

Absolute Thermal Conductivity Measurements – 7 Things That Make for a Good GHP

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Insulating materials are becoming increasingly important in a variety of applications including the insulation of buildings. Better insulation lowers energy consumption and thus the heating costs for any individual household or industrial company. This also reduces CO₂ emissions – an essential contribution toward keeping global warming in check. The temperature-dependent thermal conductivity, λ , is the key parameter since this material property determines the energy flow through the insulation. By means of the NETZSCH GHP 456 *Titan*[®] Guarded Hot Plate apparatus (see figure 1), the entire thermal resistance of large, relatively thick samples can be easily determined, yielding a representative value of the materials' thermal conductivity.



1 NETZSCH GHP 456 *Titan*[®] in open position. A sample or two identical samples (not shown) are located between the hot plate (1) and guard ring (2) and the lower (3) and upper cold plates (4), respectively. Additionally shown are the three-part surrounding furnace (5), insulation (6), feed-throughs (7), hoisting device (8) and gas connection (9).



1. Absolute Method

The big advantage of the GHP method is that it is an absolute method; i.e., no calibration or correction is required at all. The thermal conductivity values result, in the stationary state, simply from the entire heat output measured, Q, the average sample thickness, d, the measurement area, A, and the set and measured mean temperature gradient, ΔT , along the sample or the two samples, as the case may be (the factor 2 results for two samples):

$$\lambda = \frac{Q \cdot d}{2A \cdot \Delta T}$$

2. Wide Temperature Range

Insulating materials can be applied in an extremely wide temperature range, e.g., as cryo-insulation or insulation of high-temperature furnaces. This is the reason why the NETZSCH GHP 456 *Titan*[®] is available in two versions: For the range from -160°C to 250°C and for the range from -160°C to 600°C.

3. Vacuum

The vacuum-tight design of the NETZSCH GHP 456 *Titan*[®] is a prerequisite for defined atmospheres at the samples' site: normal conditions, dry air or inert, oxygen-free purge gas can therefore be applied. Moreover, it is also possible to measure in a vacuum under pressures of down to 10⁻⁴ mbar. All these possibilities are particularly interesting for porous or fibrous insulations since in these cases, the thermal conductivity of the atmosphere in the free sample volume represents a significant portion of the sample's total effective thermal conductivity.

4. Conformity to Standards

The setup and usage of a GHP apparatus are described in international standards such as ISO 8302 or ASTM C 177; for the high-temperature range, the technical specification DIN CNT/TS 15548-1 exists. The setup, dimensions and temperature sensors of the NETZSCH GHP 456 *Titan*[®] are based on these standards. Adherence to the accuracy levels specified by the standards for the thermal conductivity values is, of course, decisive.

5. Ease-of-Use

In line with the simple measuring principle, operation of the NETZSCH GHP 456 *Titan*[®] is also very easy: The apparatus is opened and closed by means of an electronic hoisting device; in between, the operator inserts the sample(s) from the front. Measurement and the generation of a complete measurement report is all handled by the software.

6. Robustness

The NETZSCH GHP 456 *Titan*[®] features robust mechanics and temperature stability – prerequisites for good reproducibility of the measurements. Maintenance requirements are relatively low.

7. Measurement Accuracy

Does a GHP deliver correct thermal conductivity values? Are these values within the tolerances required by the standards? These important questions can only be answered by comparing measured data with trusted literature values [1]. In the field of insulating materials, certified reference values are available; e.g., for the materials NIST SRM 1450D (NIST = National Institute of Standards, USA) in the temperature range from 7°C to 67°C and IRMM 440 (IRMM = Institute of Reference Materials and Measurement, Belgium) in the temperature range from -170°C to 50°C. The thermal conductivities of both materials can be measured with high accuracy by means of the NETZSCH GHP 456 *Titan*®. This is demonstrated for IRMM 440 over a very wide temperature range (see figure 2).

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2 Thermal conductivity of IRMM 440, measured with the NETZSCH GHP 456 Titan® in comparison with the values certified by IRMM (solid line). The dotted lines represent the extended uncertainty budget of the IRMM values (±5% below -10°C, ±1% above -10°C), while the error bars reflect the combined standard measurement uncertainties.



3 Thermal condcutivity of an expanded glass granulate measured by means of the NETZSCH GHP 456 *Titan*[®] in comparison with the published VDI/keymark values (solid line). The dotted lines represent the standard uncertainty of the VDI/keymark values (±3%) while the error bars reflect the combined standard measurement uncertainties.

Above 67°C, i.e., in the entire high-temperature range, there are unfortunately no appropriate certified materials. However, insulating materials with sufficiently accurate published thermal conductivity values do exist: Figure 3 shows a comparison between the well-known VDI/ keymark values for an expanded glass granulate (Liaver GmbH & Co. KG) and the measured values obtained by means of the NETZSCH GHP 456 *Titan*[®]. The agreement in the range from 50°C to 500°C is better than 3%.

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4 Thermal conductivity of SilCal1100, measured by means of the NETZSCH GHP 456 *Titan*[®] in comparison with the published round-robin values (solid line). The dotted line represent the standard uncertainties of the round-robin values (±3% increasing up to ±7%) while the error bars reflect the combined standard measurement uncertainties.

Porous calcium silicate SilCal1100 (CALSITHERM Silikatbaustoffe GmbH) had also already been investigated in detail and round-robin results had been published. Of course, this material was also measured by means of the NETZSCH GHP 456 *Titan*[®] (see figure 4): The agreement with the round-robin values is approx. 1-2% at 100°C and approx. 5% at 600°C.

Along with the accuracy, also the combined uncertainty of each individual GHP measurement is an issue that generally depends on the measurement conditions (mean sample temperature, temperature gradient used) and the sample's properties (thermal conductivity, thickness). As a result, optimization of the measurement parameters allows for minimization of the uncertainty and also for an increase in the accuracy to a certain extent.

With the examples of IRMM 440, expanded glass granulate and SilCal1100, it was demonstrated that the NETZSCH GHP 456 *Titan*[®] fulfills the accuracy requirements of $\pm 2\%$ at room temperature and $\pm 5\%$ across the entire temperature range, as stipulated by standard ISO 8302.

The GHP 456 *Titan*[®] is thus a high-performance instrument for the absolute measurement of thermal conductivity. Additionally, NETZSCH offers the certified standard materials NIST SRM 1450D and IRMM 440 as well as SilCal1100 (incl. works certificate).

Literature

[1] A. Schindler, G. Neumann, D. Stobitzer und S. Vidi, Accuracy of a guarded hot plate (GHP) in the temperature range between -160 °C and 700 °C, *High Temperatures* – *High Pressures*, Vol. 45, 2016, pp. 81-96

The Authors

Dr. Alexander Schindler has worked in the fields of experimental physics, thermal analysis and thermophysical properties for over 18 years. At NETZSCH, he has been employed in the Applications Laboratory as well as in the Hardware and Software Development. He is a known expert in thermal characterization methods and their applications.

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 Development, Sales and Marketing. One of his focal points is industrial quality assurance for insulating materials and associated applications, especially with respect to new and more efficient technologies.