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### Introduction

The objective of manufacturers of vacuum-insulation panels (VIPs) is to achieve the best possible insulation effect in the least possible installation space. To illustrate the insulating properties of VIPs, a thickness comparison is often carried out with conventional glass wool insulation, polystyrene particle foam (EPS), extruded polystyrene foam (XPS) and/or polyurethane foams; the differences in thickness are significant.

There are numerous other ways, beyond their application as a construction material, of employing VIPs as space-saving, highly efficient means of thermal insulation. Whether in the field of cold chain management, aerospace, medical technology, household appliances, etc.: Whenever it's important to have the lowest possible thermal conductivity within a small amount of space, VIPs are the product of choice. Quality assurance of the thermal conductivity – the most important parameter of VIPs – is therefore of particular importance. For most insulating materials being used as construction materials today, there are relevant product standards available (e.g. DIN EN 13162 to DIN EN 13171), as well as standards for conformity assessment (DIN EN 13172) specifying quality assurance guidelines for insulating materials. The thermal conductivity is determined, for example, in accordance with ISO 8301 with a stationary method by means of a guarded hot plate instrument. This is a method that determines the thermal conductivity of insulating materials with an accuracy of ±3% after calibration on an internationally recognized thermal conductivity reference material (NIST SRM 1450D or IRMM-440). The purpose of the calibration is to ensure that the heat-flow sensors of the instrument deliver precise results for the measuring range that is relevant for the samples.

However – per ISO 8301, paragraph 2.4 – the calibration materials should possess thermal transport properties similar to those of the sample to be tested. In comparing the thermal resistance of a one-inch-thick NIST 1450D standard reference material at 20°C (0.8 m<sup>2</sup>K/W) to the thermal resistance of a vacuum-insulating panel of the same thickness, it can be seen that the thermal resistance of the VIP is eight times (!) higher than that of the reference material. These can no longer be considered to be "similar thermal transport properties".

Heat-flow sensors – even those of very high quality – are to a certain extent non-linear in their measuring range, which is why a calibration is carried out for the heat flow range to be measured.

A legitimate question therefore is:

How can the thermal conductivity of VIPs be determined if the properties of the available reference materials and those of the products to be measured are so different?



### A Question of the Heat Flow

One possibility would be to measure a VIP by means of absolute thermal conductivity methods (e.g. a guarded hot-plate method), determine its thermal conductivity and then calibrate with exactly that sample. It would then have to be ensured, however, that this internal reference material remains stable over a very long time and that the internal pressure of the VIP does not change, since ultimately, the stability of the calibration – according to European standards for insulating materials – must be checked daily and it must be documented that the calibration remains within a tolerance band of  $\pm 1\%$ .

We therefore should not only take the thermal conductivity and thermal resistance into account, but also start comparing the measurement conditions with each other. Wouldn't it be more relevant, in terms of practice, to establish similarity of the measurement conditions and not similarity of the materials? This brings us to the approach which leads the <u>VIP manufacture</u>r to the desired results:

In our first approximation, let us look at the heat flow occurring in a VIP with a thickness of 20 mm at the time of the measurement with a mean test temperature of 14°C and a temperature gradient of 20 K. The consideration is based on the following equation:

$$q_{VIP} = \lambda \cdot \frac{\Delta T}{d}$$

with

$$\label{eq:lambda} \begin{split} \lambda &= \text{set value of the thermal conductivity in W/m·K} \\ \Delta T &= \text{temperature gradient during the measurement in K} \\ d &= \text{thickness of the sample in m} \end{split}$$

This results in:

$$q_{VIP} = 0.0038 \frac{W}{m \cdot K} \cdot \frac{20 K}{0.02 m} = 3.8 W/m^2$$

At the time of the measurement under the above-mentioned conditions, the heat flow in the sample amounts to  $3.8 \text{ W/m}^2$ .

Let us now compare the heat flow which prevails in the referene material NIST 1450D under the above-mentioned conditions:

$$q_{1450D} = 0.0317 \frac{W}{m \cdot K} \cdot \frac{20 \, K}{0.0254 \, m} = 24.96 \, W/m^2$$

The heat flow during calibration – provided calibration is carried out under the same measurement conditions – is significantly higher, at ~25  $W/m^2$ .

In order to achieve an optimum result, one should take care that the heat flow for which the apparatus is calibrated is close to that of the material to be measured.

Determining the Conditions for an Alternative Calibration

How can the heat flow during calibration now be adjusted to the heat flow which is to be measured later?

Chaning the thickness of a reference material is only possible to a limited extent since the materials are only available in one thickness. One possibility would to be stack the reference samples for calibration, in order to increase the thermal resistance and thickness, respectively. Then one runs the risk, however, of creating undefined contact resistances between the samples in the stack, which again results in increased measurement uncertainty.

To avoid this, the temperature gardient can be adjusted: We solve the above-mentioned equation for the gradient and, for a heat flow of  $3.8 \text{ W/m}^2$ , obtain:

$$\Delta T_{1450D} = \frac{0.0254 \ m \cdot 3.8 \ W/m^2}{0.0317 \ W/(m \cdot K)} = 3.04 \ K$$

According to this calculation, a temperature gradient of 3 K yields the same heat flow as for a VIP with a thickness of 20 mm and a gradietn of 20 K at a mean temperature of 14°C. Hence, calibration of the guarded heat-flow apparatus should be carried out with a gradient of 3 K. ISO 8301 recommends, however, not selecting a gradient lower than 5 K during the measurement.

This means that it is not only the calibration that needs to be adjusted, but also the method of measurement for quality assurance.



Higher Temperature Gradient in Quality Assurance Measurement

We therefore increase the gradient to 30 K:

$$q_{VIP} = 0.0038 \frac{W}{m \cdot K} \cdot \frac{30 K}{0.02 m} = 5.7 W/m^2$$

and recalculate the calibration routine:

$$\Delta T_{1450D} = \frac{0.0254 \ m \cdot 5.7 \ W/m^2}{0.0317 \ W/(m \cdot K)} = 4.56 \ K$$

We obtain a quotient of 4.56 K and are thus already significantly closer to the minimally recommended temperature gradient for a guarded heat-flow meter system.

Verification of the Theory with a Practical Example

In order to substantiate the theory of adjusting the calibration to the actual heat flow, we look at a series of measurements on different VIPs.

First, figure 1 presents the results from a screening test on different thicknesses with the following parameters:

- 1. Mean measurement temperature: 14°C
- 2. Temperature gradient: 20 K
- 3. Defined pressure: 17 kPa
- 4. Calibration: calibration with standard reference material 1450D, gradient: 20 K



Results of a series of measurements on VIPs with increasing thickness: The thicker the sample, the higher the scatter of the results. The measurement series was carried out with a calibration of a standard reference material as the basis.

#### Thermal Conductivity vs. thickness



This results in the following temperature gradients for

the alternative calibration, calculated according to the

The minimum recommended gradient of 5 K was

intentionally undershot in order to achieve the best possible accuracy and to explore the method's abilities

above-mentioned equations:

1.6K 2.4K

3. 3 K

and limits.

# How Can the Thermal Conductivity of Vacuum-Insulation Panels Best Be Determined, Considering That There is No Reference Material Available for Such Low Thermal Conductivities?

It is shown in figure 2 how far apart the heat flows are from the measured output signals of the heat-flow meter during the measurement series and calibration. Recorded is the heat flow (in W/m·K) above the output signal of the heat-flow meter (in  $\mu$ V).

In order to determine the effectiveness of the method in detail, three classes of current heat flows were determined for individual thicknesses, and the instrument was re-calibrated with these.

These three heat-flow classes are:

- 1. 7.50 W/m<sup>2</sup>
- 2. 5.00 W/m<sup>2</sup>
- 3. 3.75 W/m<sup>2</sup>



#### Comparison of the heat flux and output-signal while calibration and first measurement series

2 In the lower left corner, the graph shows the current heat flows during the measurement series on VIP and – top right – the current heat flow of the calibration; the calibration factor is on the secondary axis (right)



After calibration of the instrument with an NIST 1450D standard reference material with gradients of 6 K, 4 K

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and 3 K, the comparison of the current heat flows of the measurement series and calibration looks like this:



The graph demonstrates how well the current heat flows of the three determined heat-flow calibrations fit with the current heat flows of the measurement series.



#### Comparison of the heat flux and output-signal while calibration and first measurement series

4 For comparison: Measurement series 1 with calibration of the standard reference material and measurement series 2 with changed gradients and adjusted calibration; calibration factor on secondary axis (right)



Adjustment of the measurement parameters and adapted 4. Calibration: calibration yields the situation shown in figure 5. The parameters for the measurement were:

Underlying calibrations as appropriate for the current heat flow in the three classes mentioned above

- 1. Mean measurement temperature: 14°C
- 2. Defined pressure: 17 kPa

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3. Temperature gradient: for 10-mm samples: 20 K for 20-mm samples: 30 K for 25-mm samples: 40 K for 35-mm samples: 40 K for 45-mm samples: 40 K



### Thermal Conductivity and Density vs. Thickness

The graph shows that the results of the measured thermal conductivity are very stable over the entire thickness range. Deviations depend on the density (and in certain cases on the vacuum value, which it was not possible to determine); on the secondary axis (right) is the density of the individual samples.



### Conclusion

The thermal conductivity of vacuum-insulating panels (VIPs) can be reliably determined in quality assurance with means of a guarded heat-flow meter in accordance with ISO 8301 (or ASTM C518). The method is cost-efficient in comparison with other options, and the instrument is very easy to operate. By means of a heat-flow dependent calibration, a guarded heat-flow meter can be very easily adapted for materials with differing thermal transport properties while the tests still remain 100% traceable to internationally recognized reference materials (even if these exhibit other thermal transport properties).

This method can – to a certain extent – also be applied to particularly thick or thin samples or to materials with higher thermal conductivity.

With an HFM 446-series guarded heat-flow meter by NETZSCH, it is possible to set user methods for various materials which are based on different calibrations. This allows for a wide variety of applications fields to be covered – or for a method to be adjusted so as to be a very close fit for a certain material.

The measurements described were carried out with an HFM 446 *Lambda Medium* (figure 6). This model is capable of going below the temperature gradient of 5 K which is prescribed as the minimum in the norms.



6 NETZSCH-HFM 446 Lambda Medium

### The Author

Alexander Frenzl has been employed in the Development Department at NETZSCH Analyzing & Testing since 2005. In 2008, he became Head of the Mechanical Development Department and, as such, has been involved in the development of all NETZSCH instruments. Since 2014, Alexander Frenzl has been the Business Segment Manager for Glass, Ceramics and Building Materials and serves as an interface between our Development, Sales and Marketing Departments. One of his focal points is industrial quality assurance for insulating materials and associated applications, especially with respect to new and more efficient technologies.

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