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A Guarded Hot-Plate Apparatus for Thermal Conductivity Measurements over the Temperature Range −75 to 200°C

W. Hemminger[1] **and R. Jugel**[1]

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A guarded hot-plate apparatus for small circular samples has been developed for the temperature range from −75 to 200°C. To avoid edge losses, the apparatus is immersed in a liquid whose temperature is a few degrees lower than the mean temperature of the samples. A detailed evaluation procedure with several correction calculations leads to a remaining uncertainty of measurement of 0.5% for measurements on glass samples. This has been confirmed by experiment. Measurements on glass and on insulation material showed that the developed apparatus and the evaluation procedure applied can be used in a relatively wide range of thermal conductivity values (factor 50).

KEY WORDS: glass; hot-plate apparatus; insulation material; thermal conductivity; thermocouple corrections.

1. INTRODUCTION

The thermal conductivity of poorly conducting materials is generally measured under steady-state conditions using a guarded hot-plate apparatus. A new guarded hot-plate apparatus which covers a temperature range within which a great number of such materials are used in practice has been developed on the basis of proven design principles [1]. The new apparatus is designed for performing measurements on small circular samples in an isothermal environment at a temperature equal to that of the cold plate. In order to keep the uncertainty of the results as small as possible and to know the uncertainty, it is necessary to analyze the error sources and to correct for remaining uncertainties.

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[1] Physikalisch-Technische Bundesanstalt, Bundesallee 100, D-3300 Braunschweig, Federal Republic of Germany.

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Such analyses are carried out for measurements on glass; the evaluation procedure is described and the uncertainty of measurements is checked by experiment. The thermal conductivity of the glass samples was also measured (in a smaller temperature range) using a second apparatus to test the conformity of both devices. The usability of the new guarded hot-plate apparatus for an insulating material of very low thermal conductivity was demonstrated by measuring in a nitrogen and helium atmosphere.

2. MEASURING INSTRUMENT

The design of the guarded hot-plate apparatus is shown in Fig. 1. The specimen (A) is arranged between the hot plate (B) (100 mm in diameter) and the cold plate (C). The heat flux, electrically produced in the hot plate, is to flow to the cold plate, if possible without any loss. For this purpose, the hot plate is shielded toward the top by a heated guard plate (D) and toward the circumference by a heated guard ring (E). In order to keep the heat losses from the sample edge toward the outside small, the sample is surrounded by a guard ring (F), in which approximately the same temperature gradient as in the specimen is produced with the aid of the heated guard ring. The specimen and its guard ring, as well as the hot plate and its guard heaters, are separated by gaps 2 mm in width. During the measurement, the temperatures of the guard heaters are matched as closely as possible to the temperature of the hot plate. Remaining deviations are measured and taken into account as corrections. The hot plates and the cold plate consist of nickel-plated copper. The entire apparatus is mounted in a steel casing (G), which is immersed in a temperature-controlled liquid (J). An ethyl alcohol bath is used for the temperature range −75 to 30°C and a silicon oil (100 AP) bath is used from 40 to 200°C. The only breaks in the isothermal surroundings are the ducts (I) for the measurement leads and supply lines and the central duct for the push rod (H), which holds the component parts together by adjustable spring pressure.

3. THERMOCOUPLES

All 10 thermocouples (copper–constantan, 0.2 mm in diameter) were calibrated with the aid of a platinum resistance thermometer. For this purpose, the thermocouples and the thermometer were placed in a copper block, which in turn was placed in temperature-controlled liquid baths. In the temperature range from −75 to 195°C, calibration was carried out at intervals of 30°C. A polynomial fit (third order) was obtained from the mean values of the voltage of all 10 thermocouples at the respective tem-

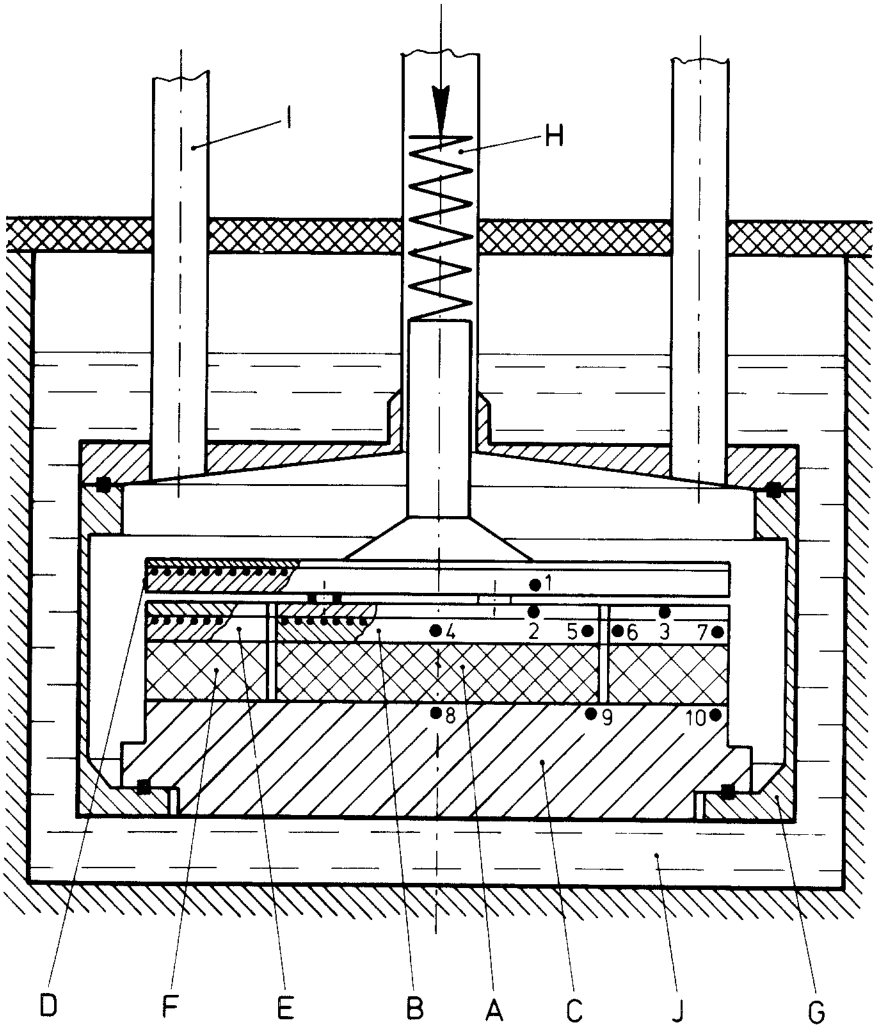

Fig. 1. Guarded hot-plate apparatus for the temperature range −75 to 200°C. A, Specimen; B, hot plate; C, cold plate; D, heated guard plate; E, heated guard ring; F, specimen guard ring; G, casing; H, push rod; I, ducts; J, liquid bath.

perature; its value deviated from the measured values by about 0.015°C at the most.

At the transition of the thermocouple wires from areas at measuring temperature to ambient temperature, additional small thermoelectric powers are generated in the range of this temperature gradient, due to inhomogeneities of the wires. This leads to small differences in the thermoelectric voltages of the individual thermocouples. After the thermocouples have been installed in the apparatus, the transition from the measuring temperature to the ambient temperature never takes place exactly at the same point as during the calibration. The small differences of the thermocouples therefore change a little. Consequently, the correction of these errors can be carried out only after the thermocouples have been mounted and fixed (see Refs. 2 and 3).

4. PREREQUISITES AND ERROR SOURCES

For guarded hot-plate apparatuses, the thermal conductivity is determined according to the following relation:

$$\lambda = P_0 d/(A\,\Delta T_0)$$

where P_0 is the heat flux through the sample, d is the sample thickness, A is the sample cross section, and ΔT_0 is the temperature difference between the sample surfaces.

The above equation has been derived for a steady-state, one-dimensional, linear temperature field with a constant heat-flux density P_0/A. The amount of the local temperature gradient may be replaced by the difference quotient $\Delta T_0/d$ only if these conditions are closely met. The thermal conductivity determined in this way has the character of a mean value for the mean sample temperature (mean temperature). The error due to the usually nonlinear temperature dependence of the thermal conductivity in the range ΔT_0 is reduced to an insignificant amount if ΔT_0 values as small as possible are used. By measuring the same sample of Pyrex glass with a ΔT_0 of 5 and 10 K, it was determined that no measurable error was found for the only easily bent curve $\lambda(\zeta)$ for Pyrex glass (Fig. 2).

Prerequisites for a homogeneous vector field of the heat-flux density are isothermal surfaces of the samples and the avoidance of edge heat losses. The requirement of isothermal sample surfaces is fulfilled as closely as possible by using copper of a high thermal conductivity for the hot and cold plate and by providing thermal contact of the cold plate with the bath liquid over a wide area. In addition, a uniform and good thermal contact between the surface-ground surfaces of the sample and the surface-ground surfaces of the hot and cold plate is ensured. At temperatures above −45°C this is achieved by contacting with silicon oil. Below −45°C, the consistency of the silicon oil changes to such an extent that it is no longer suitable for contacting. Thin gas layers (helium and nitrogen here) are used in this temperature range.

The requirement that edge heat losses from the sample be avoided is fulfilled by means of a guard ring surrounding the sample. In the ideal case,

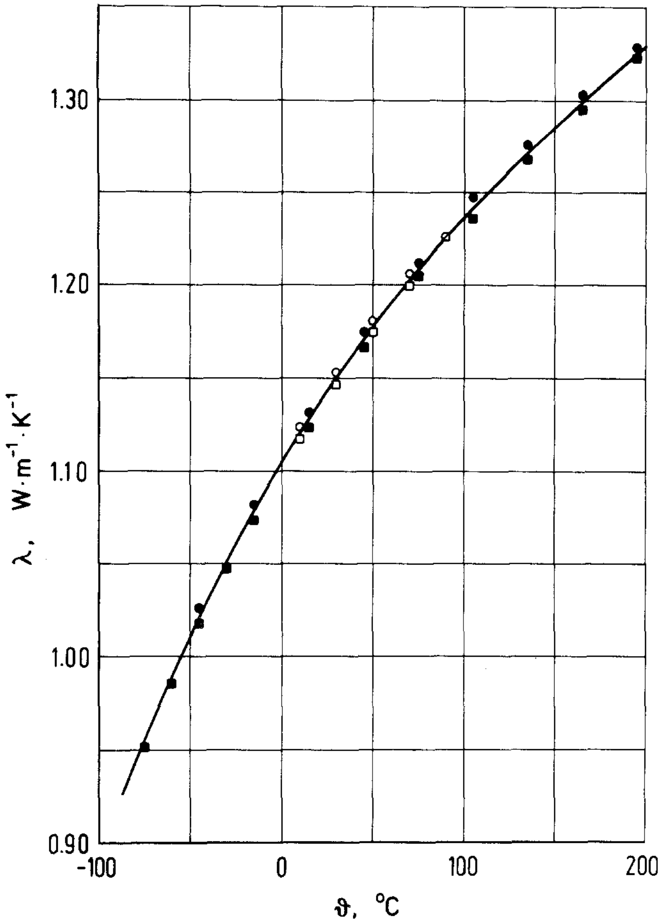

Fig. 2. Thermal conductivity of Pyrex glass, measured values and polynomial fit. (●) Sample 1, apparatus 1; (○) sample 1, apparatus 2; (■) sample 2, apparatus 1; (□) sample 2, apparatus 2.

the lateral surface of the sample and the opposite inner lateral surface of the guard ring have the same temperature profile. Rigorously, this is possible only if the specimen guard ring and the specimen consist of the same material and if they have the same thermal contact resistances. Numerous tests with different guard-ring materials have, however, shown that it is generally sufficient that the material fulfills the following conditions: it must be of a low thermal conductivity and be as snugly fitting as possible so that the temperature differences at the contact points to the heated guard ring and the cold plate remain small compared with the overall temperature differences, even without an additional contact agent being used. Moreover, the temperature coefficient of the thermal conductivity of the guard ring material should be similar to that of the sample material. The measurements on Pyrex glass were carried out with a guard-ring material with $\lambda \approx 0.3\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$. Control measurements with a guard-ring material with a thermal conductivity of $\lambda \approx 0.03\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$ showed very good agreement. As the whole apparatus is encased and completely immersed in the bath, the ambient temperature is equal to the cold-plate temperature and therefore deviates from the mean sample temperature only by the small amount of $\Delta T_0/2$. Edge errors caused by heat transfer from the outer edge of the guard ring to the environment therefore remain negligibly small [4].

5. UNCERTAINTIES AND CORRECTIONS FOR MEASUREMENTS ON PYREX GLASS

5.1. Geometry

The diameter of the glass samples (100 mm) is determined with an uncertainty of 0.05 mm; the thickness of the samples (approx. 20 mm), with an uncertainty of 0.005 mm. Provided that the sample is plane-parallel, cylindrical, and free from edge damage, no uncertainties greater than 0.1 and 0.025% are expected for the determination of the sample cross section A and the thickness d, respectively.

5.2. Temperature Difference ΔT_0

The temperature difference between the sample surfaces ($\Delta T_0 \approx 5$ K) is not measured directly on the test piece but calculated from temperature measurements in the hot plate (T_4, T_5) and the cold plate (T_8, T_9).

With the temperatures in the middle and at the edge of the plates (T_4, T_8 and T_5, T_9), the characteristic mean temperatures $T_4 + T_5/2$ (hot-plate temperature) and $T_8 + T_9/2$ (cold-plate temperature) are calculated. From these, the temperature difference

$$\Delta T = (T_4 + T_5)/2 - (T_8 + T_9)/2$$

results.

By determining the two corrections ΔT_{m} (temperature difference in the metal layer between the sample surface and the temperature measuring point) and ΔT_{c} (temperature difference in the contact layer) from the heat-flux density P_0/A, the layer thicknesses of the metals (copper and nickel), and the contact agent (oil or gas) and its thermal conductivity, the temperature difference between the sample surfaces used for determining the thermal conductivity is found to be $\Delta T_0 = \Delta T - (\Delta T_m + \Delta T_c)$. In detail, the following is determined.

5.2.1. Temperature Difference ΔT

All thermocouple voltages are measured sequentially under steady-state conditions using a potentiometer; they are then measured again in a different order. The mean value from 10 individual measurements serves as the measured value. Typical standard deviations amount to approximately 1 mK. The uncertainties for $(T_4 + T_5)/2$ and $(T_8 + T_9)/2$, therefore, are 1 mK, i.e., 2 mK for ΔT. The measured differences between the temperature T_4 and the temperature T_5 or between T_8 and T_9 amount, on average, to 10 mK. They cover the actual deviations from the isothermal state and individual indication errors of the individual thermocouples. From this, there results an additional uncertainty for $(T_4 + T_5)/2$ and $(T_8 + T_9)/2$ of 5 mK, i.e., 10 mK for ΔT. Thus, the total uncertainty for ΔT is 12 mK. Unknown systematic uncertainties of these thermocouples have not been considered. They are allowed for by a method decribed in Ref. 2, which is applied for the determination of the measurement errors of all temperature differences influencing the measured result. This correction is explained in the section on the calculation of P_0.

5.2.2. Temperature Difference ΔT_m

The distance between the centers of the thermocouple bores and the surface of the hot or cold plate is 3.3 mm in copper (uncertainty, 0.5 mm) and about 0.04 mm in nickel. Using the thermal conductivities of copper and nickel, the correction ΔT_m is calculated as a function of the temperature. For measurements on Pyrex glass the maximum correction was 7 mK, with an uncertainty of 1 mK resulting from the uncertainty of the layer thickness and of 0.5 mK due to the uncertainty of the thermal conductivity values. The overall uncertainty of ΔT_m thus amounts to 1.5 mK.

5.2.3. Temperature Difference ΔT_c

For measurements in the temperature range from −45 to 195°C, silicon oil (DC 200; viscosity, 12,500 $\mathrm{mm}^2 \cdot \mathrm{s}^{-1}$ at 20°C) is used as the contact agent. The average thickness of both contact layers amounts to 15×10^{-6} m, as determined by weighing the oil. The thermal conductivity of the oil is known to within 1 %. Changes in the layer thickness caused by small losses of oil in the course of the measurements lead to estimated uncertainties of 15% in ΔT_c. The total uncertainty (16%) referred to a maximum of 40 mK for ΔT_c amounts to 6 mK.

Below −45°C the silicon oil is replaced with a gas of a high thermal conductivity (helium). The thickness of the gas layer is adjusted by three aluminium spacers (3 mm in diameter; thickness, 8×10^{-6} m). The magnitude of the roughness of the polished surfaces of the metal plates and of the glass sample is of the order of that of the spacer thickness and must therefore be allowed for. For the roughness an amount of 1.25×10^{-6} m per surface (metal and glass) has been estimated. The gas layers between the glass surface and the metal surface sum up to $(8 + 2 \times 1.25) \times 10^{-6}$ m each. The calculation of the correction for both contacting layers must therefore be based on a gas-layer thickness of 21×10^{-6} m. The thermal conductivity of helium was taken from the literature (Ref. 5, Vol. 3); the temperature difference ΔT_c calculated with it amounted to 46 mK at the most. The uncertainty in the determination of the gas-layer thickness is estimated to be 3×10^{-6} m (15%) due to the estimated surface roughness and small deviations from the ideal surface flatness of the plates. With an estimated uncertainty of 5% for the thermal conductivity of helium, the uncertainty for ΔT_c amounts to 20% (9 mK referred to a maximum of 46 mK for ΔT_c).

In order to check the results, additional measurements were carried out using nitrogen, whose conductivity is substantially poorer, as the interface gas. The maximum correction increased to 292 mK. In accordance with the previous estimation, a higher uncertainty of 58 mK results in this case (1.16 % for $\Delta T \approx 5$ K). The maximum deviations of the individual values of 0.3% from the mean value for the gas interfaces, which are represented in Fig. 3 for different media (oil, He, N_2), show that the uncertainty for the gas contacting is, in reality, smaller than has been assumed above.

The uncertainty of ΔT_0 as the sum of the individual uncertainties amounts to 22.5 mK (0.45% for $\Delta T_0 \approx 5$ K). According to the error propagation law, the uncertainty amounts to 15 mK (0.3%) if the individual uncertainties of ΔT, ΔT_m, and ΔT_c are calculated by addition.

5.3. Heat Flux P_0 Through the Sample

Edge losses are kept negligibly low by means of the isothermal surrounding (see Section 4 and Ref. 2). The unbalance losses due to temperature differences between the hot plate and its surrounding parts must be evaluated by experiment, since all theoretical results are based on simplified assumptions (see, e.g., Ref. 8).

The following is valid:

$$P_0 = P + P_X - \sum P_V$$

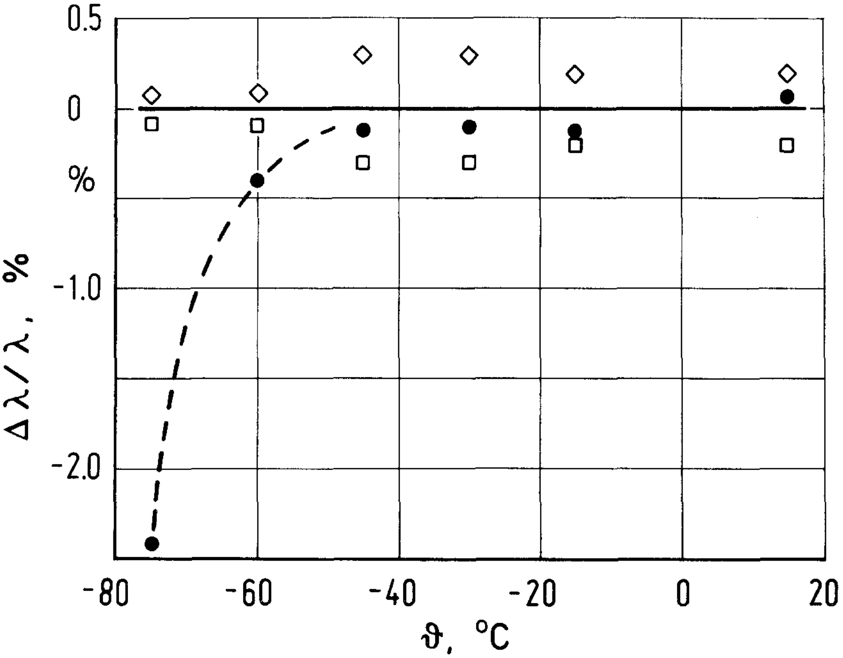

Fig. 3. Differences in thermal conductivity of Pyrex glass measured with various fluid interface layers between the plates (related to the mean value $\bar{\lambda}$ of thermal conductivity measured with helium and nitrogen). (●) Oil; (□) helium; (◇) nitrogen.

where P is the heating power produced in the hot plate; P_X is the correction to allow for the errors in the measurement of the temperature differences ΔT, $\Delta T_1 = T_2 - T_1$, and $\Delta T_2 = (T_2 + T_5)/2 - (T_3 + T_6)/2$; and $\sum P_V$ is the sum of the unbalance losses, in particular, the unbalance loss between the hot plate and the heated guard plate (P_{V1}) + the unbalance loss between the hot plate and the heated guard ring (P_{V2}) + the unbalance loss between the hot plate and the cold plate in the gap between the specimen and the guard ring (P_{V3}).

The heating power P produced in the hot plate is determined with an uncertainty of 0.04% by measuring the voltage and the current.

Determination of the unbalance losses is as follows:

$$P_{V1} = C_1\,\Delta T_1 = C_1(T_2 - T_1)$$

$$P_{V2} = C_2\,\Delta T_2 = C_2((T_2 + T_5)/2 - (T_3 + T_6)/2)$$

$$P_{V3} = (\lambda_{\text{gap}}\,A_{\text{gap}}/2)\,\Delta T/d$$

The proportionality factors C_1 and C_2 are determined by the "mismatch" method [2, 6]; in the following it is explained for C_1 (P_{V3} is calculated [7]).

A test piece of unknown thermal conductivity and a specimen guard ring are installed in the apparatus. The heating power P is adjusted such that the temperature difference on the test piece is approximately 5 K. The temperatures of the heated guard plate and the heated guard ring are adjusted so that the temperature difference ΔT_2 becomes almost zero and P_{V2} can consequently be neglected. P, ΔT, and ΔT_1 are measured in the steady state. For these boundary conditions the "conductance"

$$\Lambda = (P - C_1\,\Delta T_1)/\Delta T = (P - P_{V1})/\Delta T$$

is calculated for the present "sample + gap" arrangement. Then the temperature difference ΔT_1 is varied by about 1 to 2 K (mismatch), with the mismatch power P remaining unchanged, and the new value $\Delta T_1'$ is obtained. Due to mismatching, ΔT changes to $\Delta T'$. In order to avoid errors, the temperatures have to be readjusted so that the same mean temperatures $(T_4 + T_5 + T_8 + T_9)/2$ result for $\Delta T'$ and ΔT, and ΔT_2 once more becomes almost zero. $\Delta T'$ and $\Delta T_1'$ are again measured in the steady state. Now the conductance is

$$\Lambda = (P - C_1\,\Delta T_1')/\Delta T' = (P - P_{V1})/\Delta T'$$

Equating both terms for Λ yields

$$C_1 = P(\Delta T - \Delta T')/(\Delta T\,\Delta T_1' - \Delta T_1\,\Delta T')$$

In practice ΔT_1 is adjusted to approximately 0 or -1 K for the first experiment; for the second experiment $\Delta T_1 = 1$ K is chosen, so that the mismatch becomes approximately 1 or 2 K.

Numerous experiments with different ΔT_1 values (differing in amount and sign) showed a linear dependence of the loss P_{V1} on ΔT_1.

The factor C_2 for calculating P_{V2} is determined analogously to C_1, via a "mismatch" of ΔT_2 and $\Delta T_1 \approx 0$. The factors C_1 and C_2 are temperature dependent. They were determined at -75, 15, and 165°C; intermediate values have been interpolated. Consequently, an overall determination of the losses between the hot plate and the guard heaters via the 2-mm-wide gaps (conduction and radiation) and heat losses by conduction via the wires (heater and thermocouples) and spacers can be made using C_1 and C_2. If helium or nitrogen is used as the interface layer at low temperatures, the apparatus is filled with this gas and the factors C_1 and C_2 have to be newly determined.

The uncertainty of C_1 and C_2 is estimated to be 5%; it does not have any effect, as the corrections P_{V1} and P_{V2} calculated with C_1 and C_2 were maximally 0.1% of P_0.

The unbalance loss P_{V3} is calculated according to Ref. 7. It is assumed that half the heat flux which flows in the 2-mm-wide gap between the specimen and the guard ring to the cold plate originates from the hot plate, and the other half from the heated guard ring. P_{V3} is thus calculated from half the cross-sectional area of the gap ($A_{\mathrm{gap}}/2$), the thermal conductivity λ_{gap} of the filling gas (air, He, or N_2) at the respective mean temperature, the sample thickness (d), and the temperature difference (ΔT). The greatest uncertainty of this correction results from the assumption that half the heat flux flowing in the gap is delivered from the hot plate. It cannot be controlled if this precondition is fulfilled. The uncertainty of the correction P_{V3} is therefore estimated as 20%. In the case of the measurements with Pyrex glass, this correction for air or nitrogen amounted to a maximum of 0.1% of P_0, from which an uncertainty of 0.02% results for P_0 (helium, 0.1% uncertainty for P_0).

6. DETERMINATION OF THE CORRECTION P_X

It has already been mentioned in Section 3 that there are small differences in the thermoelectric power of the individual thermocouples. As a result, the temperature differences ΔT, ΔT_1, and ΔT_2 are affected by small errors. The method of individual correction of each thermocouple described in Ref. 2 is replaced by another method, also described in Ref. 2, for the additive coverage of all individual errors (expressed by the power correction P_X). P_X is determined as follows.

The specimen to be measured is installed in the apparatus together with the appropriate guard ring. The heating power P is adjusted so that a ΔT which is typical for measurements of the thermal conductivity (approx. 5 K) is obtained at the specimen. The temperatures of the heated guard plate and the heated guard ring are adjusted in such a way that the temperature differences ΔT_1 and ΔT_2 are as small as possible. P, ΔT, ΔT_1, and ΔT_2 are measured in the steady state. From this, the losses P_{V1}, P_{V2}, and P_{V3} and thus also $P-\sum P_V$ can be determined as described in Section 5.3.

For further calculation, for the values of this first measurement, the designations P_Z^* and ΔT^* are introduced for $P-\sum P_V$ and ΔT, respectively. In a subsequent measurement with $P=0$ at the same mean temperature, the guard heaters are again adjusted so that ΔT_1 and ΔT_2 become as small as possible. These temperature differences and the new ΔT, called ΔT^{**}, are again measured in the steady state. $P-\sum P_V$, denoted P_Z^{**}, is again determined according to Section 5.3. The values P_Z^{**} determined from the measurement with $P=0$ covers only the corrections $\sum P_V$. When these corrections and the ΔT^{**} measured at the same time are correct, then $P_Z^*/\Delta T^* = P_Z^{**}/\Delta T^{**}$ must be fulfilled.

This cannot be achieved in practice due to small deviations of the individual thermocouples from one another; an overall power correction, P_X, is therefore introduced for the errors of the thermocouples. This correction is calculated as follows:

$$(P_Z^* + P_X)/\Delta T^* = (P_Z^{**} + P_X)/\Delta T^{**}$$

From this,

$$P_X = (P_Z^* \, \Delta T^{**} - P_Z^{**} \Delta T^*)/(\Delta T^* - \Delta T^{**})$$

The errors covered in this way could, of course, also be expressed as a temperature correction ΔT_X for ΔT:

$$(\Delta T^* + \Delta T_X)/P_Z^* = (\Delta T^{**} + \Delta T_X)/P_Z^{**}$$

From this,

$$\Delta T_X = (\Delta T^* P_Z^{**} - \Delta T^{**} P_Z^*)/(P_Z^* - P_Z^{**})$$

In both cases the calculated correction is valid only for the specimen used for determining the correction, with the mean temperature being identical for both measurements. The method previously used to calculate the individual correction for each thermocouple [2] showed a linear temperature dependence for the correction. The correction described was therefore determined for each specimen at the lowest and the highest mean temperature and linearly interpolated for the higher temperatures.

For the two liquid baths used, the point of transition from the measuring to the ambient temperature is not the same for the thermocouples. Moreover, at measuring temperatures below 0°C (the temperature for the reference junctions of the thermocouples), a change of the sign occurs for the temperature dependence of the correction. For these two reasons, the correction P_X had to be determined separately in both baths. In the measurements on Pyrex glass, P_X amounted to a maximum of 0.41 % over the whole temperature range. This maximum correction was calculated from the following measured values.

Experiment 1: $P_Z^* = 2.4705$ W; $\Delta T^* = 5.013$ K.

Experiment 2: $P_Z^{**} = 0.0050$ W; $\Delta T^{**} = -0.011$ K.

With these values, $P_X = -0.0106$ W.

On the basis of the correction determined in this way, $P_0 = P_Z^* + P_X = 2.5599$ W for experiment 1. This value is used to calculate the thermal conductivity.

For measurements on Pyrex glass the uncertainty of P_X was determined by repeat measurements. It amounted to 0.0015 W, i.e., between 0.05 and 0.08%, depending on P_0 (2 to 2.8 W). Consequently, the total uncertainty of P_0 is 0.14% (additive value).

According to the error propagation law, the total uncertainty for the thermal conductivity of Pyrex glass—including the individual uncertainties for P_0 (additive value, 0.14%), d (0.025%), A (0.1%), and ΔT_0 (additive value, 0.45%)—amounts to 0.48%, i.e., approximately 0.5%. Here the uncertainty of ΔT_0 represents the most important contribution. This small overall uncertainty was achieved only because the corrections for the thermal coupling of the specimen to the apparatus could be well established and because P_0 was great compared to the corrections $P_X - \sum P_V$. The measured values of the two Pyrex glass samples showed that the uncertainty of measurement of 0.5% is realistic. The mean systematic difference between the measured values of the two samples amounts to 0.7%. At the different measuring temperatures the individual differences lie between 0.5 and 0.9%, which is plausible for an uncertainty of measurement of 0.5%.

7. MEASUREMENTS ON PYREX GLASS [9]

The samples were manufactured from two different plates. The measured density difference lies within the scope of the uncertainty of measurement (2.222 and 2.224 $\mathrm{g \cdot cm^{-3}}$). Each sample was measured both in the described apparatus and in a second apparatus with a temperature range from 10 to about 100°C. In the temperature range covered by both apparatuses, no systematic difference in the measured values was found (Fig. 2). The thermal conductivities for −30, −60, and −75°C were measured on only one of the two test pieces. For values which were measured several times in one device, a maximum repeatibility scatter of 0.15% resulted. Doubling the heat-flux density in the test piece did not cause any change in the measured values.

The normally used guard ring, made of a material with a thermal conductivity of approximately 0.3 $\mathrm{W \cdot m^{-1} \cdot K^{-1}}$, was replaced by a guard ring with a thermal conductivity of approximately 0.03 $\mathrm{W \cdot m^{-1} \cdot K^{-1}}$. No influence on the results was observed. The described agreement of the results, even after the test conditions had been varied several times, allows the conclusion to be drawn that the unknown systematic deviations left are negligibly small.

Over the temperature range investigated, the temperature dependence of the thermal conductivity (mean values for both apparatuses) of Pyrex glass can be described by a polynomial of the third order (see Fig. 2).

$$\lambda(\vartheta) = 1.104 + 1.65 \times 10^{-3}\,\vartheta - 3.92 \times 10^{-6}\,\vartheta^2 + 6.63 \times 10^{-9}\,\vartheta^3$$

Table I. Thermal Conductivity of Pyrex Glass from the Present Work (PTB) and According to Ref. 10

ζ (°C)	$\lambda(\mathrm{W \cdot m^{-1} \cdot K^{-1}})$ PTB	$\lambda(\mathrm{W \cdot m^{-1} \cdot K^{-1}})$ Ref. 10	$\frac{\lambda(\mathrm{PTB}) - \lambda[10]}{\lambda(\mathrm{PTB})} \times 100$ (%)
−100	(0.893)	0.854	(4.4)
−50	1.011	0.984	2.7
0	1.104	1.093	1.0
50	1.177	1.185	−0.7
100	1.236	1.273	−3.0

(temperature range, −75 to 195°C; temperature ϑ in °C). The maximum relative deviations of the fitted curve from the individual mean values amount to 0.1%; on average, to 0.03%. Taking the uncertainty of measurement of 0.5% into account, the polynomial approximation is excellent over the whole temperature range. Since it is not the intention of the present work to discuss the numerous measurement results on Pyrex-type glasses (see, e.g., Ref. 5, Vol. 1), only one set of literature data is shown in Table I (from Table 2 (Glass A, B) in Ref. 10), together with the corresponding results according to this work.

8. MEASUREMENTS ON INSULATION MATERIAL

Subsequent to the masurements on glass, the thermal conductivity of an insulation material (Microtherm) was determined in the temperature range from −60 to 180°C. The sample thickness was 10 mm; the apparent density, 0.436 $\mathrm{g \cdot cm^{-3}}$ (in the dried state). The water content was 1.9% at an air humidity of 36%. The small pore size of the material, consisting of fine, pressed powder, leads to a thermal conductivity lower than that of the filling gas. This effect becomes most obvious for the case of the higher-conductivity gases. Measurements were carried out in the nitrogen and helium atmosphere in order to demonstrate this. In addition to the measured values, Fig. 4 also shows the thermal conductivities of the pure gases (according to Ref. 5). After the placing of the sample, which had been dried previously at 120°C, the apparatus was evacuated, heated at 120°C, and then filled with nitrogen or helium. The gas filling was renewed before each measurement. In contrast to the measurements on glass, the specimen and guard ring consisted of a single-piece plate. Half the cross-sectional area of the normally existing gap is added to the area A of the sample; the correction P_{V3} needs not be considered. A special contacting of the specimen to the apparatus (oil or gas layer) could be dispensed with in view of the very low thermal conductivity of the material. Consequently, the correction ΔT_c does not apply here. The correction ΔT_m can also be dispensed with in view of the large ratio $\lambda_{metal}/\lambda_{sample}$. Hence it follows that $\Delta T = \Delta T_0$. The measurements were carried out with $\Delta T = 20$ K, resulting in a minimum

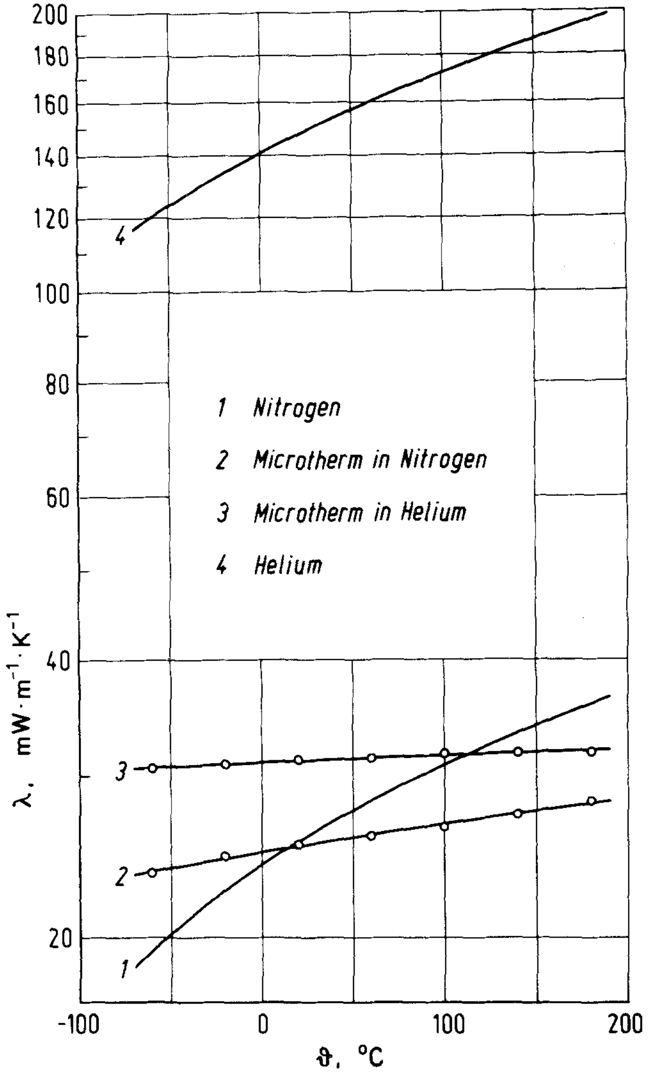

Fig. 4. Thermal conductivity of an insulation material (Microtherm) in various gas atmospheres. Thermal conductivities of helium and nitrogen were taken from the literature [5].

heating power P of 0.36 W. Regarding the uncertainty of ΔT_0, only the uncertainty of ΔT is left, which, referred to $\Delta T = 20$ K, becomes negligibly small. However, due to the small power P, the uncertainty of P_0 gains considerably in significance. The greatest uncertainties, however, occur in the determination of the area A and the thickness d. As the specimen and guard ring consist of a single piece, an effective sample diameter of 102 mm is assumed. As for the determination of the correction P_{V3}, it is assumed that half the heat flux for the area between the hot plate and the heated guard ring, which is not covered by heaters, originates from the hot plate. In this case, the uncertainty of this assumption stands for the uncertainty of the actual sample crosssection. The uncertainty of the thickness determination becomes greater, as the sample is only half as thick as the glass samples and as the material of the sample is softer. Here, too, it is very difficult to judge whether steady-state conditions exist, due to the very low power P. The resulting uncertainty can only be estimated from the repeatibility of values measured several times; it depends to a large extent on the experience of the experimenter. The overall uncertainty of these measured values is estimated to be 2%. In nitrogen and helium, the temperature dependence of the thermal conductivity can be represented by a straight line. The maximum deviations of the least-squares fit amount to 0.68% (in nitrogen) and 0.54 % (in helium). For values measured several times, a maximum scatter in repeatibility of 0.45% resulted in relation to the mean value. This scatter is clearly smaller than the estimated uncertainty of measurement of 2%.

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