INSTITUTE OF PHYSICS PUBLISHING MEASUREMENT SCIENCE AND TECHNOLOGY
Meas. Sci. Technol. **12** (2001) R89–R98 PII: S0957-0233(01)18334-7

REVIEW ARTICLE

Thermal conductivity of insulations using guarded hot plates, including recent developments and sources of reference materials

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Received 26 October 2000, accepted for publication 24 April 2001
Published 6 November 2001
Online at stacks.iop.org/MST/12/R89

Abstract
The hot plate technique for measuring the thermal conductivities (the exact term for the quantity measured is thermal transmission, which, depending on the material being measured, can have components of convective, radiative and conductive heat transfer; it is commonly referred to as the effective or apparent thermal conductivity) of insulating materials has been in existence in various forms since 1898. A brief historical survey of the early development of the experimental technique is followed by a brief description of the basic principles of the method of measurement. The technique has since become very well established and is documented in the written standard ISO8302:1991. It is now unarguably recognized as the most accurate technique for determining the thermal conductivity of insulations, having an uncertainty of about 1.5% over a limited temperature range near ambient.

Details of two guarded hot plate apparatuses designed and constructed at NPL over the last decade or so, one to measure insulations up to 250 mm thick at or around room temperature and the other to measure insulations and refractories at temperatures up to 850 °C, are given. Finally, there is a section on certified reference materials required for validating the performance of newly built guarded hot plate apparatus and for calibrating heat flow meter apparatus, a type of hot plate apparatus commonly used for quality control purposes in insulation manufacturing plant. A brief overview of these reference materials includes details of their availability, thermal conductivities and temperature ranges.

Keywords: thermal conductivity, guarded hot plate, thermal insulations, certified reference materials

1. Introduction

Whilst thermal conductivity measurements on metals date back at least to the late eighteenth century, it was not until 1898 that a quantitative method for studying poor conductors or thermally insulating materials was developed by Lees. He used a pair of specimens in the form of thin circular discs, which were clamped between three copper plates. A known amount of heat was generated electrically in the central plate and conducted in the axial direction through the specimens to the two outer plates. The temperature at the surfaces of the specimens was assumed to be the same as that of the adjacent copper plates and was measured using thermocouples. The thermal conductivity, λ, was deduced from the power supplied, Q, the mean difference in temperature between the plates, $T_h - T_c$, the cross-sectional area of the plates, A, and the mean thickness of the specimens, l, using an equation originally developed by Fourier for conduction of heat in one dimension:

$$\lambda = \frac{Ql}{2A(T_h - T_c)}.$$

The factor of two in the denominator arises because under ideal conditions, the heat flux from the central plate is shared equally between the two specimens.

In 1911 the method was improved by Poensgen [1] in Germany, who introduced an annular copper guard around the heater plate and maintained it at the same temperature as the heater plate to prevent radial heat losses. He also provided annular shielding around the specimens between the guard and cooled plates using materials of relatively low conductivity such as cork dust, magnesia and alumina, thereby maintaining additional guarding around the specimen and restricting sideways heat loss. This method provided better linear heat flow in the axial direction than could be obtained by the unguarded disc method of Lees and allowed more reliable measurements to be undertaken on thicker materials.

A further modification to the method was also made in Germany by Jakob, who developed an apparatus requiring a single disc-shaped specimen. In this case, heat was constrained to flow only in one direction from the main heater plate by providing an auxiliary guard plate on the opposite side to the specimen at the same temperature as the heater plate but separated from it by a layer of insulation. The thermal conductivity for measurements of this type is given by

$$\lambda = \frac{Ql}{A(T_h - T_c)}.$$

The symbols are the same as in equation (1) but no longer represent mean values.

More recent developments in the method are associated with

(i) the scale or size of apparatus as the use of thicker insulations has become more common;
(ii) incorporation of additional guards to further minimize lateral heat flow and edge heat losses;
(iii) improved and more accurate instrumentation, e.g. the use of multi-ranging digital voltmeters, and electronic thermocouple cold junction compensation,
(iv) computer systems for acquiring and analysing data and automation of the process of measurement; and
(v) improved temperature control systems using electronically controlled baths and power supplies and instrument interfacing.

Many of these new techniques have been incorporated into the International Standard ISO8302:1991 [2]. This standard is a distillation of the best practices contained in many of the national standards that were available at the time, e.g. BS874:Section 2.1:1986 [3], ASTM C177-85 and DIN 52 612:Part 1:1984, plus new information from the world's leading researchers working on developments of the technique.

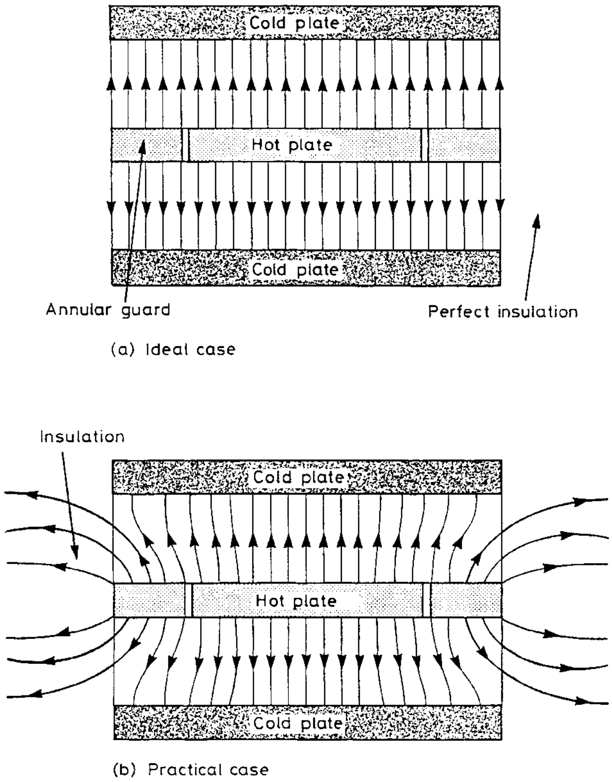

Figure 1. Distributions of heat flux lines in a double-sided guarded hot plate in (*a*) ideal and (*b*) practical cases.

As the measurement technique has improved, so the requirement for reference materials has grown, especially following the introduction and development of the heat flow meter apparatus, an alternative form of the hot plate apparatus that uses transducers to measure the heat flux density in the specimen. Reference materials are required for validation and auditing of guarded hot plates and the calibration of heat flow meter apparatus. There are many sources of reference materials available for the hot plate methods, mainly from national standards laboratories in the USA and Europe.

2. Basic principles of the method

2.1. Physical concepts

The principle of the guarded hot plate method is to generate a known unidirectional heat flux through the specimens so that they appear as slabs of infinite width bounded by parallel planes. To achieve these aims, it is customary to use a heater plate comprising two parts, a central plate (or metering area) surrounded by an annular guard and separated from it by a small air gap, which acts as a thermal barrier. Heat flows from the metering area of the heater plate through the specimens to cold plates maintained at a stable lower temperature constant to within a few hundredths of a degree Celsius. Electrical current is supplied to the guard independently of the metering area heater and the heat flux produced serves to maintain the heat flow perpendicular to the hot face in the central metering region, thereby creating isothermal planes across the measured region of the specimen (figure 1). The convention is to define the area of the specimen as the area contained within a line drawn mid-way between the gap separating the guard and metering area heaters. However, in some circumstances, for instance with specimens of moderately high thermal conductivity, say greater than 0.75 W m^{-1} K^{-1}, heat transfer across the gap via the specimen can be reduced by making the specimens the same size as the metering area so that the conduction area is of the same size as the metering area. In this case the guard area is filled with low conductivity insulation.

2.2. Practical aspects

In order to obtain ideal conditions, a number of measures must be taken. The plates of the apparatus must be as flat as possible and should be made from highly conducting material so that there is good uniformity of temperature across them. They should also have high emissivity surfaces, particularly when one is measuring low-density insulations, for which radiative heat transfer is an important component of the apparent thermal conductivity. The temperature balance between the guard and the metering area must also be maintained within close limits ($\simeq$0.01 °C) in order to prevent lateral heat exchange. A general rule is that the width of the guard should be at least 0.25 times the width of the metering area and no less than the thickness of the specimen for effective shielding. The contact resistance between the plates and the specimen should be as uniform as possible over the whole surface to ensure that there is uniform heat flow into the specimen.

It is important that the thermocouples mounted in the hot and cold plates are in good thermal contact with the plates themselves and that they read the required temperature. For materials with thermal conductivities greater than about 0.15 W m^{-1} K^{-1} the thermocouples should be in good contact with and preferably mounted on the specimen surfaces; soft rubber interface sheets can be used to improve the thermal contact between the apparatus plates and the specimens. To ensure that the thermocouples indicate the correct plate temperature, they should run parallel to the surfaces in an isothermal region to limit heat flow along them, which would otherwise cause an error in the temperature being measured. To limit the magnitude of such errors the thermocouples should be made from fine wire ($\simeq$0.2 mm thick or less) and their thermal contact resistance with the plates/specimens should be reduced using a contact medium such as zinc oxide or a suitable cement.

The control of the heat flow between the edges of the specimens and the apparatus plates is particularly important both for materials of low thermal conductivity and for thick specimens. A full discussion of how to determine these edge heat flows is given in a theoretical analysis of a special case by Woodside [4], experimental work by Woodside and Wilson [5] and a general theoretical analysis by Bode [6]. More detailed discussions of the theory and practical design of guarded hot plate and heat flow meter apparatus can be found in the written standards ISO8301:1991 and ISO8302:1991, in the review by Klarsfeld [7] and in ASTM STP879 [8].

3. The NPL guarded hot plate apparatus

3.1. The NPL single-sided combined heat flow meter and guarded hot plate

This novel apparatus was developed in the mid-1980s for accurate measurement of the thermal conductivity of low density insulating materials from 25 to 300 mm thick (figure 2). The estimated measurement uncertainties are in the range 1.3–2.4% for specimens between 50 and 250 mm thick. It serves the dual role of a guarded hot plate (GHP) and heat flow meter (HFM) apparatus, conforming with the written standards for the GHP apparatus BS 874:1981, section 2.1 and ISO 8302, as well as with that for the HFM apparatus ISO 8301 [9]. This was the first guarded hot plate of its type that was capable of measuring such thick insulation material to this level of accuracy. A brief overview of the apparatus developed by Salmon [10] is given below.

3.1.1. The design concept and construction details. Figure 2 shows the main features of the apparatus, in which heat flows upwards from the guarded heater plate through the specimen to an isothermal cold plate maintained at a lower temperature, heat flow downwards and laterally being minimized by auxiliary and edge guards, respectively. This arrangement restricts the measurement of fibrous materials to those with high enough density not to exhibit heat transfer due to convection currents within the material, the lower density limit for measurement being about 5 kg m^{-3}. The temperature balance between the metering and guard sections of the main heater, which are separated by a small gap, is maintained by using the output of a thermopile to control the power supplied to the guard heater. An auxiliary guard, which lies below the main heater and is separated from it by an insulating slab, ensures that no heat generated in the main heater flows downwards away from the specimen. The metering area heater can be operated in the constant temperature mode to rapidly achieve thermal equilibrium, or in the constant power mode for accurate measurements.

The cold plate of the apparatus is maintained at constant temperature during measurements by a combination of fluid circulation and electrical heating. A heat flux transducer mounted on the surface of the cold plate is used to monitor the rate of heat flow through the specimen. Linear temperature gradient edge guards are used to further reduce edge heat losses. When measurements are made at low mean specimen temperatures dry compressed air is passed through the apparatus to prevent condensation on parts at temperatures below the dew point.

Copper track printed circuit boards (PCBs), designed at the NPL, are used as heating elements in all the heater plates of the apparatus to ensure uniform heating over the 610 mm-square plate area. The guarded heater element has a second pair of copper tracks brought from the meter area to measure the electrical potential across the heating element accurately. The main heater plate is made of two thin copper plates with a heating element and a 20-way thermopile constructed from 0.1 mm diameter thermocouple wire clamped between them. The thermopile is adjacent to the upper working surface of the heater so that its junctions span the guard-meter area gap of the plate in contact with the specimen.

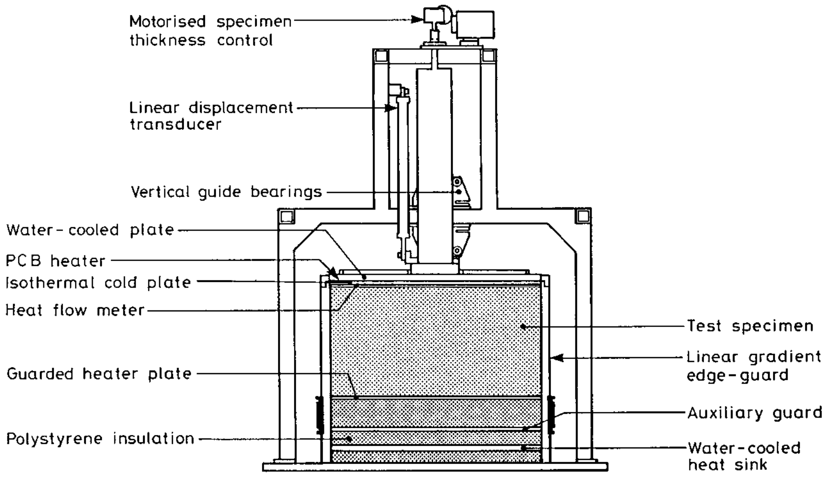

Figure 2. Essential features of the NPL 610 mm guarded hot plate/heat flow meter apparatus.

The height of the cold plate is controlled using a motor-driven fixed screw and the separation from the heater plate measured by a hybrid track displacement transducer. The transducer is calibrated *in situ* using gauge 25–300 mm thick blocks. The maximum deviation in the separation of the hot and cold plates is less than 0.3 mm. The 250 mm-square heat flux transducer (HFT) in the cold plate is calibrated in the apparatus using specimens about 37 mm thick.

The edge-guard heater is mounted on a copper former having the same width as the vertical separation between the main heater plate and the auxiliary guard. The stainless steel edge guard is clamped to the copper former at one end and to the edges of the cold plate at the other. The temperature of the copper former is controlled to be approximately the same as the temperature of the lateral guard. The 25 mm gap between the edge-guard assembly and the main heater and specimen stack is filled with insulating material to reduce heat transfer. The outside surface of the edge-guard assembly is insulated with extruded polystyrene to reduce heat transfer to the laboratory environment.

The apparatus has been partially automated using temperature controllers and a computer linked via both IEEE and RS422 instrument interfaces. The computer is programmed to determine thermal equilibrium at each required temperature and take a set of readings using a scanning system, which has relays with a low thermal offset, and a high accuracy digital voltmeter with a resolution of 0.1 μV. The equilibrium readings consist of a detailed print out of the original input data, plate temperatures and calculated thermal conductivities.

The thermocouple wire for the measurement of plate temperatures was selected very carefully. Some 1200 m of 0.2 mm diameter type E wire was wound onto four rolls and thermocouples made from both ends of each roll were calibrated by the NPL Temperature Standards section. The apparatus was wired up using the roll having the closest matching calibrations at both ends. In addition the thermocouple wire used from the roll was also checked for uniformity and homogeneity by heating it along its length and rejecting any bits of wire producing an unacceptably large thermal EMF. All the thermocouples are connected to copper wires inside an isothermal junction box before connection to the scanning unit. A reference voltage proportional to the temperature of the junctions is provided by an electronic ice-point reference unit. The computer program corrects all thermocouple voltages to a reference of 0 °C by adding on this reference voltage and then deriving temperatures using polynomial coefficients obtained for the calibrated wire.

Table 1. The estimated overall uncertainty in thermal conductivity as a function of the specimen thickness for the 610 mm guarded hot plate apparatus.

Specimen thickness (mm)	25	50	150	250
Total uncertainty (%)	1.83	1.23	1.54	2.43

Voltage-programmable power supply units and temperature controllers are used to maintain the various heater plates at stable temperatures. Differential thermocouples are used as the sensing elements to maintain temperature balances between the lateral guard and metering area heater, the auxiliary guard and lower centre heater and the edge guard and lateral guard. The cold plate is maintained at the required temperature by circulating fluid from a cooling bath through pipes attached to the plate. A very uniform temperature across the plate and good stability is obtained by using a PCB heater and a type E thermocouple as the control sensor.

The accuracy obtainable with the apparatus depends on the thickness of the specimens being measured. Table 1 shows the maximum overall uncertainty in the thermal conductivity measurement determined from the sum of the estimated uncertainties in the measurement. The main sources of uncertainty are associated with the determinations of specimen thickness, the temperature drop through the specimen and edge heat losses. The highest accuracy of $\pm 1.3\%$ is obtained for specimens between 50 and 75 mm thick. For specimens less than 50 mm thick uncertainties associated with the thickness measurement increase, whilst, for specimens greater than 75 mm thick, edge heat losses become larger, in both cases resulting in an increase in overall uncertainty. Estimates of the size of the edge heat losses are based on the observed differences between the power received by the heat flux transducer on the cold plate and the power generated in the metering area of the main heater plate.

3.1.2. A summary of the performance of the apparatus. The incorporation of a heat flux transducer on the cold plate of the apparatus provides two distinct advantages in the measurement of thermal conductivity. Firstly, the apparatus can be operated in the heat flow meter mode for more rapid but slightly less accurate measurements than when it is used in the guarded hot-plate mode. Secondly, since the arithmetic mean of the heat flux through a specimen can be determined accurately from the heat fluxes measured at the hot and cold plates, the measurement uncertainty is approximately halved. This is particularly useful under experimental conditions for which there is more likely to be non-uniform heat flow, for example when very thick specimens are being measured at temperatures significantly above or below ambient and the insulation around the edge guarding system is insufficient for a linear temperature gradient to be maintained.

An intercomparison of the 610 mm GHP apparatus with well established NPL 305 mm GHPs shows that agreement to within ±2% is obtained for specimens of 75 mm glass fibre board, which is well within the combined estimated uncertainties of the two apparatuses. Measurements with the 610 mm GHP apparatus are also fully self-consistent over the full thickness range up to 300 mm. Results obtained on a 300 mm thick specimen made from 75 mm thick glass fibre boards stacked together agree to within better than 1.5% with values calculated from individual measurements on the component parts.

3.2. The high temperature guarded hot plate

The NPL high temperature guarded hot plate was designed to provide accurate thermal conductivity measurements in the UK and calibrated transfer standards for performance checking and developing secondary methods such as the line source and panel test methods. An important feature of the high temperature apparatus, unlike the panel test methods for refractory materials (ASTM C201 [11] and BS1902 [12]), is its ability to determine the thermal conductivities of specimens using small differences in temperature of about 50 °C across the specimen throughout its measurement range of up to 850 °C, thereby minimizing any uncertainties associated with nonlinear thermal conductivity versus temperature behaviour. Brief details of the apparatus are given below; a more detailed description and assessment of its performance are given in a report by Salmon [13].

3.2.1. Design criteria. The basic design requirements for the proposed apparatus were to

(i) measure solid insulations and refractories having thicknesses in the range 25–50 mm,
(ii) measure materials with thermal conductivities of up to 1 W m^{-1} K^{-1} and thermal resistances from 0.05 to 3.2 m^2 K W^{-1},
(iii) cover the temperature range from 100 to 850 °C,
(iv) obtain a measurement uncertainty of about ±5% over the whole range of temperature and thermal resistance and
(v) fully automate the apparatus.

3.2.2. Design and construction details. A cylindrical double-sided design of 305 mm diameter was chosen in preference to a conventional square design used near ambient temperatures. The radial symmetry of the apparatus greatly simplifies the design of the lateral guard and the edge-guard system required to minimize edge heat losses at high temperatures. In addition, the number of power supply units and temperature controllers is significantly reduced, lowering construction costs. Figure 3 shows a line drawing of the basic design for the high temperature guarded hot plate.

The guarded heater plate consists of two 305 mm diameter plates about 10 mm thick bolted together. The plates are machined from Inconel 600, a high temperature nickel alloy with very good dimensional stability at temperatures up to at least 900 °C. As with the heated cold plates, one plate is machined with grooves to provide a location for the guard and metering area heating elements. The second plate has shallow grooves cut into one face to allow a thin wire, differential thermocouple to be wound across the guard–metering gap. The central metering area of the plates is separated from the concentric guard area by a 2 mm gap cut through the plates centred on a 150 mm diameter circle, three small bridging pieces being left in position to provide mechanical support for the metering area. The outer surface of each plate is machined flat to within better than 0.05 mm across the whole face. Grooves are machined into the faces to take sheathed mineral insulated thermocouples.

The two chilled plates act as heat sinks for heat flowing through the specimen. Each consists of a 30 mm thick aluminium alloy plate with cooling channels cut concentrically into the top surface. Fluid from a temperature controlled bath is pumped through the channels to maintain the temperature of the plate at 20 °C. Heated cold plates are separated from the chilled cold plates by a thick block of calcium silicate insulation. Since the chilled cold plate runs at 20 °C, the thermal conductivity of insulation and specimen and the temperature drop through the specimen determine the lower temperature limit of the heated cold plate. The two heated cold plates are made to a guarded design with the central area surrounded by a concentric lateral guard area. The 10 mm thick plates are made from Inconel alloy. Sheathed heating elements are set into grooves machined into the back surface of each plate both in the central area and in the guard section. The surface adjacent to the specimen is machined flat to within better than 0.05 mm across the face and has grooves to take four mineral insulated Nicrosil–Nisil (Type N) thermocouples and a differential thermocouple.

The edge-guard assembly consists of two semi-cylindrical Inconel 600 plates that surround the specimen stack to leave a gap of about 25 mm between the specimens and the edge guard. The edge-guard plates extend at least 30 mm beyond the heated cold plates when 50 mm thick specimens are being measured, the overlap obviously being greater for thinner specimens. Separate heating elements are wound onto the outside of each guard plate and connected in series to a power supply. The outer surfaces of the guard plates are insulated with microporous insulation encased in a stainless steel lining, the thickness of insulation being sufficient to keep the temperature of the stainless steel below about 60 °C when the edge guard is at 850 °C.

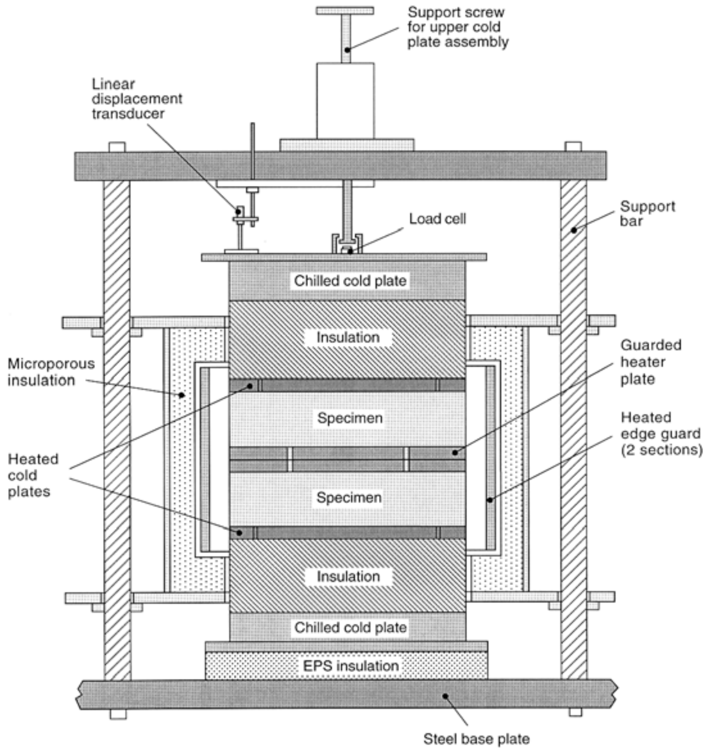

Figure 3. The NPL 305 mm diameter high temperature guarded hot-plate apparatus.

Type N thermocouples are used throughout the apparatus in the form of mineral insulated thermocouple wire in a NiCroSil sheath of 1.5 mm diameter and of sufficient length to allow the sheath terminations to be made at room temperature. Samples from the batch of sheathed thermocouples were calibrated by the NPL Temperature Standards section. The data acquisition and temperature control systems and software routines are similar to those already described above for the 610 mm guarded hot plate.

The edge-guard assembly incorporates a hinge system that allows the two semi-cylindrical parts of the guard to swing open for easy access to the specimen stack for changing specimens. When it has been assembled, the cold and chilled plate assembly rests on top of the specimen stack and moves up and down with the expansion and contraction of the apparatus during a measurement. Movement of the upper chilled plate is monitored with reference to the base plate of the apparatus using three linear displacement transducers. The transducer system is calibrated to give the thermal expansion of the specimens by calibrating out the expansion of the apparatus using microporous silica glass specimens that have a comparatively low coefficient of expansion. Checks are made using specimens whose coefficients of expansion are known and it is found that the method provides a good first-order correction to take into account the expansion of the specimens.

3.2.3. The performance of the apparatus. One of the major sources of uncertainty in measuring dense materials of relatively high conductivity in a hot-plate apparatus with thermocouples embedded in the plates is associated with the presence of air at the interfaces between the specimen and plates. Compared with the specimens, air has a relatively low thermal conductivity, so the presence of air films at the interface can greatly affect both the uniformity of the distribution of heat flux through the material and its apparent thermal resistance. However, the effect becomes progressively less severe at higher temperatures due to the increase in radiative heat transfer across the air gap. In this apparatus good thermal contact between specimens and plates is achieved by ensuring that all the plate surfaces are flat to within better than 0.05 mm and that specimen surfaces are as flat as practicable depending on the material.

The other main sources of uncertainty in thermal conductivity measurement arise from the measurement of the temperature drop through the specimens, specimen thickness and edge heat losses. Minor sources of uncertainty arise from measurement of the heater power delivered to the metering area and the dimensions of the latter. The uncertainties in determining the basic parameters measured directly, such as the heater power, temperature and specimen dimensions, have been calculated. Factors taken into account in the calculation include associated uncertainties in the calibration of the appropriate measuring instruments and transducers. Estimates of the uncertainties associated with lateral and edge-guard imbalances have been determined experimentally. By adding the individual uncertainties in quadrature an overall uncertainty of $\pm 5\%$ within 95% confidence limits has been determined for measurements covering the temperature range from 100 to 850 °C. Reproducibility tests carried out on samples re-assembled and measured in the apparatus give agreement with earlier values to within better than 1%. The apparatus is currently used for the calibration of transfer standards of high-density calcium silicate, microporous insulation and foamed glass.

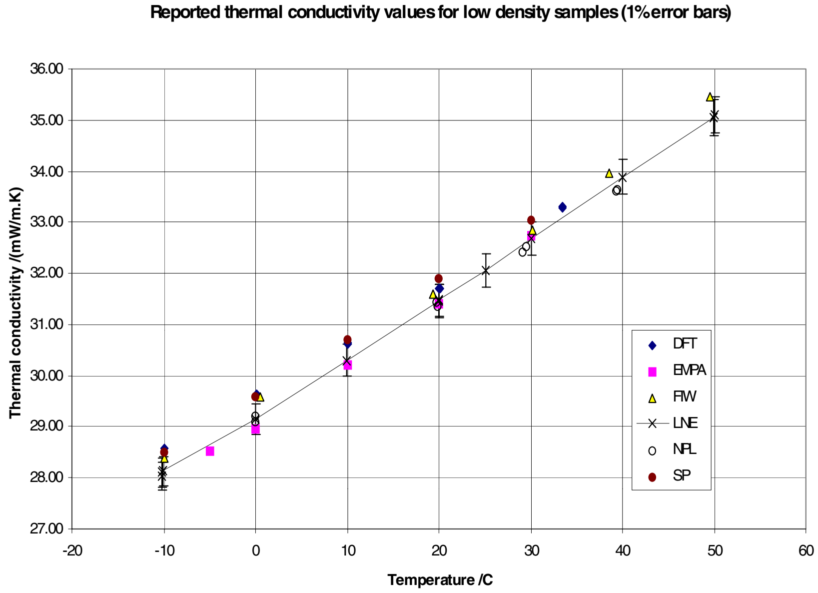

Figure 4. The thermal conductivity of IRMM-440 versus temperature over the range −10 to +50 °C.

In summary, the performance of the apparatus has been checked extensively through measurements on microporous insulation and calcium silicate. In addition, an intercomparison of the apparatus with other guarded hot-plate apparatus in Europe, as part of a BCR programme to certify a low-density calcium silicate material as a reference material, gave agreement to within better than 3% between the results from participating laboratories.

4. Reference materials

There are many comparative techniques for measuring the thermal conductivities of materials; for example, the heat flow meter apparatus and axial heat flow apparatus. In addition, many transient techniques, depending on the analytical model used for determining thermal conductivity from the temperature rise time data, require calibration to account for thermal resistances at interfaces. It is important (and also a requirement for laboratories seeking accreditation to the new international standard ISO 17025 [14]) not only that users of the guarded hot plate should verify their equipment with reference materials but also users of these secondary methods should calibrate their equipment and/or check their experimental techniques and procedures very carefully using appropriate certified reference materials similar in type to the products they measure routinely. There are several certified reference materials available for these purposes from the National Institute for Science and Technology (NIST) in the USA and the Institute for Reference Materials and Measurement (IRMM) in the European Union. Some of the reference standards available are discussed below

4.1. A European thermal insulation reference material for moderate temperatures

A measurement intercomparison programme has been completed on the characterization and evaluation of properties of a new thermal insulation reference material developed for use within the European Union. This material is a moderate density ($\simeq$70 kg m^{-3}) fibre glass board produced by Isover Saint Gobain to replace the previous European reference material stocks (BCR CRM064) that had become depleted.

Careful characterization by the manufacturer and the Laboratoire National d'Essais (LNE) indicated that this stock of material was highly reproducible and stable. Measurements of thermal conductivity were undertaken over the range −10 to +50 °C by using guarded hot plates in accordance with the ISO 8302 standard and a strict protocol. In total individual national standard or equivalent laboratories from six European Union countries (France, Germany, Italy, Sweden, Switzerland and the United Kingdom) undertook the measurements.

The results, analysed both statistically and technically by Venutti *et al* at the LNE [15] are summarized in figure 4. The detailed analysis shows that the overall uncertainty for the thermal conductivity of the batch of material is ±0.8%. This extremely good agreement validates the precision claims for the individual participating apparatuses (±2% or better) and the efficacy of the current international standard for the temperature range close to ambient. The material is now available as IRMM-440 in four different sizes from 300 mm by 300 mm to 1000 mm by 1000 mm, all 35 mm thick. The certified thermal conductivity over the temperature range −10 to +50 °C is given by the equation

$$\lambda = 0.029\,3949 + 0.000\,106\Theta + 2.047 \times 10^{-7}\Theta^2 \text{ W m}^{-1}\text{ K}^{-1} \quad (1)$$

where Θ is the mean specimen temperature in degrees centigrade. The equation is valid with an uncertainty of ±0.000 28 W m^{-1} K^{-1} for a specimen density in the range 64–78 kg m^{-3}.

4.2. Pyrex glass (BCR-039A, B and C)

The material consisted of a consignment of Dow Corning 7740 glass plates that had been prepared by Société Corning France

Table 2. Reference values of the thermal conductivity of low-density calcium silicate obtained using the parallel hot-wire method.

Temperature (°C)	Thermal conductivity ($mW\ m^{-1}\ K^{-1}$)	Standard deviation ($mW\ m^{-1}\ K^{-1}$)
25	97	8
250	108	7
500	132	9
700	155	10
900	184	12

(Sovirel) for use as thermal conductivity reference material for guarded hot-plate apparatus having plate sizes of between 300 and 500 mm. In total 81 plates with densities in the range 2222–2226 kg m^{-3} were prepared; the thermal conductivity of the glass does not vary significantly over this limited range. Certification measurements were carried out by four laboratories, the PTB, NPL, FIW and IFT, on samples 20, 30 and 50 mm thick, and the results analysed by Williams and Shawyer [16].

In the temperature range −75 to +195 °C the certified thermal conductivity of the material at temperature Θ is given by

$$\lambda = (1.1036 + 1.659 \times 10^{-3}\Theta - 3.982 \times 10^{-6}\Theta^2 + 6.764 \times 10^{-9}\Theta^3)\ \mathrm{W\ m^{-1}\ K^{-1}}$$

with an uncertainty in thermal conductivity of ±1.7% at the 95% confidence level. The conductivity has also been certified in the temperature range −130 to −75 °C with an uncertainty of ±3%.

4.3. Calcium silicate, low density

This reference material consists of a carefully prepared batch of low-density calcium silicate whose thermal conductivity has been measured by several European laboratories using parallel hot-wire and guarded hot-plate methods at temperatures up to 900 °C. The material is available from the European Union in the form of bricks of dimensions 75 mm × 115 mm × 230 mm with a density of 274 kg m^{-3} ± 2% throughout the batch. The results were summarized by Franken [17] of Hoogovens.

The thermal conductivity was measured by five laboratories using the guarded hot-plate method and, although the results from two of the laboratories had to be rejected as outliers, the remaining three sets of results were in reasonable agreement. Surprisingly, there was also fairly good agreement between hot-wire and guarded hot-plate results. Because of the anisotropy exhibited by the material it was expected that the guarded hot-plate results measured with the heat flux through the thickness should be about 6% lower than the hot-wire results in which the heat flux is radial and includes a through-the-thickness component. Insufficient thermal conductivity values were obtained using the guarded hot-plate apparatus to assign certified values to the material.

Eight laboratories carried out hot-wire measurements. However, as far as is known, although reference thermal conductivity values were assigned to the materials, the uncertainty in the measurements and the spread in the results obtained by the participants meant that these values could not be certified. The hot-wire reference values are shown in table 2 together with the standard deviation for the results at each temperature in the range 25–900 °C.

4.4. NIST reference materials

The NIST has certified a number of reference materials for thermal conductivity that are used throughout the world

4.4.1. High density glass fibre board SRM 1450c. This is the latest standard reference material issued by the NIST in the well known 1450 series of glass fibre boards that were first issued in May 1978 [18]. It is available as 25 mm thick boards, has a density in the region 150–165 kg m^{-3} and has been assigned certified thermal conductivity values in the temperature range 280–340 K. These values are given by the expression

$$\lambda = -7.7663 \times 10^{-3} + 5.6153 \times 10^{-5}\rho + 1.0859 \times 10^{-4}T$$

where ρ is the density of the board in kg m^{-3} and T is the mean temperature of the specimen in kelvins.

4.4.2. Low density fibrous blanket SRM 1451. This is a glass fibre blanket of nominal thickness 25.4 mm supplied as 610 mm × 610 mm specimens with densities ranging from 12 to 15 kg m^{-3}. Certified values are given for the thermal resistance of the blanket as a function of temperature and density. These resistances are calculated from experimental thermal conductivity data which were fitted to the model

$$\lambda(T, \rho) = a_1 + a_2\rho + a_3T/\rho + a_5 \exp\{-[(T - 180)/75]^2\}.$$

For the data on this material the coefficients have the values $a_1 = -0.1059$, $a_2 = 0.1378$, $a_3 = 0.077\,14$, $a_4 = 8472 \times 10^{-9}$ and $a_5 = 1.339$; ρ is the density of the specimen in kg m^{-3}, T is the temperature in kelvins and $\lambda(T, \rho)$ is the apparent thermal conductivity in mW m^{-1} K^{-1}. Full details are given in the NBS special publication 260-103 by Hust [19].

4.4.3. Thin expanded polystyrene board SRM 1453. The NIST has acquired and characterized a batch of 12.5 mm expanded polystyrene board of density 40 kg m^{-3} to assist the users of standard fenestration test methods for the thermal evaluation of windows, for which it would normally be used sandwiched between two 3 mm thick glass plates. It can, however, also be used for the calibration of heat flow meter apparatus and validation of guarded hot-plate apparatus. Thermal resistances have been assigned to the material over the temperature range 281–313 K [20]. The thermal conductivity of the material is represented by the equation

$$\lambda = 6.3054 \times 10^{-4} - 4.1993 \times 10^{-5}\rho + 1.1650 \times 10^{-4}T$$

where ρ is the density of the board in kg m^{-3} and T is the mean temperature of the specimen in kelvins.

5. Intercomparison of existing and proposed national reference materials

The aim of this NPL initiated international intercomparison was to establish the degree of agreement among the results of standard guarded hot-plate measurements of the national measurement organizations and thus the mutual acceptability of the available and future reference materials. Hence a programme of measurements has been carried out on some of the national reference materials currently available in various countries in order to ensure that the respective reference materials can be used internationally with an accepted level of precision in their values.

Measurements were carried out by the NPL in the UK, the NIST in the USA, the National Research Council in Canada, the LNE in France and the Japanese Testing Centre for Construction Materials in Japan. The reference materials measured were the European fibrous glass board (IRMM-440), a lower density fibrous glass blanket (SRM 1451) and a polystyrene bead board (SRM 1453) from the USA and a high-density mixed oxide–glass fibre from Japan.

Measurements have been completed and a statistical analysis of the results has been carried out by the NIST and a report is being prepared (private communication from R R Zarr, NIST). Initial results show that there is a laboratory-to-laboratory difference for each material that is material dependent, i.e. there is a possible material–laboratory interaction. Despite this, for the tests at 297.15 K, excluding two sets of outlying data for which a valid explanation exists, the maximum difference between laboratories varies from ± 0.35 to $\pm 1.4\%$ depending on the material tested. The results thus illustrate that any of these references will be suitable for use worldwide. This is especially important for the calibration of heat flow meter apparatus. Some NPL results obtained using the heat flow meter apparatus during a recent study [21] on expanded polystyrene, extruded polystyrene and a rockwool indicate that real differences in values of the order of between 2 and 3% are possible, depending on which type and source of calibration material is used. The use of certified reference materials for the above purpose is highly recommended and the ready availability and acceptance of various national materials having values of a common known precision will further improve this secondary method of measurement and help to obtain more uniformity in results obtained by use of this technique worldwide.

6. Conclusions

A brief history of the development of the guarded hot-plate method has been given and it has been shown that, although the method has a long history in recent years the ease of operation and the accuracy achievable have improved considerably. This has mainly been brought about by the advent of computing techniques allowing numerical modelling to minimize edge heat losses, improvements in data logging and analysis systems and advanced temperature controllers that rapidly establish very stable plate temperatures.

In addition the method has become very well documented through the various national and international standards such as ISO8302. However, there is still some room for improvement in these standards, particularly to cover measurements at higher temperatures, at which heat transfer by radiation starts to become the predominant mode of heat transfer in some materials.

As the overall accuracy of the apparatus has improved it has increasingly been used for the production of reference materials for calibrating secondary methods such as the heat flow meter method. There is now a range of reference materials of various thicknesses and thermal conductivities becoming available for use at near ambient temperature, as discussed in section 4. However, there is still a demand for reference materials for use at high temperatures and materials of lower thermal conductivities, comparable to those of the best insulations being produced commercially. This is particularly important for the measurement of the advanced insulation panels that are starting to appear and thicker reference materials will be required owing to the trend to use ever thicker insulations in homes, offices and industrial premises to meet the increasing demands to reduce global emissions of CO_2.

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