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Accuracy of a guarded hot plate (GHP) in the temperature range between −160°C and 700°C

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Presented in this work are measurements of the thermal conductivity of insulation materials in the temperature range between −160°C and 700°C using the GHP (guarded hot plate) technique. The samples investigated are the reference materials IRMM-440 (IRMM = Institute of Reference Materials and Measurements [1]) and NIST SRM 1450D (NIST = National Institute of Standards & Technology [2]) certified in the limited temperature interval between about −10°C and 70°C, calcium silicate type SilCal1100 [3] and expanded glass granulate [4]. All samples cover a range of temperature-dependent thermal conductivity values between 0.013 W/(m·K) and 0.16 W/(m·K). The experimentally observed accuracy of about ±1% at room temperature and ±5% at minimum and maximum temperatures, respectively, is in agreement with combined standard uncertainty values (coverage factor $k = 1$) for the type [5] GHP and measurement conditions applied. The results on SilCal1100 and expanded glass granulate – both candidates for a standard material for insulating materials at high temperatures – are compared with previous results from recent interlaboratory comparisons [6,7].

Keywords: Thermal conductivity, guarded hot plate, GHP, insulation materials

1 INTRODUCTION

Insulation materials, which have in most cases a fibrous, cellular or granular microstructure, are of great importance regarding lowering of the global energy consumption and thus of the CO_2 emission by means of, e.g., better

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insulation of our buildings [8]. The temperature range in which insulation materials are used is extremely wide since applications vary from cryogenic insulations [9] to high-temperature insulations of furnaces [10]. Knowledge of the temperature-dependent thermal conductivity is a key parameter because it determines the heat flow across the insulation. The guarded hot plate (GHP) technique for measuring the temperature-dependent thermal conductivity of insulations is of particular interest since it is an absolute method without any calibration standard required [11–13]. Further advantages of this stationary technique are comparatively high accuracy and the fact that the thermal conductivity of relatively large specimen is measured across the entire specimen thickness leading to representative results.

This work deals mainly with the accuracy of a commercially available GHP [5] in the temperature range between −160°C and 700°C comparing experimental results with theoretical considerations. A proof of the accuracy of a GHP is actually hindered by the lack of suitable reference materials in the entire temperature range [14]. Two reference materials for insulating materials, IRMM-440 [1] and SRM 1450D supplied by NIST [2], were measured in the temperature range between −160°C and 60°C. IRMM-440 is, however, certified just in the limited temperature interval between −10°C and 50°C and SRM 1450D just between 7°C and 67°C. The microporous calcium silicate material SilCal1100 [3] investigated in this work at temperatures of up to 700°C is a promising candidate for a high-temperature reference for insulation materials [6]. It fulfills at least all pre-conditions: high-temperature stability, low thermal conductivity, isotropy and homogeneity on a macroscopic scale. Stiffness and mechanical stability allow for easy specimen preparation, and it is inexpensive as well as commercially available. The existence of a considerable amount of published thermal conductivity measurements is of course another advantage. Further tests were carried out in the temperature range between 50°C and 500°C on a particular commercially available expanded glass granulate [4] the thermal conductivity of which was already investigated in detail, too [7]. This insulating microporous granular material is practically incompressible, homogeneous, isotrop and has furthermore the advantage, that flexible specimen geometries can be realized. Specimen preparation, however, is time-consuming.

2 EXPERIMENTAL DETAILS

In a GHP working in the double-sided mode, hot plate and guard ring, which is minimizing lateral heat losses, are sandwiched between two specimen of the same material and approximately the same thickness. Located above the top specimen and below the lower specimen are two cold plates for the establishment of a precise temperature gradient ΔT across the specimen. Thermal conductivity values are calculated from the heating power of the hot plate, its metering area, the temperature gradient and the thickness of the specimen applying the steady-state heat transfer equation (see next section).

The design of the type [5] GHP shown in figure 1 is based on ASTM and ISO standards [11–13] and it must furthermore fulfill the demands of a commercially available instrument with regard to serviceability and robustness. Labels 6, 7, 8 and 9 refer to insulation, feedthroughs for temperature sensors, hoisting device and purge gas inlet, respectively. On the backside and thus not visible are further feedthroughs for temperature sensors, for heaters and for cooling lines for the cold plates as well as a flange for evacuating the instrument. The dimensions of the plates and thus the maximum specimen dimensions are $(300 \times 300)\,mm^2$, the metering area is $(150 \times 150)\,mm^2$, and the maximum thickness of a specimen is 100 mm. It has to be noted that DIN EN 12667 [13] recommends a maximum specimen thickness of 45mm for the design of the type [5] GHP. The entire plate stack is surrounded by a sectional furnace (5) minimizing radial heat losses and wire heat losses; the upper front part of the sectional furnace is not displayed. A vacuum-tight housing allows for measurements under defined gas atmospheres or under vacuum down to the range of 10^{-4} mbar.

There are two versions of the type [5] GHP both particularly suitable for insulation materials with thermal conductivities in the range between about $1{\cdot}10^{-3}\,W/(m{\cdot}K)$ and $1\,W/(m{\cdot}K)$. The low-temperature version, which is for

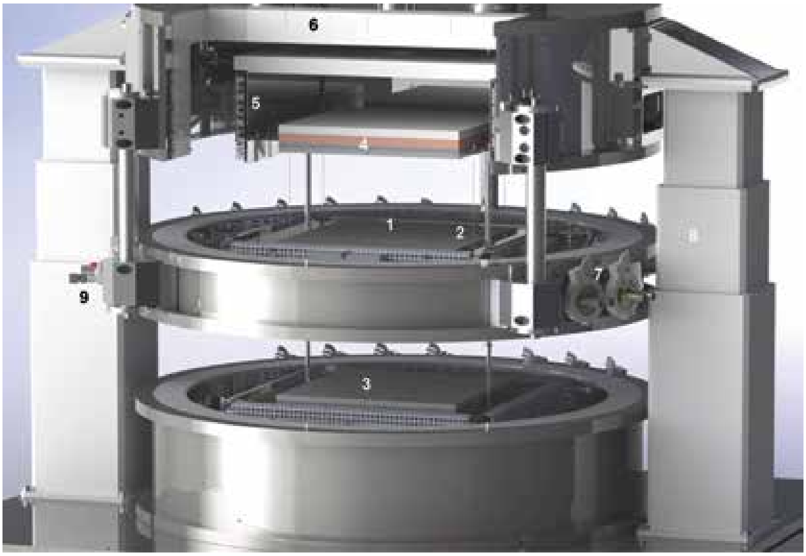

FIGURE 1
Type [5] guarded hot plate (GHP) apparatus in opened state for two specimen (not displayed) sandwiched between hot plate (1) and guard ring (2) in the center and the lower (3) and upper cold plate (4) from below and above.

the range between -160°C and 250°C mean specimen temperature, has plates made of aluminum alloy while the high-temperature version for the range between -160°C and typically 600°C maximum specimen temperature has plates made of tungsten alloy. For this work, three high-temperature versions of type [5] GHP were used where two were equipped with 29 sheathed type N thermocouple temperature sensors, respectively, and one instrument with 29 sheathed type Pt100 temperature sensors. The noise of both kinds of temperature sensors is in the lower mK range, the temperature-dependent accuracy discussed below is also similar for both kinds. The type N thermocouples are significantly more robust at higher temperatures than the Pt100 sensors used. Due to the smaller temperature range, the accuracy of the type Pt100 temperature sensors built in the low-temperature version of the type [5] GHP is slightly higher. This leads to a slightly higher accuracy regarding thermal conductivity measurements compared to the high-temperature version focused in this work.

In the temperature range between -160°C and 60°C, the cold plates of the GHPs as well as the sectional furnace were cooled using LN_2 while air cooling was applied in the temperature range between 100°C and 300°C. Above 300°C, measurements were done without any active cooling.

Certified reference materials for insulating materials, IRMM-440 [1] and SRM 1450D supplied by NIST [2], were measured in the temperature range between -160°C and 60°C using different ΔT values of 10, 20 and 30 K. Measurements were carried out in a GHP [5] equipped with thermocouples. The resin-bonded glass fiber boards IRMM-440 with dimensions of $(300 \times 300 \times 34.4)\,mm^3$ have densities in the range between 65 kg/m^3 and 75 kg/m^3. Type SRM 1450D specimen with dimensions of $(300 \times 300 \times 24.5)\,mm^3$ and densities in the range between 114 kg/m^3 and 124 kg/m^3 are resin-bonded fibrous glass boards, too. Rigid spacers made of calcium silicate were placed in the corners in order to ensure a defined thickness of the compressible type IRMM-440 and SRM 1450D specimen.

GHP measurements on SilCal1100 [3] were carried out in the temperature range between 0°C and 700°C using different ΔT values of 10, 20 and 30 K, too. Two type [5] GHPs both equipped with thermocouple temperature sensors as well as a further GHP [5] equipped with Pt100 temperature sensors were applied. The SilCal1100 specimen used for this work were supplied as $(300 \times 300 \times 30)\,mm^3$ plates with a density of $(250 \pm 15)\,kg/m^3$. The SilCal1100 specimen were furthermore treated at 850°C for 12 hours by the supplier.

The expanded glass granulate [4,7] was measured in the temperature range between 50°C and 500°C at $\Delta T = 30$ K using a GHP [5] equipped with thermocouple temperature sensors. This insulating microporous granular material with grain sizes of 1-2 mm and a loose bulk density of $(220 \pm 20)\,kg/m^3$ is temperature resistant at least up to 550°C. Two calcium silicate frames were applied for lateral confinement; their height of 52 mm defined the thicknesses of the two specimen measured.

After insertion and before the measurements started, all specimen were conditioned in the GHP instruments by heating them up under vacuum to 60–70°C in case of IRMM440 and SRM 1450D, and up to about 100–120°C in case of SilCal1100 and expanded glass granulate. After about one hour, the GHPs were backfilled and purged again with dry nitrogen gas but still kept at these temperatures for about another four hours. This procedure should minimize the influence of moisture on the measured thermal conductivity values. It was experienced that a significantly longer duration of the conditioning procedure of 48 hours did not reveal significantly different results. Second or third heating experiments furthermore confirmed the first heating results also indicating sufficient conditioning of the specimen.

3 THEORETICAL CONSIDERATIONS

Since a GHP is measuring under steady-state conditions, the thermal conductivity λ is according to Fourier's law for one dimension

$$\lambda = \frac{Q \cdot d}{2A \cdot \Delta T} \tag{1}$$

where Q is the entire heating power flowing from the hot plate symmetrically through both samples, A is the effective metering area of the hot plate which is delimited by the center line of the gap between hot plate and guard ring, d is the mean thickness of the two specimen used in the double-sided mode and $\Delta T = T_{hp} - T_{cp}$ the mean temperature difference between the hot plate and the cold plates and thus the gradient across the specimen. Equation (1) is valid for a GHP with symmetrical arrangement of two identical specimens with the same thickness and the same temperature gradient between the hot and cold side, respectively. The thermal conductivity λ should furthermore have a linear temperature dependence in the temperature range between T_{hp} and T_{cp} which is limiting the maximum value of ΔT that should be applied. The mean specimen temperature T_{mean} is defined as $(T_{hp} + T_{cp})/2$. The guard ring temperature T_{gr} is nominally equal to T_{hp} in order to eliminate heat flows in lateral direction.

Detailed analysis of the uncertainties in particular GHP instruments was carried out earlier [15,16]. According to ASTM C177, the relative uncertainty $u(\lambda)/\lambda$ should be expressed in the following way [11]:

$$\left(\frac{u(\lambda)}{\lambda}\right)^2 = \left(\frac{u(d)}{d}\right)^2 + \left(\frac{u(A)}{A}\right)^2 + \left(\frac{u(\Delta T)}{\Delta T}\right)^2 + \left(\frac{u(Q)}{Q}\right)^2. \tag{2}$$

Equation (2) which is in accordance with GUM (guide to the expression of uncertainty in measurement [17] is the combined standard uncertainty (coverage factor $k = 1$). The uncertainty of the thickness $u(d)$ is limited by the accuracy of the thickness determination, which is in the range of ±0.15 mm for the specimen of this work, and the temperature dependence of the thickness described by the expansion coefficient of the calcium silicate samples and spacers (see above) which is of the order of $5{\cdot}10^{-6}K^{-1}$. The temperature-dependent uncertainty of *u*(*A*) is mainly due to the thermal expansion of the plate material where we assume an expansion coefficient of $5{\cdot}10^{-6}K^{-1}$ for the tungsten alloy used for the plates of the type [5] GHP. Estimated values of *u*(*d*)/*d* and *u*(*A*)/*A* can be seen from table 1.

The uncertainty *u*(Δ*T*) significantly depends on the temperature-dependent uncertainty of the temperature sensors where it is useful to distinguish between systematical and statistical uncertainty as illustrated in figure 2. The systematic uncertainty of the temperature calibrated sensors is about ±0.5 K in the whole temperature range which is about the uncertainty of T_{mean}. Since $T_{hp} = \frac{1}{n_{hp}} \cdot \sum_{i=0}^{n_{hp}} T_{hp\_i}$ and $T_{cp1,2} = \frac{1}{n_{cp}} \cdot \sum_{i=0}^{n_{cp}} T_{cp1,2\_i}$ one obtains

$$u(\Delta T) = u(T_{hp} - T_{cp}) = \sqrt{\left(\frac{1}{n_{hp}} + \frac{1}{2n_{cp}}\right) \cdot u^2(T_i) + u^2(\Delta T)_{syst}} \qquad (3)$$

where n_{hp} and n_{cp} are the number of temperature sensors in the hot plate and in each of the two cold plates ($n_{hp} = 9$ and $n_{cp} = 5$ in case of the type [5] GHP) and $u(T_i)$ is the statistical uncertainty of each individual temperature sensor which means the mean deviation among the sensors. The factor 2 in equation (3) is due to the fact that there are two cold plates and the mean cold plate temperature is $T_{cp} = 1/2 \cdot \left(T_{cp1} + T_{cp2}\right)$. The uncertainty $u(\Delta T)_{syst}$ is the difference of the systematic uncertainties of the temperature sensors between T_{hp} and T_{cp} as indicated in figure 2 where we assume $u(\Delta T)_{syst} = \pm 0.1$ K as constant. Using the values $u(T_i) = \pm 0.4\,K$ at −160°C, $u(T_i) = \pm 50$ mK at room temperature and $u(T_i) = \pm 0.7$ K at 700°C, where these values are experimentally observed for the type [5] GHP, one obtains $u(\Delta T) = \pm 0.21$ K at −160°C, $u(\Delta T) = \pm 0.10$ K at room temperature and $u(\Delta T) = \pm 0.34$ K at 700°C. Relative uncertainties $u(\Delta T)/\Delta T$ for $\Delta T = 20$ K can be seen from table 1.

The relative uncertainty *u*(*Q*)/*Q* consists of two contributions: One constant part $u(Q)_{el}/Q$ being due to the uncertainty of the electrical power measurement can be estimated to about ±0.2%. The second part $u(Q)_{gap}/Q$ depending again on the measurement conditions is due to parasitic heat flows mainly due to so called gap imbalance between the hot plate and guard ring leading to an undesired heat flow in lateral direction and thus to an uncertainty $u(Q)_{gap}$ [11,12]. The gap imbalance $u(T_{hp} - T_{gr})$ can be calculated to

$$u(T_{hp} - T_{gr}) = \sqrt{\frac{1}{n_{hp}} + \frac{1}{n_{gr}}} \cdot u(T_i) \qquad (4)$$

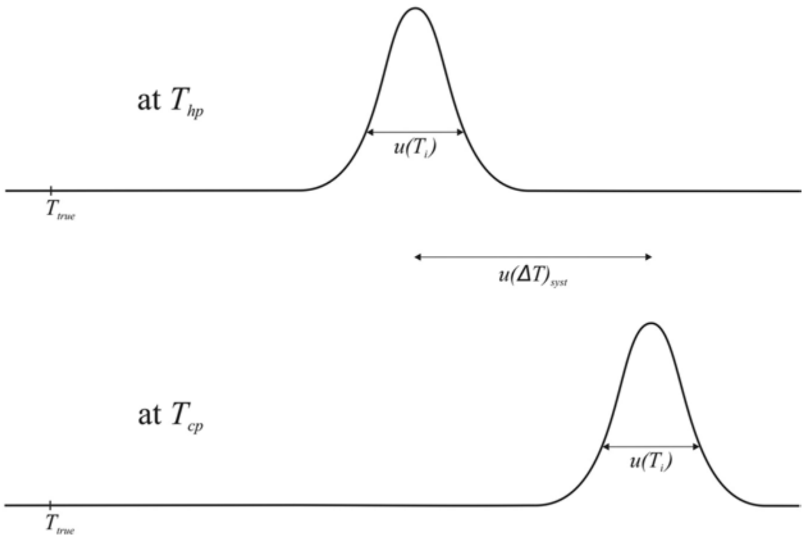

FIGURE 2
Uncertainty of an ensemble of GHP temperature sensors at T_{hp} and T_{cp} (illustration). The distance between the peak temperatures and T_{true} is the systematic uncertainty, respectively, which differs at T_{hp} and T_{cp} by a value of $u(\Delta T)_{syst}$. The value $u(T_i)$ is the statistical uncertainty of an individual sensor reflected by the mean deviation among the sensors (see text).

TABLE 1
Uncertainty values of a GHP [5] at three different mean temperatures and λ-values where $d = 30\,\text{mm}$, $A = 0.0225\,\text{m}^2$, $u(Q)_{el}/Q = \pm 0.2\%$ and $\Delta T = 20\,\text{K}$ was assumed for all cases. The uncertainties $u(T_i)$ of the individual temperature sensors are experimentally observed values whereas $u(d)/d$, $u(A)/A$, $u(\Delta T)/\Delta T$, $u(Q)_{gap}/Q$ and the combined standard uncertainty $u(\lambda)/\lambda$ are calculated (coverage factor k = 1, see text.)

	$u(d)/d$	$u(A)/A$	$u(T_i)$	$u(\Delta T)/\Delta T$	$u(Q)_{gap}/Q$	$u(\lambda)/\lambda$
$T_{mean} = -160°\text{C}$ $\lambda = 0.013\,\text{W/(m·K)}$	±0.6%	±0.2%	±0.4 K	±1.1%	±5.0%	±5.1%
$T_{mean} = 25°\text{C}$ $\lambda = 0.03\,\text{W/(m·K)}$	±0.5%	±0.1%	±50 mK	±0.5%	±0.5%	±0.9%
$T_{mean} = 700°\text{C}$ $\lambda = 0.16\,\text{W/(m·K)}$	±0.8%	±0.7%	±0.7 K	±1.7%	±9.2%	±9.4%

where n_{gr} is the number of temperature sensors in the guard ring ($n_{gr} = 8$ in case of the type [5] GHP). In contrast to equation (3), the systematic error of the temperature sensors plays no role for $u(T_{hp} - T_{gr})$ expressed in equation (4), because $T_{hp} \cong T_{gr}$. The relationship between gap imbalance $u(T_{hp} - T_{gr})$ and its consequence $u(Q)_{gap}$ can be expressed as $u(Q)_{gap} = C(T) \cdot u(T_{hp} - T_{gr})$. The temperature-dependent quantity $C(T)$ which depends on the apparatus design but also on the properties of the specimen can be estimated theoretically [16,18] but also measured directly by means of intentionally applied gap imbalance [16,19]. We measured for NIST1450D at −160°C and 25°C and for SilCal1100 at 700°C values for $C(T)$ of 0.1, 0.2 and 1.3 W/K leading to values of $u(Q)_{gap}$ of approximately 0.019 W, 0.005 W and 0.442 W at those temperatures. Mostly due to the increasing uncertainty $u(T_i)$ of the temperature sensors, $u(Q)_{gap}/Q$ is increasing considerably at lowest and highest temperatures (see table 1) clearly exceeding the 0.5% maximum allowed value of ISO 8302 [12]. On the one hand, ISO 8302 might not be written for a temperature range as wide as −160°C…700°C. On the other hand, the impact of gap imbalance could in principle be eliminated by an active correction of the heating power as described in [15]. However, it is unclear if such corrections were in accordance with standards like [11–13]. In this work, no such power corrections or any other corrections are applied.

One can calculate a relative uncertainty $u(\lambda)/\lambda = \pm 5.1\%$ assuming $T_{mean} = -160°C$, $\Delta T = 20\,K$, $d = 30\,mm$, and $\lambda = 0.013\,W/(m\cdot K)$. For $T_{mean} = 25°C$, $\Delta T = 20\,K$, $d = 30\,mm$, and $\lambda = 0.03\,W/(m\cdot K)$, one obtains $u(\lambda)/\lambda = \pm 0.9\%$; for $T_{mean} = 700°C$, $\Delta T = 20\,K$, $d = 30\,mm$, and $\lambda = 0.16\,W/(m\cdot K)$, one obtains $u(\lambda)/\lambda = \pm 9.4\%$. These numbers summarized in table 1 are at 25°C and −160°C in accordance with the demands of, e.g., ISO 8302 [12] of ±2% at room temperature and ±5% at maximum temperature of the apparatus. At 700°C, a higher value of ΔT than 20K has to be used in order to decrease the uncertainty of ±9.4% to a lower value (see below). For example for $\Delta T = 30\,K$ one obtains $u(\lambda)/\lambda = \pm 6.3\%$, and for $\Delta T = 40\,K$ one obtains $u(\lambda)/\lambda = \pm 4.7\%$. As mentioned above, the uncertainties $u(\lambda)/\lambda$ are combined standard uncertainties (coverage factor $k = 1$), respectively.

It is, however, important to emphasize that the actual uncertainty $u(\lambda)$ and also $u(\lambda)/\lambda$ strongly depends on the measurement conditions – in particular on the specimen's thermal conductivity and thickness, the mean specimen temperature and ΔT used. The latter is a key parameter for investigation of the actual uncertainty of λ through experiments [19]. Introducing the decisive uncertainties $u(Q)$ and $u(\Delta T)$ into equation (1) one obtains

$$\lambda + u(\lambda) = \frac{(Q + u(Q)) \cdot d}{2A \cdot (\Delta T + u(\Delta T))} \tag{5}$$

and it can be seen that with increasing ΔT (leading also to an increase of Q), the uncertainties $u(\Delta T)$ and $u(Q)$ get less important and thus $u(\lambda)/\lambda$ will decrease. It is thus recommended to measure λ-values at different values of ΔT and to consider the dependence of λ on ΔT and also the λ-value λ_∞ extrapolated for $\Delta T \to \infty$ or in practice $1/\Delta T \to 0$ as it is shown in [19].

Another, not yet mentioned uncertainty of the measured thermal conductivity λ is due to the contact resistances between the specimen and the hot and cold plates of the GHP instrument depending mostly on the specimen's surfaces and hardness [20]. Contact resistances have a smaller impact on the measured λ the higher the thermal resistance of the specimen is; this can be achieved by a larger specimen thickness d and a smaller λ of the sample. In principle, contact resistances could be determined by measuring λ at different thicknesses of the same sample material [11]. Since the heating power Q and thus $u(Q)/Q$ depends according to equation (5) on the specimen thickness d, we suggest to measure λ for each specimen thickness also for several values of ΔT and consider λ_∞ as a function of d or $1/d$. This procedure is beyond the scope of this work and the λ-values presented can be regarded as effective λ-values including also the impact of contact resistances.

In summary, it is - apart from measurements of λ at different values of ΔT, generally recommended to work at sufficiently large specimen thicknesses in order to minimize the relative uncertainty $u(d)/d$ and also the impact of contact resistances. But at the same time, the heating power Q and temperature gradient ΔT should be sufficiently high in order to minimize $u(Q)/Q$ and $u(\Delta T)/\Delta T$. This leads finally to a compromise where the magnitude of the values of ΔT is adjusted depending on the magnitudes of λ and d of the specimen.

4 EXPERIMENTAL RESULTS AND DISCUSSION

The thermal conductivity λ of the certified reference material IRMM-440 [1] was measured in the temperature range between -160°C and 50°C. The inset of figure 3 shows that the λ-values measured between -10°C and 50°C using different values of ΔT and the values extrapolated for $\Delta T \to \infty$ in the way described above agree within about $\pm 1\%$ with the reference values. This result matches with the combined standard uncertainty discussed above. The λ-values measured at -50°C, -100°C and -160°C decrease approximately linear with decreasing temperature as it is also observed for the dataset measured by DFT (Dipartimento di Fisica Tecnica, Padova, Italy) reported in [1]. At these temperatures, the results of this work are within $\pm 5\%$ in agreement with the dataset measured by DFT (see figure 3).

The corresponding results for the standard reference material SRM 1450D [2] depicted in figure 4 are qualitatively very similar to those obtained for IRMM-440 discussed above. There is again good agreement with the certified values within about $\pm 1\%$. It must be noted that SRM 1450D has by about 6% higher λ-values compared to IRMM-440.

The thermal conductivity λ of the insulating material calcium silicate type SilCal1100 described above was also measured using a GHP [5] equipped with thermocouple temperature sensors at different values of ΔT of 10, 20 and 30 K (see figure 5). Exactly the same pair of specimen plates was also measured at the certified research institute FIW (Forschungsinstitut für Wärmeschutz e.V. München [21]) using one of their GHP instruments with a

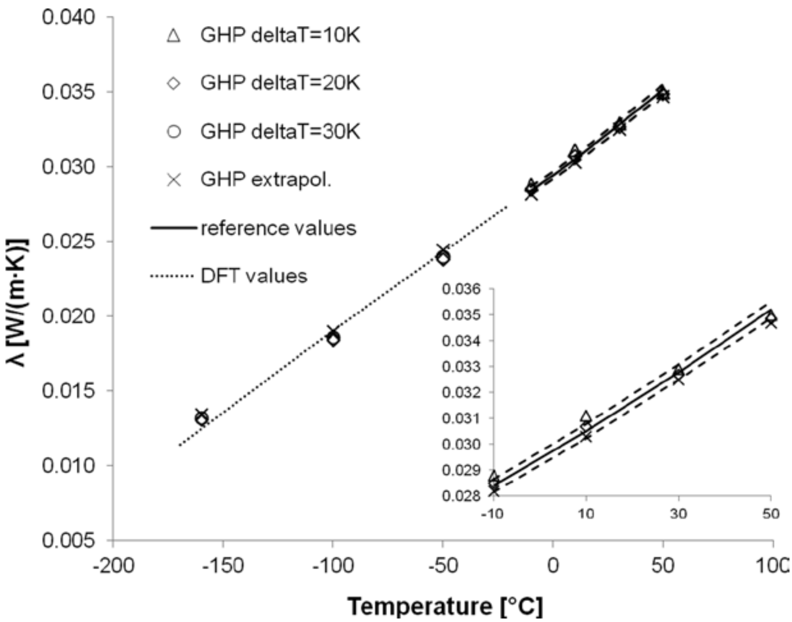

FIGURE 3
Temperature-dependent thermal conductivity λ of IRMM-440 measured with GHP at different values of ΔT of 10, 20 and 30 K and the resulting λ-values extrapolated to $\Delta T = \infty$ in comparison with the certified reference values (full line) with an uncertainty of ±0.9% (dashed lines). The dotted line represents a supplementary dataset ("DFT") from the certification report [1].

nominal uncertainty of ±3%. Their GHP unit was designed and is nominally measuring according to ASTM C177 [11]. At 50°C, our thermal conductivity results measured at different values of ΔT and also the λ-value measured by FIW are each in the range between 0.082 and 0.083 W/(m·K) indicating the high accuracy of about 1% of both instruments under these measurement conditions. With increasing temperature of up to 600°C, the difference between our results and the λ-values measured by FIW increases up to about 4%. The difference between the λ-value measured at 600°C with $\Delta T = 30$ K and the value extrapolated to $\Delta T = \infty$ is about 5% which is also in accordance with estimations above for the type [5] GHP at high temperatures.
The thermal conductivity λ of SilCal1100 was recently investigated in detail in terms of an interlaboratory comparison with seven participating laboratories [6]. By means of various stationary and instationary methods using various specimen geometries, 14 datasets were measured in the temperature range between room temperature and about 800°C. Those results represented by the solid line in figure 5 are in the range of 0.085W/(m·K) at room temperature and 0.155 W/(m·K) at 700°C where the relative uncertainty increases from ±3.5% to ±6.5%. It can be seen that the thermal conductivity values

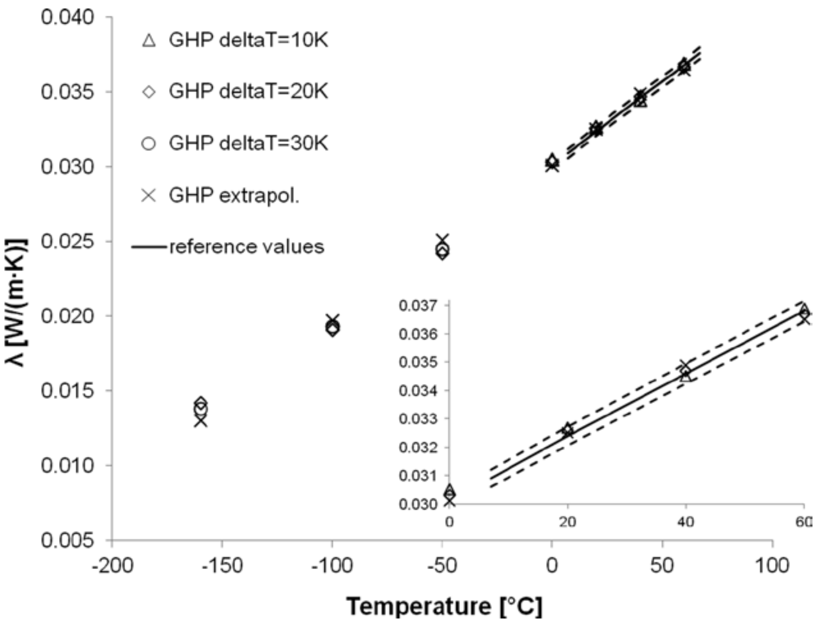

FIGURE 4
Temperature-dependent thermal conductivity λ of NIST SRM1450D measured with the GHP at different values of ΔT of 10, 20 and 30 K and the resulting λ-values extrapolated to $\Delta T = \infty$ in comparison with the certified NIST reference values (full line) with an uncertainty of $\pm 1\%$ (dashed lines).

measured on the SilCal1100 specimen of this work are clearly lower than the mean λ-values from the interlaboratory comparison. Systematically different results between stationary methods like GHP and instationary methods both contributing to the interlaboratory comparison were, however, not observed [6]. Therefore, we measured three further pairs of SilCal1100 specimen from the same batch under the same conditions using the same type of instrument (a further type [5] GHP equipped with thermocouple temperature sensors) in order to check the reproducibility. A summary of these results is shown in figure 6: These specimen sets revealed λ-values with parallel shifts of about $\pm 3\%$ relative to each other. It is important to note that the repeatability of a measurement of the same pair of specimen in the same instrument is better than 1%. The same pair of specimen C+D did even – within 1–2% – reveal the same λ-values when measured in another instrument of the same type [5] (see figure 6). This indicates on the one hand that different GHP instruments of this type measure comparable λ-values when using exactly the same specimen. On the other hand, the shift of our results shown in figure 5 compared to the interlaboratory results is thus mostly due to the differences between each individual specimen used. These differences might be due to sample

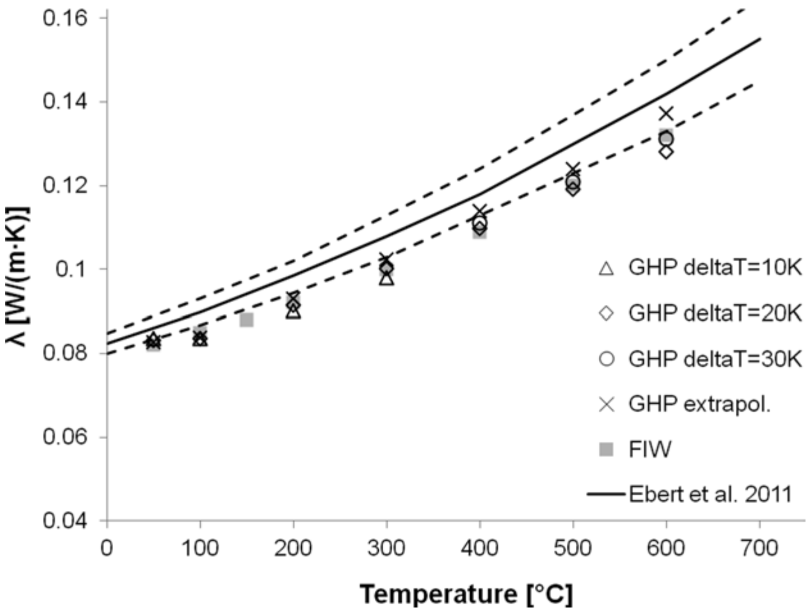

FIGURE 5
Temperature-dependent thermal conductivity λ of SilCal1100 measured with a GHP at different values of ΔT of 10, 20 and 30 K and the resulting λ-values extrapolated to $\Delta T = \infty$ in comparison with the results measured at FIW using exactly the same pair of specimen. The solid line represents the mean λ-values from an interlaboratory comparison [6] of type SilCal1100 samples, the dashed lines the relative uncertainty of these results increasing from ±3.5% at room temperature to ±6.5% at 700°C.

inhomogeneities but also due to slightly different contact resistances between the specimen and plates of the GHP. Our mean thermal conductivity data on all type SilCal1100 specimen measured are also systematically smaller than the mean values of the recent interlaboratory comparison by about 3% (see figure 6). This observation could be explained by the fact that the type SilCal1100 specimen used for the interlaboratory comparison were not from the same batch as our specimen. Our results on various specimen from the same batch do furthermore suggest that the uncertainty of the results of the interlaboratory comparison is also significantly due to the differences between each individual specimen since all the participating laboratories used different specimen prepared from the same material [6].

Further conclusions regarding the accuracy of thermal conductivity testing instruments – especially the type of GHP discussed in this work – can be drawn from the test results obtained for a particular expanded glass granulate described in detail above [4,7]. Figure 7 depicts the thermal conductivity values in the temperature range between 50°C and 500°C in comparison with the VDI/Keymark data for this material. The latter which represent a collection

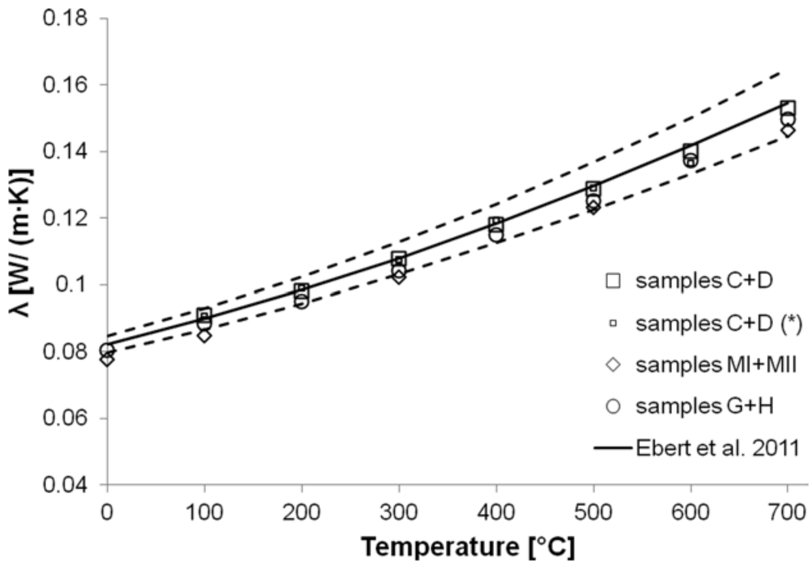

FIGURE 6
Temperature-dependent thermal conductivity λ of three pairs of SilCal1100 specimen from the same batch measured with the same GHP instrument (equipped with thermocouple temperature sensors). An exception is dataset (*) which was measured in another GHP unit of the same type (equipped with type Pt100 temperature sensors). For clarity, just the λ-values extrapolated to $\Delta T = \infty$ are displayed. The solid line represents the mean λ-values from an interlaboratory comparison [6] of type SilCal1100 samples, the dashed lines the relative uncertainty of these results increasing from ±3.5% at room temperature to ±6.5% at 700°C.

of 195 data points measured using various different techniques such as GHP, hot bridge, hot wire etc., and using different specimen geometries have an uncertainty of ±3%. The mean deviation of a recent interlaboratory comparison [7] to which again several different measurement techniques contributed was about ±6%. The GHP data of this work are within about ±3% in agreement with the mean VDI/Keymark data. The combined standard uncertainty (coverage factor $k = 1$, see considerations above) of the thermal conductivity values is represented by the uncertainty bars with a magnitude of about ±1% at 50°C and ±5% at 500°C.

5 CONCLUSION

GHP measurements of the thermal conductivity of the certified reference materials IRMM-440 [1] and SRM 1450D [2] revealed an accuracy of the instrument of about ±1% in the certified temperature interval between −10°C and +60°C and about ±5% in the temperature range between −160°C and −50°C. A comparison between the thermal conductivity results on various

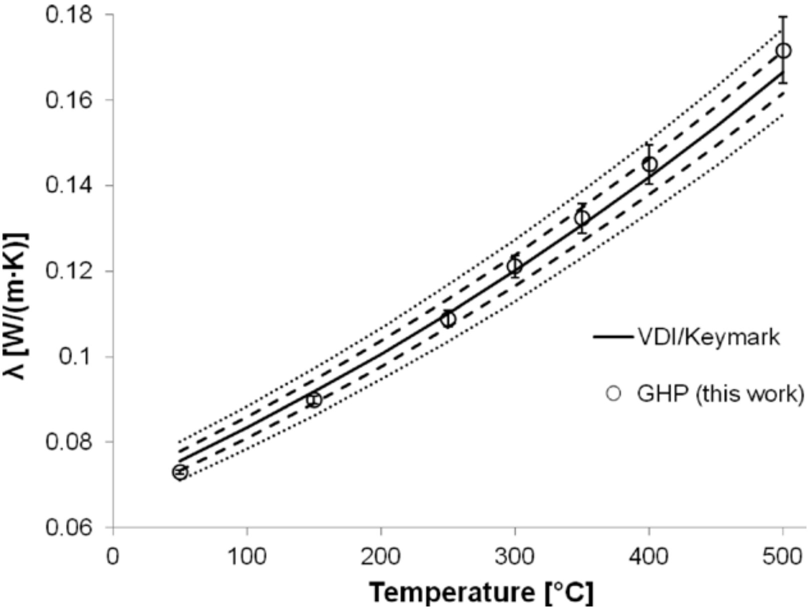

FIGURE 7
Temperature-dependent thermal conductivity λ of expanded glass granulate measured with a GHP at $\Delta T = 30\,K$ in comparison with the mean VDI/Keymark data for this material (full line). The dashed lines represent the uncertainty of the VDI/Keymark data of ±3%, the dotted lines the mean deviation of ±6% of the 14 datasets contributed to a recent interlaboratory comparison [7].

calcium silicate type SilCal1100 [3] specimen, which were measured with three different type [5] GHP instruments, and comparative measurements done at the research institute FIW [21] using another type of GHP, and furthermore the results of a recent interlaboratory comparison [6] showed that the GHP instruments have an accuracy of about ±1% at room temperature increasing up to about ±5% at 700°C. Measurements on expanded glass granulate [4] with well known thermal conductivity [7] revealed an accuracy of the GHP instrument of about ±3% in the temperature range between 50°C and 500°C. It should, however, be emphasized again that the nominal thermal conductivity values of the samples investigated have also an uncertainty which is in the same range as the accuracy values of the GHP instruments discussed above. For an entirely proven accuracy, one may thus multiply the accuracy figures above by a factor of $\sqrt{2}$.

The observed accuracy of the GHP instruments is in agreement with the combined standard uncertainty (coverage factor $k = 1$) discussed. It must be noted, however, that the accuracy of a GHP generally depends on the specimen properties, especially on the actual thermal conductivity and thickness of the specimen but also on the measurement conditions applied. The temperature-dependent accuracy of the plate and guard temperatures as well as the magnitude of the temperature gradient ΔT across the specimen are predominant impact factors. Measurements of the thermal conductivity at different values of ΔT are recommended for an experimental indication of the actual uncertainty. The magnitude of ΔT is typically used for an adjustment of the heating power which should be sufficiently high.

Both SilCal1100 and the expanded glass granulate are candidates for a standard material for insulating materials at high temperatures. In case of SilCal1100, we found as a drawback that different specimen from the same batch revealed significantly different thermal conductivity values: The λ-values measured in the temperature interval between 0°C and 700°C showed parallel shifts in the range of about $\pm 3\%$. The expanded glass granulate has the disadvantage that specimen preparation is time-consuming.

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