Example of equipment manual for HFM “black box” equipment

"LASERCOMP FOX 600"
HEAT FLOW METER apparatus

LC000313

Product Quality Laboratory
Denmark

Version 2005-10-28
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1. **Introduction**

This assessment report provides information showing that the Product Quality Laboratory (PQL) as well as the specified heat flow meter (HFM) apparatus fulfil the requirements of NB SG 19 N 58. This includes the laboratory documentation required in EN1946, parts 1 and 3, as well as the equipment requirements of EN12667.

**Certification:** The PQL is certified according to ISO 9001:2000, and is participating in thermal conductivity inter comparison measurements with other laboratories.

**Internal testing only:** The PQL only performs internal testing and is not accredited or notified. However, the intention is to work as if it were a notified laboratory.

**Laboratory competence:** The PQL has more than twenty years of experience with thermal conductivity ($\lambda_{10}$), density and SLR (standard air resistance) measurements according to the relevant ISO and EN test and product standards for insulation materials.

**The PQL staff and used equipment:** The PQL staff is very experienced. Two of the operators (Pernille Søgaard Jensen and Jens Hansen) have more than twenty years’ experience with $\lambda_{10}$ measurements.

The state-of-the-art $\lambda_{10}$ equipment is supplied by LaserComp and is designed to fulfil the requirements of the standards EN 12667, ISO 8301 and EN 1946-3.

This equipment manual refers to the PQL LaserComp FOX 600 apparatus no. LC000313. This equipment manual serves as a master for the Rockwool Group’s LaserComp FOX 600 apparatus. Specific assessment tests will be reported separately related to each individual apparatus.
2. **Test method**

The test method is described in detail in the test method T.RI 421-1, [1].

The measurement is only performed on dried samples, hence moisture transport does not need to be considered.

3. **Equipment performance specifications**

The performance of the equipment has been checked for the limits shown in table 1 which only represents a narrow range of the measuring capacity of the equipment. The limits stated by the supplier are given in [3].

<table>
<thead>
<tr>
<th>Parameter</th>
<th>FOX 600</th>
<th>Unit</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Minimum thickness non-rigid products (e.g. MW and FEF(^1))</td>
<td>0.03</td>
<td>m</td>
<td>EN 1946-3 (4.4.7) EEQ-04-2666 [2]</td>
</tr>
<tr>
<td>Minimum thickness of rigid products(^2) (e.g. EPS, XPS)</td>
<td>cf. table 2: Flatness tolerances related to specimen thermal resistance in 1946-3.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maximum thickness</td>
<td>0.150</td>
<td>m</td>
<td>EN 1946-3 (4.4.2) RDQ-04-3161rev. [12]</td>
</tr>
<tr>
<td>Minimum thermal conductivity for practical testing</td>
<td>0.019</td>
<td>W/mK</td>
<td>RI database</td>
</tr>
<tr>
<td>Maximum thermal conductivity for practical testing</td>
<td>0.048</td>
<td>W/mK</td>
<td>RI database</td>
</tr>
<tr>
<td>Temperature difference across specimen</td>
<td>20 (^\circ)K</td>
<td>T.RI 421-1 [1]</td>
<td></td>
</tr>
<tr>
<td>Temperature cold side(^3)</td>
<td>0 ± 0.1 (^\circ)C</td>
<td>T.RI 421-1 [1]</td>
<td></td>
</tr>
<tr>
<td>Temperature hot side</td>
<td>20 ± 0.1 (^\circ)C</td>
<td>T.RI 421-1 [1]</td>
<td></td>
</tr>
<tr>
<td>Room temperature</td>
<td>18 ± 1 (^\circ)C</td>
<td>T.RI 421-1 [1]</td>
<td></td>
</tr>
<tr>
<td>Max. laboratory humidity</td>
<td>30 v.18(^\circ)C % RH</td>
<td>T.RI 421-1 [1]</td>
<td></td>
</tr>
</tbody>
</table>

\(^1\)Elastomeric foam products.

\(^2\)Cellular glass is outside our area of expertise – the use of contact sheets may be considered if indicative measurements are performed.

\(^3\)The cold and hot plates are able to operate with the limits -10\(^\circ\)C and 65\(^\circ\)C respectively, according to ref. [3].
Table 2: Specification of internal control specimens used for FOX 600.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Control specimen</th>
<th>Unit</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal conductivity range</td>
<td>0.0310 - 0.0365</td>
<td>W/mK</td>
<td>RI database</td>
</tr>
<tr>
<td>Thickness range</td>
<td>0.0345 – 0.1505</td>
<td>m</td>
<td>RI database</td>
</tr>
<tr>
<td>Types of material</td>
<td>MW, EPS</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

4. Equipment description

4.1. Principle of operation and type of apparatus

The HFM method is used for determining thermal resistance. The LaserComp FOX 600 apparatus is designed as a “Single-specimen symmetrical configuration” as illustrated in figure 2b in EN12667. The design of the apparatus is according to ASTM C518-91 and ISO 8301.

A test specimen is placed centrally in the apparatus between the hot and cold plates. The lower plate is stationary, and the upper plate can move up- and downwards by four independently controlled motors. Using the supplier default conditions the upper plate temperature is controlled at 0°C, and the lower plate temperature at 20°C. However, in order to avoid air movement at the edges of the specimen the upper plate must be controlled at 20°C. The apparatus is placed in a climate controlled room with a temperature of 18°C (+/- 1 °C) and a dry environment with the relative humidity adjusted to approx. 25% (max. 30%).

A HFM and one thermocouple are embedded on each plate. The average signal of the upper and lower HFM is proportional to the heat flow through the specimen and is used to calculate the thermal conductivity of the specimen. The temperature difference is calculated as the difference between the temperatures measured at the centre of each plate.

4.2. Principal dimensions of apparatus

The overall specifications of the apparatus according to the supplier [3] are summarised in table 3:

Table 3: Overall specification of FOX 600

<table>
<thead>
<tr>
<th>LaserComp</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Model</td>
<td>FOX 600</td>
</tr>
<tr>
<td>Id. No.</td>
<td>000313</td>
</tr>
<tr>
<td>Supplier</td>
<td>LaserComp Inc., Massachusetts, USA</td>
</tr>
<tr>
<td>Max. specimen size</td>
<td>610 x 610 mm²</td>
</tr>
<tr>
<td>Min. specimen size</td>
<td>560 x 560 mm² without insulation</td>
</tr>
<tr>
<td>Measuring area</td>
<td>305 x 305 mm²</td>
</tr>
<tr>
<td>Heat flow sensors</td>
<td>&gt;1000 thermocouples</td>
</tr>
</tbody>
</table>
4.3. The design of the equipment

A sketch of the apparatus is shown in figure 1.

Figure 1: Sketch of FOX 600

Thickness measurement
The FOX 600 apparatus consists of the test chamber and the key-pad display section. When the door of the chamber is opened, the specimen can be placed between two metal plates. The lower plate is stationary and the upper plate can move up- and downwards by four independently controlled motors. Four thickness read-out digital sensors independently monitor the position of each corner of the upper plate. Each time a specimen is inserted into the apparatus, and the stack is closed, the average thickness of the specimen is determined within $\pm 0.15$ mm, ref. [4].

Heating and cooling plates
The plates are made of 5/8" (~16 mm) aluminium and are equipped with a liquid heating/cooling system based on Peltier elements with secondary cooling by water. With cooling water at max 18°C the temperature of the plates can be independently controlled within $\pm 0.02$°C, ref. [3].

Heat flow meters
The HFMs are fixed to the surfaces of both plates and are covering an area of 305 mm by 305 mm. The HFM thickness is about 1 mm. Type E thermocouples are fixed in the center of each HFM within ~0.08 mm of the surface in contact with the specimen and provide readings of the temperatures of both specimen surfaces.
4.4. Electrical components

The apparatus is controlled by a Digital Signal Processor (DSP) which controls all apparatus operation, such as motor control for precise determination of the specimen thickness, readings of signals from the HFMs and thermocouples. The DSP also performs a PID algorithm to control temperatures of both plates at set-points temperatures.

A 24 bit Analog Digital Converter (ADC) converts the analog signals of thermocouples and HFMs to digital signals with a resolution of 0.6 µV giving a resolution of the temperature measurement of 0.01°C, ref. [3].

The user of the apparatus does not have access to the raw electrical signals from HFMs and thermocouples. The measurement and data acquisition is a “black box” which cannot be controlled, and this creates an issue complying with the specifics of ISO 8301 and EN 12667.

4.5. Data acquisition, data analysis and related computer programs

The thermal equilibrium status is monitored and evaluated every 6 minutes (duration of 1 block). A set of equilibrium criteria have to be met independently before the specimen is declared to be in thermal equilibrium. Typically, the last 5 blocks at steady-state conditions are used to calculate the results.

During the run, a set of 512 successive data acquisition cycles is organized in one block. The duration of one block is 6 minutes. The data consist of the upper and lower plate’s temperatures, and the upper and lower plate’s transducers signals. The data in each block are averaged.

The block averages are compared to the respective average values of the previous block. Once this comparison satisfies all the equilibrium criteria, the specimen is accepted to be in thermal equilibrium. The thermal conductivity is calculated based on the results from the last 5 blocks which fulfilled the equilibrium criteria. The equilibrium criteria are described in detail in [3].

**Temperature equilibrium criterion:**
Default 0.2°C. The average temperature of each plate must be equal to the plate’s set-point temperature within ± 0.2°C.

**Between-block HFM equilibrium criterion:**
Default 150 µV. To meet this criterion, HFM average signal of two successive blocks must be the same within the criterion.
HFM percent equilibrium criterion:
Default 2.0% for thickness < 50 mm, and 2.5% for other thicknesses. To meet this criterion the HFM average signal of two successive blocks must be the same within the criterion.

Number of blocks of percent equilibrium:
Default: 8 blocks. The number of successive blocks satisfying the percent equilibrium criterion required, declaring that thermal equilibrium has been reached, and the results can be calculated.

Steady-state criterion:
It requires that the average HFM signal for successive blocks that meet the percent equilibrium criterion does not change monotonically. The difference between two blocks must change sign or be equal to zero. If this criterion is not met, the test is prolonged, even if the other criteria are satisfied, and more blocks are collected until the steady-state criterion is fulfilled.

The settings of the HFM percent equilibrium criterion and minimum number of blocks to be used when testing mineral wool (MW) have been investigated depending on specimen density and thickness [5]. These settings are used within the RI Group and are mapped in the RI test method [1].

The apparatus is connected to a personal computer through a RS-232 interface. This interaction is controlled by LaserComp’s "WinTherm32" software [6]. All setup data and test data are automatically stored in the computer. After a test run or calibration run the data acquisition process is performed and a report is generated.

Test results and test parameters (including raw data from the HFM) are collected and stored in a database using a database interface program developed by LaserComp for the data export. In the database specimen-related information about production parameters, product density and dimensions are added by the operator.

5. Equipment design and uncertainty analysis

5.1. Uncertainty sources
Uncertainty in the final thermal conductivity measurement may be influenced by uncertainty introduced from the following sources:

- The measuring equipment
- The specimen measured
- Imported uncertainties – calibration specimen
- Operator
- The environment
The test procedure [1] is designed to eliminate or minimise uncertainties from the measurement process, the specimen measured, the operator and the environment. These sources will not be treated further in this manual. However, deviation of the measurement on control specimens treated in section 6.2 will reflect the uncertainty containing all the elements.

The uncertainty sources related to the final measurement have been divided into three groups:

- Uncertainty related to components in the apparatus, e.g. uncertainty in measuring heat flux, \((W/m^2)\), thickness (m) and temperature difference (K). The uncertainty budget of the apparatus is estimated in [8] according to GUM [7].

- Uncertainty related to test conditions being not ideal e.g. the temperature of the measuring plates not being uniform and/or edge heat loss. These contributions are further described in sections 5.2 and 5.4.

- Imported uncertainty related to the calibration specimen, cf. in section 5.7.

The largest possible uncertainty due to the three groups of uncertainties when measuring thermal conductivity is estimated in section 5.12. The accuracy shall be within the level defined by the SDG-5 implementation group i.e. ± 1.5% [10] and is intended to be demonstrated in an annual program on test specimens traceable to the European \(\lambda_{10}\) level.

5.2. Edge heat losses and maximum specimen thickness

According to ISO 8301 (2.2.5.3) the edge heat loss error shall be kept within 0.5%. As no suitable theoretical analysis is available to predict the maximum allowed specimen thickness, the analysis for the guarded hot plate may be used as a guide (ISO 8301, 1.7.2.2). On this basis Table 1 in EN1946-3 sets up the limits for maximum and minimum specimen thicknesses by apparatus design.

In the case of a single specimen symmetrical configuration, an overall apparatus length of 600 mm, a metering section of 300 mm and a guard width of 150 mm, the maximum allowed specimen thickness which fulfills the above criterion is 150 mm according to table 1 in EN1946-3.

5.2.1. Evaluation of edge heat losses

The edge temperature profile has been measured on two edges of a 150 mm, 121 kg/m³ Rockwool specimen under two different test
conditions: when the edge of the specimen is in direct contact with the edge of the apparatus, and when there is an air gap of approx. 20 mm between the specimen and the edges of the apparatus. The measured temperature profiles are shown in figure 2.

![Edge temperature profiles](image)

**Figure 2:** Edge temperature profile. Results from two tests are shown; with contact between specimen edge and apparatus and with an air gap of approx 2 cm between specimen and apparatus. Specimen thickness 150 mm and room temperature 18°C.

The measured values at the center of the two edges were 12.2°C when there was contact between specimen edge and apparatus. With an air gap of 2 cm around the specimen the temperature at the centre decreases to 9.4°C and 6.2°C, respectively. The temperature profile with an air gap is clearly influenced by convection in the gap, which is due to the apparatus having the cold surface as the upper plate. **To avoid convection in the gap it is recommended to change the temperature of the upper plate to 20°C for all future calibrations.**

For the above-mentioned case, the difference between the two measurements with and without air gap is 0.08%, which is less than the measurement repeatability with the specimen maintained in the apparatus. This indicates that the edge insulation, when measuring 150 mm thick specimens, is sufficient to minimise edge heat losses to acceptable levels.

Fluctuations in the measurement of the edge temperature with an air gap indicate the existence of convection in the air gap. Figure 3 shows the temperature measurement on edge 1 with and without an air gap.
Figure 3: Edge temperatures measured on edge 1 (back side of the apparatus) with and without an air gap between specimen and apparatus. Specimen thickness 150 mm.

The edge heat loss is zero for homogeneous isotropic specimens when the edge temperature ratio \( e = 0.5 \) (EN 1946-3, 4.4.2 note), \( e = \frac{T_e - T_2}{T_1 - T_2} \).

**Edge temperature ratio:** For an edge temperature of 12.2°C we have the temperatures: \( T_1=20°C; \) \( T_2=0°C \) and \( T_e=12.2°C \); and the edge temperature ratio becomes \( \frac{T_e - T_2}{T_1 - T_2} = \frac{(12.2 - 0)}{(20 - 0)} = 0.61 \). This is within the acceptable range of \( 0.25 < e < 0.75 \).

As the requirement for the edge temperature ratio is fulfilled, the edge heat loss can be assumed to be within 0.5% for specimen thicknesses up to 150 mm and for test specimens without convection at the edge between specimen and apparatus.

The effect of different edge temperature conditions has been modelled using a three-dimensional software program. However, this analysis is not yet finalised.

### 5.3. Minimum specimen thickness

The minimum measurable specimen thickness is related to the flatness tolerance. According to Table 1 in EN 1946-3 the minimum specimen thickness may not be less than 5% of the overall apparatus size.
Hence the minimum allowed thickness for a single specimen symmetrical configuration apparatus is 30 mm when the overall plate size is 600 mm and the metering section size is 300 mm.

The error due to the flatness tolerance may not exceed 0.5% (EN12667).

5.4. **Temperature uniformity of heating and cooling plates**

According to ISO 8301 (2.2.1.2) the temperature uniformity of the working surface of the plates must be better than 1% of the temperature difference between the plates (equals 0.2°C). According to the supplier, the heating and cooling system consists of central and peripheral groups of thermoelectric elements which are controlled independently to eliminate radial temperature gradients in the plates, and they are controlled within ± 0.02°C [3]. The temperature uniformity has been controlled and is reported in relation to the performance check.

The deviation from uniform temperature is measured to be 0.04°C and 0.16°C on the hot and cold surfaces, respectively [8]. This fulfils the criterion specified in EN1946-3, but is not as good as declared by the supplier.

The error from the surface temperature not being uniform has not been taken into account in the uncertainty budget, but this issue will be investigated further in the 3D analysis of the apparatus design.

5.5. **Uncertainty in temperature difference**

According to ISO 8301 (2.2.1.2) and EN1946-3 (4.4.5), the uncertainty in the temperature difference measured may not exceed 1%. In [8] the temperature gradient measured by the apparatus was 20.00°C, and the average gradient from the RI measurement was 20.13°C.

The uncertainty in measured temperature gradient is included in the maximum probable error in the uncertainty budget in annex A.

In the apparatus one thermocouple is mounted at the centres of the upper and lower surfaces of the plates to monitor the temperature difference. This design does not fully conform to ISO 8301 (3.2.2.2.1), which requests a minimum of 2 thermocouples on each side of the specimen. However, the performance check proves that the LaserComp design sufficiently fulfils the performance requirements.

The temperature measured by the apparatus was 0.02°C and 20.02°C, respectively while the absolute temperature measured by the calibrated thermocouples was -0.62°C and 19.51°C, respectively. The uncertainty
of the calibration is +/- 0.1°C. This corresponds to a mean temperature of 9.45°C +/- 0.1°C. However it is necessary to ensure that all testing is done within the set point of 10 ± 0.3°C as required by EN13171.

As the performance check of the temperature measurement was carried out while measuring a specimen it is recommended to repeat the test. Preferably having the same hot and cold plate temperatures or using a smaller temperature gradient between the plates to reduce any temperature errors introduced due to the interface resistance between the temperature sensors, specimen and the apparatus.

If the problem still exists after checks with a lower temperature gradient across the specimen then changes to the software temperature algorithm must be made.

5.6. Uncertainty in measurement of specimen thickness

The uncertainty in thickness adjustment has been investigated in [9] and is found to be well within the acceptable limit of 0.5% (ISO 8301). The deviation from the setting was 0.05 mm (0.10%), 0.01 mm (0.01%) and 0.12 mm (0.08%) when controlling the thicknesses 48 mm, 97mm and 148 mm, respectively. Adding the accuracy of the digital slide gauge used for the control, the deviation from settings equals 0.13%, 0.02% and 0.09%, respectively.

The uncertainty in thickness measurement is included in the uncertainty budget in annex A.

Parallelism is not as critical as flatness according to EN12667, A.3.7 and the maximum deviation from parallelism for specimen surfaces is defined by the requirements that the specimen thickness may not differ from the mean value by more than 2% EN12667, B.5

Deviation from plane parallelism is part of the calibration check and was measured to be 0.19 mm, 0.25 mm and 0.13 mm when controlling the thicknesses at 48 mm, 97mm and 148 mm, respectively. This equals a deviation in specimen thickness from mean value of less than 0.5%. The deviation from plane parallelism may be due to the unevenness in the plate surfaces the deviations have be included in the uncertainty budget for thickness measurement.

5.7. Accuracy of calibration specimen

The calibration specimens used are of type IRMM-440. The relative uncertainty of the calibration specimens at a mean temperature of 10°C according to the certificate is 0.9% at the 95% confidence interval [9].

The imported uncertainty related to the calibration specimen is included in the uncertainty budget in annex A.
5.8. Non-linearity of the calibration

The linearity as function of temperature has not been investigated, as the temperatures always are kept at 0°C and 20°C when measuring thermal conductivity in PQL.

The linearity of thermal resistance as function of thickness is shown in Figure 4 based on the calibration done in January 2004. As the thermal resistance shows a linear relationship with thickness (and heat flow) it confirms that there is no need to take edges losses into account.

![Graph showing thermal resistance as function of thickness](image)

**Figure 4**: Deviation from a linear relationship between thermal resistance and thickness when measuring a combination of calibration specimens IRMM A, B, C and D.

The uncertainty due to the graph not passing through the origin is between 0.40% at 35 mm and 0.10% at 138 mm. The requirement in EN1946-3 is that the line which interpolates the measured thermal resistance may not deviate from a straight line by more than 0.5%. The calibration check is within this limit.

5.9. Calibration drifts

The apparatus is calibrated against IRMM 440 specimens [9], which define the European $\lambda_{10}$ level using a thickness of 103 mm.

The calibration factors are verified by measuring IRMM 440 specimens at thicknesses of 34 – 69 – 103 – 138 mm. The deviation of the measured values must be below ±0.20 mW/mK of the actual IRMM
value. However, ISO 8301 (5.2.4) allows for a deviation of ±1% which is equal to ±0.3 mW/mK.

The calibration is verified once a year. The calibration drift is monitored every 14 days by using internal reference specimens. If the deviation from the known value differs by more than +/-0.2 mW/mK, action will be taken to explain the reason for the deviation. The procedure for verifying the calibration is described in [11].

Figures 5 and 6 show the results from the routine measurements monitoring the performance of the LaserComp 313 during the years 2004 and 2003 using different reference specimens.

**Figure 5:** 2004 – Result from routine monitoring measurements on LaserComp 313 using different reference specimens.

**Figure 6:** 2003 – Result from routine monitoring measurements on LaserComp 313 using different reference specimens.
A survey of the calibration status is shown in Figure 7. In February 2000 the NIST level was still used. In December 2001 the calibration file was changed to correspond to the current EU level. There has been no need to change the calibration file since December 2001.

![RL LC313 overview of calibration](image)

**Figure 7:** Survey of control of calibration level. Each point represents the average of four measurements done at four different thicknesses. The calibration file has not been changed since December 2001 when the calibration specimens were changed to IRMM specimens.

5.10. Uncertainty due to imperfect contact conditions with rigid samples

As the documentation of the apparatus is mainly based on MW this subject has not been treated. If relevant, Table 2 in EN1946-3 should be consulted.

5.11. Uncertainty due to variation of thermal conductivity with temperature

As measurements are only made at one fixed temperature (10°C) for testing this subject has not been considered.

5.12. Maximum probable uncertainty

The uncertainty budget is specified in annex A, taking the following uncertainties into account:

- Uncertainty related to components in the apparatus: uncertainty in measuring thickness (m), temperature difference (K) and heat flow (W/m²).

- Uncertainty related to test conditions not being ideal: edge heat loss, deviation from flatness of heating and cooling plates.
• Imported uncertainty related to the calibration specimen.

The uncertainty budget when measuring max/min. thicknesses and the typical range of thermal conductivity when testing MW shows the expanded uncertainty \( U(\lambda) \) to be within \( \pm 1.7\% \) at a 95% confidence interval. The uncertainty budget is shown in Tables 4 and 5 below, for specimen thicknesses of 0.050 m and 0.150 m. The uncertainty can be reduced to be within \( \pm 1.5\% \) by reducing the uncertainty in the temperature measurement.

### Table 4: Uncertainty budget when measuring a specimen thickness of 0.050 m.

<table>
<thead>
<tr>
<th>Source of uncertainty</th>
<th>Value +/-</th>
<th>Probability</th>
<th>Divisor</th>
<th>( c(i) )</th>
<th>( u(i) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness measurement</td>
<td>0.11</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.05</td>
</tr>
<tr>
<td>HFM calibration</td>
<td>1.01</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.51</td>
</tr>
<tr>
<td>Temperature drop</td>
<td>1.23</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.62</td>
</tr>
<tr>
<td>Reproducibility</td>
<td>0.09</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.05</td>
</tr>
<tr>
<td>Flatness/unevenness of surfaces</td>
<td>0.50</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.25</td>
</tr>
<tr>
<td>Combined uncertainty</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.84</td>
</tr>
<tr>
<td>Expanded uncertainty</td>
<td>k=2</td>
<td></td>
<td></td>
<td></td>
<td>1.68</td>
</tr>
</tbody>
</table>

### Table 5: Uncertainty budget when measuring a specimen thickness of 0.150 m.

<table>
<thead>
<tr>
<th>Source of uncertainty</th>
<th>Value +/-</th>
<th>Probability</th>
<th>Divisor</th>
<th>( c(i) )</th>
<th>( u(i) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness measurement</td>
<td>0.08</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.04</td>
</tr>
<tr>
<td>HFM calibration</td>
<td>0.94</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.47</td>
</tr>
<tr>
<td>Temperature drop</td>
<td>1.23</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.62</td>
</tr>
<tr>
<td>Reproducibility</td>
<td>0.24</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.12</td>
</tr>
<tr>
<td>Combined uncertainty</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.79</td>
</tr>
<tr>
<td>Expanded uncertainty</td>
<td>k=2</td>
<td></td>
<td></td>
<td></td>
<td>1.57</td>
</tr>
</tbody>
</table>

### 6. Equipment performance check

#### 6.1. Flatness

Deviation from flatness has been measured and is reported in [2].

The minimum specimen thickness is related to the flatness tolerance. The flatness tolerance of the LaserComp apparatus has shown to be within the limit of 0.15 mm which allows for measuring specimens with a minimum thickness of 30 mm. The flatness of the measuring area is less than 0.1 mm.
6.2. Temperature measurements - uniformity of heating and cooling surface

The temperature uniformity of both hot and cold surfaces together with the temperature gradient have been investigated and are reported in [8].

Ten calibrated thermocouples were placed on the surface, five on each side of a 150 mm test specimen (\(\rho \sim 121 \text{ kg/m}^3\)), as shown in Figure 8, with one in the centre and one in each corner of the measuring area. The measured temperature gradient is also shown in Figure 8.

\[
\begin{array}{cc}
10 (7) & 12 (4) \\
9 (3) & 6 (5) \\
20.26 & 20.10 \\
20.09 & 20.17 & 20.03
\end{array}
\]

*Figure 8: To the left, placement of thermocouples for control of temperature. To the right, temperature difference measured [K].*

The temperature uniformity measured on LC313 is within 1% of the temperature difference, which is 20°C, i.e. 0.2°C. The difference from uniformity is found to be 0.03°C on the warm surface and 0.20°C on the cold surface. The measurements show that the temperature uniformity of the FOX600 apparatus satisfies ISO 8301 and EN 1946-3. The temperature gradient measured by the apparatus was 20.00°C. The average gradient from the RI measurement was 20.13°C. The total error in measured temperature gradient may not exceed 1% (equals 0.2°C) according to EN1946-3, 4.4.5. The error in the centre temperature measurement for the apparatus is well within this limit.

6.3. Emissivity of apparatus surfaces

The emissivity is measured by LaserComp on their “standard apparatus” and repeated by RI on the LC313 apparatus, using the method described in EN 1946-3, Annex A. The emissivity is measured to be 0.88 and 0.89, respectively. The emissivity thereby fulfills the criterion in EN 1946-3, which demands the emissivity to be at least 0.80. The result of the test is reported in [13].

6.4. Linearity test

The linearity is demonstrated within the thickness range 30 to 170 mm with a high-density Rockwool specimen (310 kg/m\(^3\)).
The deviation in heat flow from passing through the origin is shown in Figure 9. The investigation proves the linearity of the heat flux meters; the linearity test is reported in [12].

![Linearity test - heat flow as function of temperature difference](image)

**Figure 9:** Stone wool – heat flow as function of temperature difference – deviation from passing through origin is given by the equations. The mean temperature is kept constant at 10°C.

<table>
<thead>
<tr>
<th>Temp diff.</th>
<th>Thickness 0.0323 m</th>
<th>Thickness 0.0646 m</th>
<th>Thickness 0.0966 m</th>
<th>Thickness 0.129 m</th>
<th>Thickness 0.161 m</th>
</tr>
</thead>
<tbody>
<tr>
<td>20°C</td>
<td>0.08</td>
<td>0.42</td>
<td>0.12</td>
<td>0.07</td>
<td>0.21</td>
</tr>
<tr>
<td>16°C</td>
<td>0.10</td>
<td>0.52</td>
<td>0.15</td>
<td>0.08</td>
<td>0.27</td>
</tr>
<tr>
<td>13°C</td>
<td>0.12</td>
<td>0.64</td>
<td>0.19</td>
<td>0.10</td>
<td>0.33</td>
</tr>
<tr>
<td>10°C</td>
<td>0.16</td>
<td>0.84</td>
<td>0.25</td>
<td>0.13</td>
<td>0.43</td>
</tr>
</tbody>
</table>

Table 6: Non-linearity. Error in heat flow (%) from not passing through origin.

As the linearity is found to be acceptable when using a temperature gradient between 20°C and 10°C, it may be acceptable to measure \( \lambda_{10} \) by using a temperature gradient lower than 20°C if the climate chamber is not working properly. However, in this case the measuring accuracy will decrease, and the correct number of measuring blocks to get a reliable result needs to be verified.

No explanation has been found for the error being largest at a thickness of 0.0646 m.
6.5. **Proven performance check**

The required performance test (proficiency test) has been performed according to ISO 8301 (2.5.5). Two different types of test specimens 50 mm EPS and 100 mm MW (2 of each type) have been tested at 11 European reference laboratories. PQL has been able to perform testing on these specimens before and after the Round Robin test program.

The type of instruments participating in the Round Robin test are guarded hot plates except for the PQL.

All the listed laboratories, including the PQL, have satisfactory Z-scores below 2. No systematic offset values for individual laboratories have been found.

The LaserComp HFM apparatus used by PQL is ranked 7 and 6, respectively compared to the other 11 laboratories using guarded hot plate apparatus.

The $\lambda_{10}$ values are measured at mean temperature settings $10^\circ$C $\pm 0.3^\circ$C. The true value (EU level) for each type of specimen is fixed by a measurement in a reference guarded hot plate apparatus. For the 50 mm EPS identified as E 01-01 A and B, and the 100 mm MW identified as M B01-01A and B the standard deviation defined by the inter-laboratory study is 0.20 mW/mK and 0.23 mW/mK respectively.

The test result has been evaluated according to ISO/ICE Guide 43-1 "Proficiency testing by inter-laboratory comparisons" and is shown in Table 6.

The statistical calculations in ISO/ICE Guide 43-1 (A.3.1.1) are based on Z-score factors. These scores are calculated for each measurement. The factor is the difference between the measured value and the true value divided by the defined standard deviation for the respective specimen. This means that every laboratory should have a Z-score below 2 (numeric) for having gained a satisfactory result. A Z-score above 3 is unsatisfactory. The % deviation is the deviation in relation to the true value.
Table 7: Results from inter-laboratory comparison. RI-F represents the specimen measured on LaserComp 313 before the specimen was sent for circulation and RI-E is the specimen measured after the circulation on LaserComp 313.

### EPS Reference specimens E01-01A/B

<table>
<thead>
<tr>
<th></th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>E</th>
<th>G</th>
<th>H</th>
<th>I</th>
<th>J</th>
<th>K</th>
<th>L</th>
<th>M</th>
<th>RI-F</th>
<th>RI-E</th>
</tr>
</thead>
<tbody>
<tr>
<td>% deviation</td>
<td>0.37</td>
<td>0.37</td>
<td>-0.38</td>
<td>-0.82</td>
<td>-0.47</td>
<td>0.22</td>
<td>0.82</td>
<td>-0.56</td>
<td>0.82</td>
<td>-0.08</td>
<td>0.97</td>
<td>-0.57</td>
<td>0.36</td>
</tr>
<tr>
<td>Z-score</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
</tr>
</tbody>
</table>

### MW Reference specimens M01-01A/B

<table>
<thead>
<tr>
<th></th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>E</th>
<th>G</th>
<th>H</th>
<th>I</th>
<th>J</th>
<th>K</th>
<th>L</th>
<th>M</th>
<th>RI-F</th>
<th>RI-E</th>
</tr>
</thead>
<tbody>
<tr>
<td>% deviation</td>
<td>0.19</td>
<td>1.29</td>
<td>-1.23</td>
<td>-1.23</td>
<td>0.06</td>
<td>-0.22</td>
<td>0.34</td>
<td>-0.29</td>
<td>-0.92</td>
<td>-0.29</td>
<td>0.50</td>
<td>0.38</td>
<td>-0.09</td>
</tr>
<tr>
<td>Z-score</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
</tr>
</tbody>
</table>

6.6. Repeatability and reproducibility

According to ISO 8301 (1.5.3.1) the expected repeatability with a specimen that remains in the apparatus must be better than 1%.

The repeatability as well as the reproducibility for the LaserComp 313 apparatus with specimens maintained within the apparatus as well as with specimens removed and mounted again after long-term intervals are within 0.25%, Tables 7 and 8.

Table 8: Control specimen (stone wool) maintained within the apparatus (repeatability).

<table>
<thead>
<tr>
<th></th>
<th>mm</th>
<th>1 mW/mK</th>
<th>2 mW/mK</th>
<th>3 mW/mK</th>
<th>4 mW/mK</th>
<th>5 mW/mK</th>
<th>6 mW/mK</th>
<th>mean mW/mK</th>
<th>Max dev. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>K-7</td>
<td>101</td>
<td>35.02</td>
<td>34.98</td>
<td>35.01</td>
<td>35.02</td>
<td>35.01</td>
<td>35.06</td>
<td>35.01</td>
<td>0.14</td>
</tr>
<tr>
<td>K-12</td>
<td>150</td>
<td>33.98</td>
<td>33.90</td>
<td>33.88</td>
<td>33.82</td>
<td>33.9</td>
<td>33.93</td>
<td>33.90</td>
<td>0.24</td>
</tr>
</tbody>
</table>

Table 9: Specimen (stone wool) removed and mounted again after long-term intervals (reproducibility).

<table>
<thead>
<tr>
<th></th>
<th>mm</th>
<th>1 mW/mK</th>
<th>2 mW/mK</th>
<th>3 mW/mK</th>
<th>4 mW/mK</th>
<th>5 mW/mK</th>
<th>6 mW/mK</th>
<th>mean mW/mK</th>
<th>Max dev. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>K-7</td>
<td>101</td>
<td>35.06</td>
<td>35.01</td>
<td>35.04</td>
<td>35.03</td>
<td>35.02</td>
<td>35.04</td>
<td>35.03</td>
<td>0.09</td>
</tr>
<tr>
<td>K-12</td>
<td>150</td>
<td>33.88</td>
<td>33.87</td>
<td>33.97</td>
<td>33.82</td>
<td>33.89</td>
<td>33.88</td>
<td>33.89</td>
<td>0.24</td>
</tr>
</tbody>
</table>
7. References

[1] T.RI 421-1: Test method - determination of thermal conductivity, \( \lambda_{10} \), plate.


[5] RDQ-00-0359: Standard settings when measuring \( \lambda_{10} \) using FOX600 from LaserComp.


7.1. Referred standards


EN 12939, Thermal performance of building materials and products – Determination of thermal resistance by means of guarded hot plate and heat flow meter methods – Thick products of high and medium thermal resistance.

ISO/ICE Guide 43-1 Proficiency testing by inter-laboratory comparisons.

NB SG 19 N 58 November 2002: Specific criteria and requirements on manufacturers testing instruments; HFM apparatus.
Annex A: Uncertainty budget RI LaserComp313

Purpose

The purpose of this annex is to estimate the uncertainty in measuring the thermal conductivity $\lambda_{10}$ using the FOX 600 from LaserComp. The uncertainty is determined according to the standard “The guide to the expression of uncertainty in measurement” (GUM).

Conclusion

The uncertainty budget when measuring max/min. thicknesses and the typical range of thermal conductivity when testing MW shows the expanded uncertainty $U(\lambda)$ to be within $\pm 1.7\%$ equal to a 95% confidence interval. The uncertainty can be reduced by reducing the uncertainty in the temperature measurement.

Investigation

Thickness measurement

1. The thickness measurement is controlled using calibrated precision blocks and a digital slide gauge. The value represents the average difference between the apparatus measurement and the external measurement taken at 15 points at the heated area at three different thicknesses (data reported in PEL-05-1119).

   The uncertainty from the surfaces not being perfectly flat has been taken into account using the value given in EN1946-3 as the measurement performed does not represent this uncertainty explicitly. This uncertainty is included in the “equipment related uncertainties” in Table A.4 and A.5.

2. Accuracy of calibration of digital slide gauge 0.01mm.

3. Accuracy of calibration of precision blocks (NPL-data: 1.3E-4 mm) is assumed to be neglected.

Table A.1: Uncertainty due to thickness measurement

<table>
<thead>
<tr>
<th>Source of uncertainty</th>
<th>Value +/-</th>
<th>Probability</th>
<th>Divisor</th>
<th>$\sigma(i)$</th>
<th>50 mm</th>
<th>100 mm</th>
<th>150 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Deviation in thickness measurement/ mm</td>
<td>0.05/0.01/0.12</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.05</td>
<td>0.005</td>
<td>0.04</td>
</tr>
<tr>
<td>2. Calibration of digital slide gauge/mm</td>
<td>0.01</td>
<td>N (k=1)</td>
<td>1</td>
<td>1</td>
<td>0.02</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>Combined uncertainty</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.05</td>
<td>0.01</td>
<td>0.04</td>
</tr>
<tr>
<td>Expanded uncertainty</td>
<td></td>
<td></td>
<td></td>
<td>$k=2$</td>
<td>0.11</td>
<td>0.02</td>
<td>0.08</td>
</tr>
</tbody>
</table>
**Heat flow meter**

The heat flow meters are calibrated using a stack of three IRMM reference specimens making a combined thickness of 100 mm. The linearity of the calibration is checked by stacking reference specimens in thickness 35, 70 and 140 mm. As the calibration is performed at a thickness of 100 mm checks were made to show that possible uncertainties from edge heat loss were negligible and hence do not need to be included in the uncertainty budget.

1. The uncertainty of the reference specimens is +/- 0.28 mW/mK according to the certificate resulting in an uncertainty of 0.28/30.47*100% = 0.92% at a 95% confidence interval.

2. Linearity of heat flow - the maximum error in heat flow due to non-linearity was found to be 0.42% at a thickness of 0.06 m when using $\Delta T = 20^\circ$C (RDQ-04-3161rev).

<table>
<thead>
<tr>
<th>Table A.2: Uncertainty due to heat flow meter calibration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source of uncertainty</td>
</tr>
<tr>
<td>------------------------</td>
</tr>
<tr>
<td>Distribution</td>
</tr>
<tr>
<td>1. Calibration with reference material %</td>
</tr>
<tr>
<td>2. Non-linearity %</td>
</tr>
<tr>
<td>Combined uncertainty</td>
</tr>
<tr>
<td>Expanded uncertainty</td>
</tr>
</tbody>
</table>

**Temperature difference**

The measurement of the temperature difference has been checked using a set of calibrated thermocouples as described in EEQ-04-2668rev.

1. Average deviation in measured temperature difference including the standard deviation from the five measuring points is (0.13+0.09)/20 *100% = 1.1%

2. Uncertainty in the external calibration of the thermocouples is +/- 0.1°C which equals 0.1/20 *100% = 0.5%

3. The actual mean temperature being 9.5°C instead of the set value of 10.0°C will cause an uncertainty in thermal conductivity of (36.53-36.62)/36.62*100% = 0.25%
Table A.3: Uncertainty due to measurement of temperature difference

<table>
<thead>
<tr>
<th>Source of uncertainty</th>
<th>Value +/-</th>
<th>Probability</th>
<th>Divisor</th>
<th>c(i)</th>
<th>u(i)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Distribution</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1. Deviation in temperature difference %</td>
<td>1.1</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.55</td>
</tr>
<tr>
<td>2. Calibration of thermo couples %</td>
<td>0.5</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.25</td>
</tr>
<tr>
<td>3. Deviation in mean temperature from 10°C, %</td>
<td>0.25</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.13</td>
</tr>
<tr>
<td>Combined uncertainty</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Expanded uncertainty</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Miscellaneous factors**

1. Air gaps due to deviation in flatness of specimen/surfaces. The flatness tolerance is most important when measuring thin specimens and is below 0.5 % at 95% confidence level when the conditions in table 1 in EN 1946-3 are fulfilled.

2. Environmental conditions. These have been found to have a negligible effect. Provided they remain stable they do not affect the equilibrium conditions.

**Equipment related uncertainties**

The uncertainty based on a 50 mm and a 150 mm thick specimen with a 20°C temperature drop is shown in the Tables A.4 and A.5 below. Any variation in conditions should be taken into account when calculating uncertainty for a given test. When evaluating the uncertainty for a specific test result any uncertainty related to the actual specimen must be included.

Table A.4: Uncertainty budget when measuring a specimen thickness of 0.050 m.

<table>
<thead>
<tr>
<th>Source of uncertainty</th>
<th>Value +/-</th>
<th>Probability</th>
<th>Divisor</th>
<th>c(i)</th>
<th>u(i)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Distribution</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1. Thickness measurement</td>
<td>0.11</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.05</td>
</tr>
<tr>
<td>2. HFM calibration</td>
<td>1.01</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.51</td>
</tr>
<tr>
<td>3. Temperature drop</td>
<td>1.23</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.62</td>
</tr>
<tr>
<td>4. Reproducibility</td>
<td>0.09</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.05</td>
</tr>
<tr>
<td>5. Flatness/unevenness of surfaces</td>
<td>0.50</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.25</td>
</tr>
<tr>
<td>Combined uncertainty</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Expanded uncertainty</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Source of uncertainty</th>
<th>Value +/-</th>
<th>Probability</th>
<th>Divisor</th>
<th>c(i)</th>
<th>u(i)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Distribution</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1. Thickness measurement</td>
<td>0.11</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.05</td>
</tr>
<tr>
<td>2. HFM calibration</td>
<td>1.01</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.51</td>
</tr>
<tr>
<td>3. Temperature drop</td>
<td>1.23</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.62</td>
</tr>
<tr>
<td>4. Reproducibility</td>
<td>0.09</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.05</td>
</tr>
<tr>
<td>5. Flatness/unevenness of surfaces</td>
<td>0.50</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.25</td>
</tr>
<tr>
<td>Combined uncertainty</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Expanded uncertainty</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table A.5: Uncertainty budget when measuring a specimen thickness of 0.150 m.

<table>
<thead>
<tr>
<th>Source of uncertainty</th>
<th>Value +/-</th>
<th>Probability</th>
<th>Divisor</th>
<th>c(i)</th>
<th>u(i)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Thickness measurement</td>
<td>0.08</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.04</td>
</tr>
<tr>
<td>2. HFM calibration</td>
<td>0.94</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.47</td>
</tr>
<tr>
<td>3. Temperature drop</td>
<td>1.23</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.62</td>
</tr>
<tr>
<td>4. Reproducibility</td>
<td>0.24</td>
<td>N</td>
<td>2</td>
<td>1</td>
<td>0.12</td>
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<tr>
<td>Combined uncertainty</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.79</td>
</tr>
<tr>
<td><strong>Expanded uncertainty</strong></td>
<td></td>
<td></td>
<td></td>
<td>k=2</td>
<td>1.57</td>
</tr>
</tbody>
</table>