0.25 + 0.20 0.00	- 10 0 + 10 + 20 + 30	20 20 20 20 20	+ 0.10 + 0.25 + 0.40 + 0.50 + 0.60

Table A7a: Synthesis of the errors resulting from the linearity test analysis

ANNEX 7

Laboratory	DFT			EMPA			FIW		
LOW DENSITY SAMPLE • Identification • Density	• I13a - F66b • 66.4 - 67.1 kg/m³			• A54a - C55b • 71.2 - 71.0 kg/m³			• B21b - H22b • 68.7 - 69.5 kg/m³		
• Measurements results	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]
	- 9.96	34.63	28.54	- 5.00	34.70	28.64	- 9.96	34.48	28.32
	0.08	34.65	29.61	0.00	34.70	29.07	0.52	34.49	29.58
	0.22	34.66	29.60	10.00	34.70	30.28	19.29	34.50	31.55
	10.06	34.68	30.61	20.00	34.70	31.46	30.22	34.51	32.77
	10.13	34.68	30.59	30.00	34.70	32.75	38.60	34.51	33.85
	20.05	34.71	31.70				49.48	34.52	35.31
	20.09	34.71	31.71						
	33.41	34.75	33.35						
	33.45	34.75	33.33						
HIGH DENSITY SAMPLE • Identification • Density				• E32a - E11b • 73.5 - 74.8 kg/m³			• H45a - B31a • 75.0 - 75.2 kg/m³		
• Measurements results				Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]
				- 5.00	34.75	28.74	- 9.92	34.48	28.38
				0.00	34.75	29.28	0.70	34.49	29.59
				10.00	34.75	30.32	19.22	34.50	31.55
				20.00	34.75	31.49	30.85	34.51	32.72
				30.00	34.75	32.81	50.56	34.52	35.30

Table A7b: Synthesis of the corrected thermal conductivity measurements retained for the process of certification (1/2)

Laboratory	LNE			NPL			SP		
LOW DENSITY SAMPLE • Identification • Density	• E12a - E62b • 69.3 - 70.2 kg/m³			• B15b • 71.5 kg/m³			• C55d • 66.0 kg/m³		
• Measurements results	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]
	- 10.19	34.33	28.22	- 0.05	34.27	29.18	- 10.00	34.63	28.40
	- 10.09	34.33	28.24	0.03	34.15	29.06	0.00	34.65	29.46
	0.00	34.36	29.28	19.72	34.28	31.35	10.00	34.68	30.55
	9.98	34.40	30.36	19.83	34.19	31.28	20.00	34.70	31.73
	20.00	34.43	31.46	29.50	34.26	32.45	30.00	34.72	32.86
	20.03	34.43	31.48	29.11	34.21	32.35			
	25.05	34.44	32.10	39.35	34.29	33.62			
	30.05	34.45	32.74	39.53	34.25	33.63			
	40.00	34.46	33.99						
	49.98	34.48	35.22						
	50.04	34.48	35.29						
HIGH DENSITY SAMPLE • Identification • Density	• E41a - D61a • 74.0 - 73.8 kg/m³			• E31b • 75.3 kg/m³			• F66e • 75.0 kg/m³		
• Measurements results	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]	Mean temperature [°C]	Specimen Thickness [mm]	Thermal conductivity [mW/(m.K)]
	- 9.99	34.34	28.25	0.03	34.42	29.22	- 10.00	34.53	28.41
	- 9.91	34.34	28.27	0.04	33.88	29.22	0.00	34.55	29.46
	- 0.02	34.37	29.32	19.79	34.47	31.43	10.00	34.58	30.49
	19.99	34.44	31.44	19.76	34.39	31.32	20.00	34.60	31.65
	20.00	34.44	31.43	29.57	34.50	32.56	30.00	34.62	32.74
	29.98	34.46	32.67	29.14	34.36	32.35			
	40.00	34.47	33.86	39.07	34.52	33.72			
	49.99	34.49	35.19	38.95	34.43	33.49			
	50.00	34.49	35.18						

Table A7b: Synthesis of the corrected thermal conductivity measurements retained for the process of certification (2/2)

EVALUATION OF THE THERMAL CONDUCTIVITY RESULTS

• Linear regression of the thermal conductivity results

Laboratory	Sample density	Polynomial λ [mW/(m.K)] = f(θ [°C])
DFT	LOW DENSITY: 66.8 kg/m³	$29.576 + 0.102 \times \theta + 9.56\ 10^{-6} \times \theta^3$
EMPA	LOW DENSITY: 71.1 kg/m³	$29.132 + 0.108 \times \theta + 4.32\ 10^{-4} \times \theta^2$
	HIGH DENSITY: 74.2 kg/m³	$29.268 + 0.105 \times \theta + 1.47\ 10^{-5} \times \theta^3$
FIW	LOW DENSITY: 69.1 kg/m³	$29.446 + 0.107 \times \theta + 4.56\ 10^{-6} \times \theta^3$
	HIGH DENSITY: 75.1 kg/m³	$29.463 + 0.104 \times \theta + 4.37\ 10^{-6} \times \theta^3$
LNE	LOW DENSITY: 69.8 kg/m³	$29.274 + 0.106 \times \theta + 2.84\ 10^{-4} \times \theta^2$
	HIGH DENSITY: 73.9 kg/m³	$29.275 + 0.103 \times \theta + 2.95\ 10^{-4} \times \theta^2$
NPL	LOW DENSITY: 71.5 kg/m³	$29.098 + 0.114 \times \theta$
	HIGH DENSITY: 75.3 kg/m³	$29.192 + 0.112 \times \theta$
SP	LOW DENSITY: 66.0 kg/m³	$29.483 + 0.112 \times \theta$
	HIGH DENSITY: 75.0 kg/m³	$29.467 + 0.108 \times \theta$

Table A7c: Linear regression polynomial of λ versus θ for the participants

- **Individual measurements, average, standard deviation and uncertainty (θ = - 10 °C)**

Laboratory	Thermal conductivity statistical analysis [W/(m.K)]			
	Individual measurement results	Average value	Standard deviation	Standard uncertainty
DFT	0.02854	0.02854	-	0.000173
FIW	0.02831 0.02837	0.02834	0.000039	0.000141
LNE	0.02825 0.02824 0.02825 0.02826	0.02825	0.000009	0.000151
SP	0.02840 0.02841	0.02841	0.000007	0.000103

Diagram of averages and their expanded uncertainties at the 95 % confidence interval

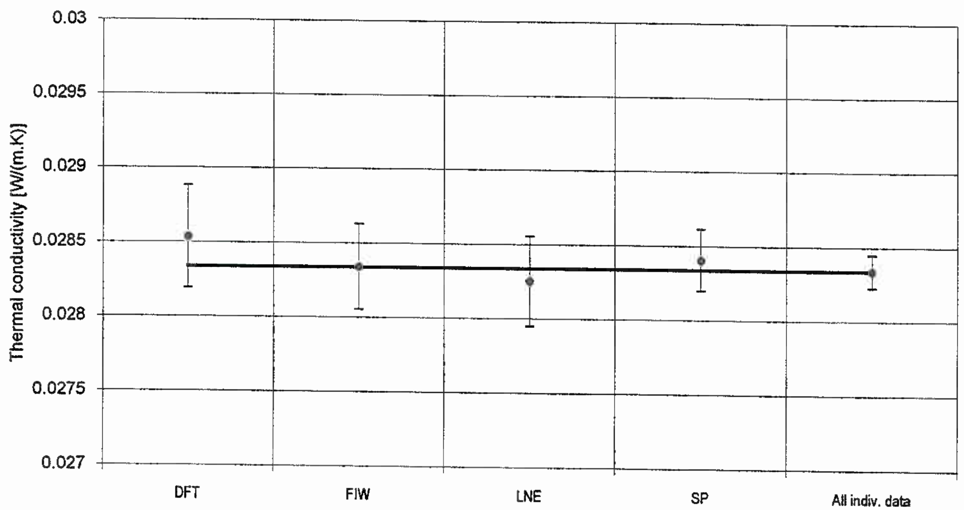

Table A7d: Statistical analysis on λ results at the temperature level θ = - 10 °C

- **Individual measurements, average, standard deviation and uncertainty (θ = 0 °C)**

Laboratory	Thermal conductivity statistical analysis [W/(m.K)]			
	Individual measurement results	Average value	Standard deviation	Standard uncertainty
DFT	0.02960 0.02958	0.02959	0.000017	0.000180
EMPA	0.02916 0.02907 0.02926 0.02928	0.02919	0.000094	0.000293
FIW	0.02952 0.02952	0.02952	0.000005	0.000147
LNE	0.02928 0.02932	0.02930	0.000030	0.000157
NPL	0.02919 0.02906 0.02922 0.02922	0.02917	0.000076	0.000223
SP	0.02946 0.02946	0.02946	0.000000	0.000107

Diagram of averages and their expanded uncertainties at the 95 % confidence interval

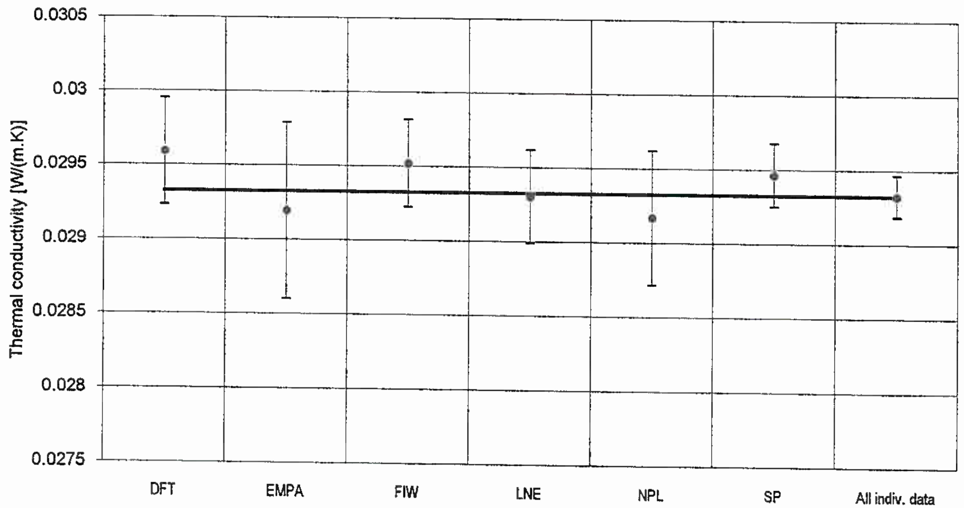

Table A7e: Statistical analysis on λ results at the temperature level θ = 0 °C

ANNEX 7

- **Individual measurements, average, standard deviation and uncertainty (θ = + 10 °C)**

Laboratory	Thermal conductivity statistical analysis [W/(m.K)]			
	Individual measurement results	Average value	Standard deviation	Standard uncertainty
DFT	0.03060 0.03057	0.03059	0.000019	0.000111
EMPA	0.03028 0.03032	0.03030	0.000028	0.000305
LNE	0.03036	0.03036	-	0.000162
SP	0.03049 0.03055	0.03052	0.000042	0.000111

Diagram of averages and their expanded uncertainties at the 95 % confidence interval

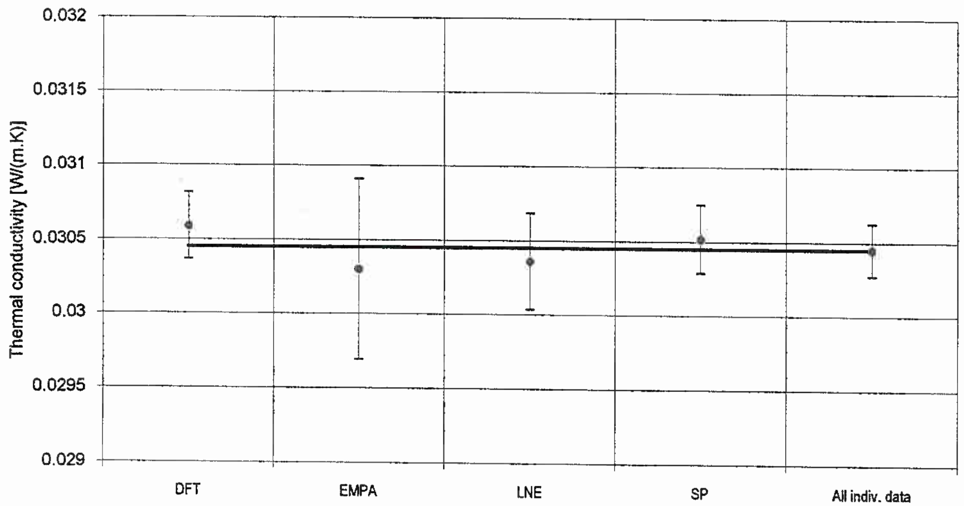

Table A7f: Statistical analysis on λ results at the temperature level θ = + 10 °C

ANNEX 7

- **Individual measurements, average, standard deviation and uncertainty (θ = + 20 °C)**

Laboratory	Thermal conductivity statistical analysis [W/(m.K)]			
	Individual measurement results	Average value	Standard deviation	Standard uncertainty
DFT	0.03169 0.03170	0.03170	0.000004	0.000115
EMPA	0.03146 0.03149	0.03147	0.000021	0.000316
FIW	0.03164 0.03163	0.03163	0.000004	0.000158
LNE	0.03146 0.03148 0.03144 0.03143	0.03145	0.000021	0.000168
NPL	0.03138 0.03130 0.03145 0.03135	0.03137	0.000065	0.000240
SP	0.03165 0.03173	0.03169	0.000056	0.000115

Diagram of averages and their expanded uncertainties at the 95 % confidence interval

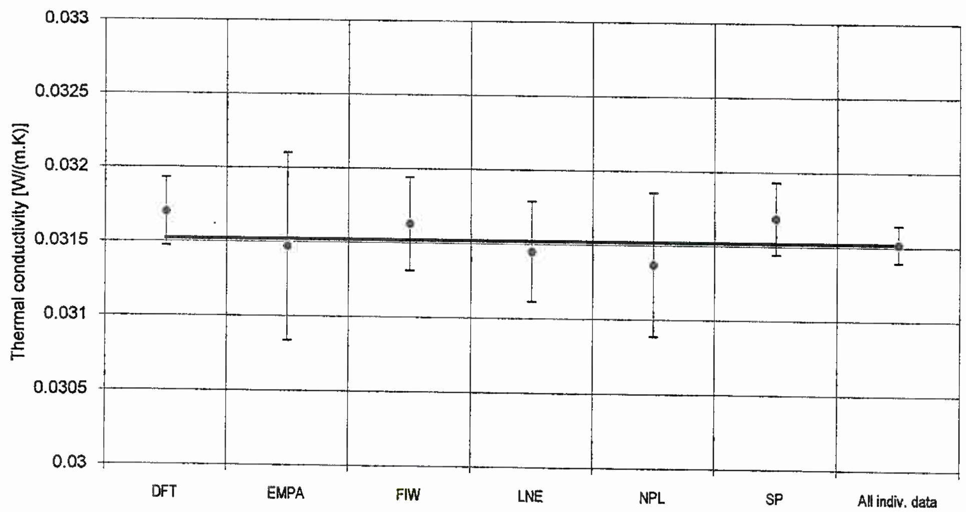

Table A7g: Statistical analysis on λ results at the temperature level θ = + 20 °C

• **Individual measurements, average, standard deviation and uncertainty (θ = + 30 °C)**

Laboratory	**Thermal conductivity statistical analysis [W/(m.K)]**			
	Individual measurement results	Average value	Standard deviation	Standard uncertainty
DFT	0.03290 0.03287	0.03289	0.000018	0.000120
EMPA	0.03275 0.03281	0.03278	0.000042	0.000329
FIW	0.03263 0.3275	0.03269	0.000086	0.000163
LNE	0.03270 0.03273 0.03267	0.03270	0.000031	0.000175
NPL	0.03250 0.03245 0.03261 0.03244	0.03250	0.000080	0.000249
SP	0.03274 0.03286	0.03280	0.000084	0.000119

Diagram of averages and their expanded uncertainties at the 95 % confidence interval

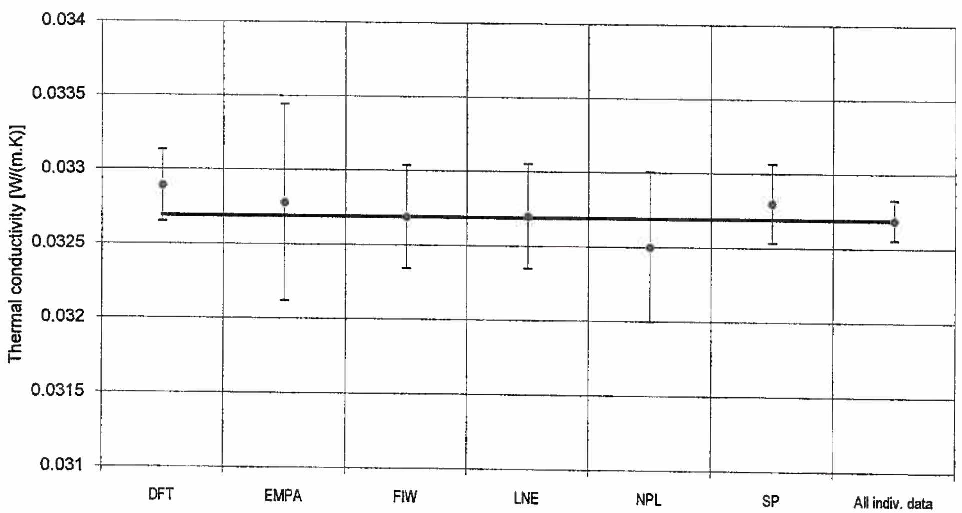

Table A7h: Statistical analysis on λ results at the temperature level θ = + 30 °C

- **Individual measurements, average, standard deviation and uncertainty (θ = + 40 °C)**

Laboratory	Thermal conductivity statistical analysis [W/(m.K)]			
	Individual measurement results	Average value	Standard deviation	Standard uncertainty
FIW	0.03403	0.03403	-	0.000170
LNE	0.03399 0.03386	0.03393	0.000092	0.000181
NPL	0.03369 0.03368 0.03382 0.03361	0.03370	0.000090	0.000258

Diagram of averages and their expanded uncertainties at the 95 % confidence interval

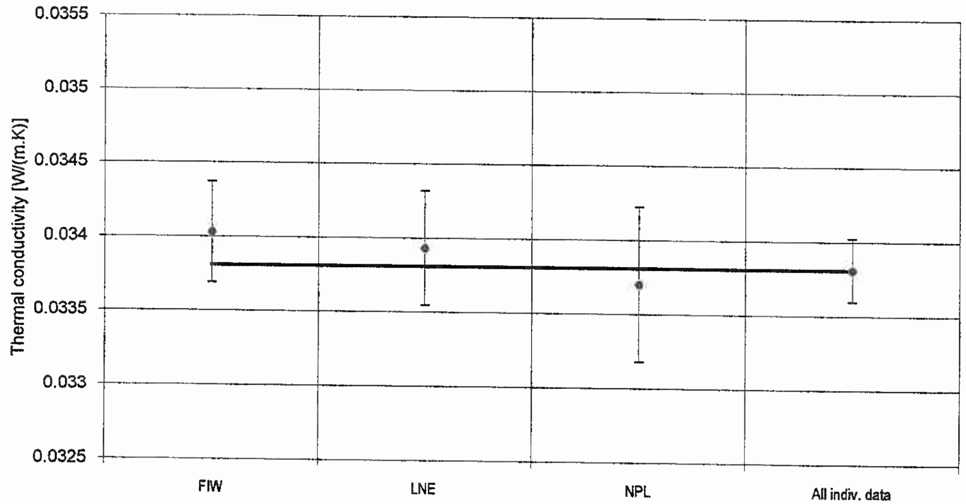

Table A7i: Statistical analysis on λ results at the temperature level θ = + 40 °C

- **Individual measurements, average, standard deviation and uncertainty (θ = + 50 °C)**

Laboratory	Thermal conductivity statistical analysis [W/(m.K)]			
	Individual measurement results	Average value	Standard deviation	Standard uncertainty
FIW	0.03522 0.03538	0.03530	0.000113	0.000176
LNE	0.03522 0.03528 0.03519 0.03518	0.03522	0.000047	0.000188

Diagram of averages and their expanded uncertainties at the 95 % confidence interval

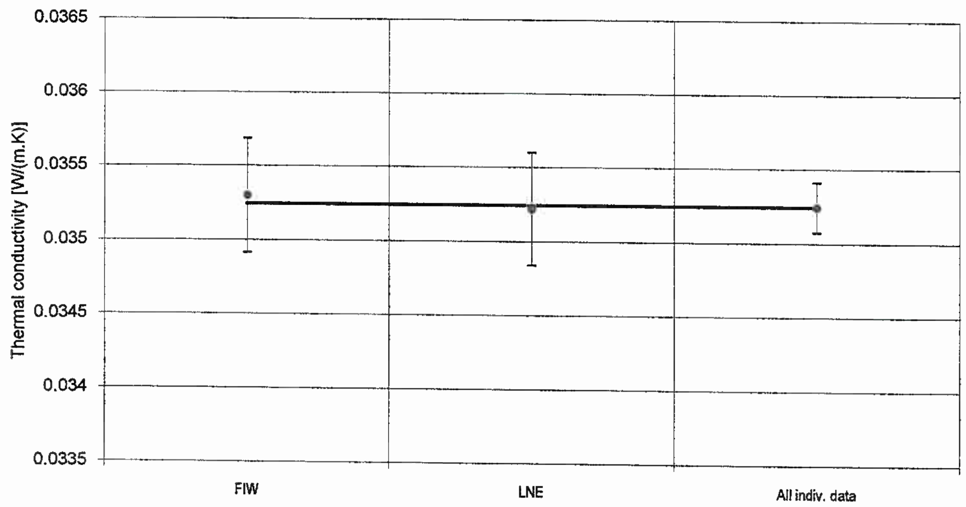

Table A7j: Statistical analysis on λ results at the temperature level θ = + 50 °C

- **Statistical analysis results (at the stage of calculating the certified values and the uncertainty)**

Thermal conductivity at the temperature level	θ = - 10 °C	θ = 0 °C	θ = + 10 °C	θ = + 20 °C	θ = + 30 °C	θ = + 40 °C	θ = + 50 °C
Number of data sets	4	6	4	6	6	3	2
Number of individual data	9	16	7	16	15	7	6
All data sets compatible two by two ? (Scheffe multiple t-test) /1/	yes	yes	yes	yes	yes	yes	yes
Outlying data sets? (Dixon test, Nalimov t-test and Grubbs test) /1/	no	no	no	no	no	no	no
Outlying variances ? (Cochran test) /1/	no	no	no	no	no	no	no
Average of data set averages [W/(m.K)]	0.02838	0.02937	0.03044	0.03155	0.03273	0.03389	0.03526
Within-data sets standard deviation [W/(m.K)]	0.00002	0.00007	0.00003	0.00004	0.00006	0.00009	0.00007
Between-data sets standard deviation [W/(m.K)]	0.00008	0.00018	0.00014	0.00014	0.00014	0.00015	0.00005
Between-data sets combined standard uncertainty significantly different from 0 ? (Snedecor F-test) /1/	no	no	no	no	no	no	no
Variance homogeneous ? (Bartlett test) /1/	yes	yes	yes	yes	yes	yes	yes
Std deviation of pooled individual data [W/(m.K)]	0.00010	0.00018	0.00013	0.00014	0.00015	0.00016	0.00008
Pooled individual data normally distributed? (Kolmogorov - Smirnov - Lilliefors test ; skewness test and kurtosis test) /1/	yes	yes	yes	yes	yes	yes	yes
Half-width of the 95 % confidence interval of the average of all individual data [W/(m.K)]	0.00008	0.00009	0.00012	0.00007	0.00008	0.00015	0.00008
Half-width of the 95 % tolerance interval of the pooled individual data [W/(m.K)]	0.00054	0.00079	0.00078	0.00060	0.00066	0.00098	0.00052

/1/ The tests are performed at the 0.05 and 0.01 levels of significance.

Table A7k: Summary of statistical data for IRMM-440

15

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