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A Critical Review of the European Guarded Hot Plate Standard For Operation at High Temperatures

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ABSTRACT

Measurements on thermal insulations by the guarded hot plate method at temperatures in the range -20°C to 80°C in accordance with both international and national standards are claimed, and have been shown consistently, to have an uncertainty of better than ±2% to ±3%. However national and international inter-comparisons have shown significantly higher uncertainties (from the order of ±10% to ±18%) when using the standard method at temperatures above 100°C up to 600°C and higher. Recently the Technical Committee 89 of the European standardization body CEN has established a working group (WG 11) to review the current standard, EN12667 especially with regard to possible differences and additional requirements that may be necessary for making reliable measurements at high temperatures. The intent is to examine the various potential sources of error, recommend means to minimize their effects and prepare a separate EN standard that addresses the issues in order to reduce the level of uncertainty to ±5% or better, for measurements at high temperatures.

The review has focused on the additional requirements to the current standard and contains their effects and reasons for proposed revisions. Particular areas discussed in the paper include:

(a) Changes to the apparatus principles and design, including gap heat interchange and better control of radiation and edge loss effects.
(b) Design parameters for the heater, including size, construction materials and heaters, adequate modelling, emittance of plates and temperature sensors.
(c) Temperature measurement and control, and the effect of thermal degradation on its performance.
(d) Specimen limitations.
(e) Operation and performance checks, including need for strict operation criteria and measurements on heating and cooling.
(f) Needs for an adequate range of reference materials and/or transfer standards.

The current status of the standard is discussed with particular respect to the needs for additional work that may be necessary to improve the precision further over a temperature range of 100°C to 850°C.

INTRODUCTION

Within the European Union, manufacturers of any thermal insulation material or product are required to provide mandatory CE marking, specified in terms of thermal conductivity and/or thermal resistance, to show that it conforms to the Community Directives for that particular product type. Proposed standards for products used for application in buildings are already in force and those for industrial applications are due shortly. The performance specifications are quite stringent, with narrow banding of values down to the 1 mWatt/m.K level, that have to be verified by regular testing. For such a requirement a measurement uncertainty of at least 5% or better is necessary for manufacturers of different insulations to remain competitive. Furthermore a greater uncertainty level can result in a user of the insulation to over- or under-design a system resulting in potential economic and/or energy losses with consequent adverse pollution impact on the environment.

At the present time, for the above verification purposes, the accepted methods of measurement for thermal insulation and similar low-thermal conductance materials at or near room temperature are the guarded hot plate and heat flow meter in accordance with international standards ISO 8302 (1) and ISO 8301 (2). Within Europe these are referenced in the European standards EN 12267 and EN 12264. International inter-comparisons using the applicable ISO standards, or their national predecessors upon which they are based, have verified that each method has an uncertainty level of inside ±2% or better for the hot plate (3-5), and ±3% for the heat flow meter (6,7).

However for higher temperatures, in the range above the order of 100°C up to 1000°C the heat flow meter technique is currently inadequate and cannot be considered due essentially to a lack both of high temperature reference materials and transfer standards and of suitable highly stable heat flux transducers of a sufficient type, size and sensitivity. Thus reliance has to be placed on use of the guarded hot plate method for providing the required performance values.

Unfortunately several inter-comparisons during the past 15 to 25 years involving different configurations and sized standard apparatus and systems in North America and Europe have shown much larger ranges of uncertainty. The first investigation carried out by the ASTM Committee C-8 on Refractory Materials involved a comparison of the ASTM C-201 water-flow calorimeter (8) and C-177 guarded hot plate (9) standard measurement methods on a refractory insulation over the approximate range 250°C to 1000°C (10). Later, two studies were undertaken under the auspices of the ASTM Committee C-16 on Thermal Insulation on stable high temperature materials over a temperature range up to 800°C. The first involved apparatus conforming to the ASTM C-177 Standard (11) and the second with apparatus conforming either to the C-177 or ISO 8302 (12) standards. Finally, in a more recent attempt to evaluate the potential effects of apparatus uncertainty on the forthcoming EU mandatory marking requirements, a study was initiated by the European Insulation Manufacturers Association to measure a stable high-density mineral wool product up to approximately 550°C using guarded hot plates conforming to ISO 8302 (13).

The results of the four studies are summarized in Table 1. They indicate that broad ranges of uncertainty exist with the use of apparatus claimed to conform to the existing standards at the particular time. The maximum uncertainty, depending upon the mean temperature, and in general, increasing with increase in temperature, ranged from the order of ±25% to ±37% (and much greater for the water-flow calorimeter

Table I. Summary of results of guarded hot plate intercomparison measurements.

Time period	Number of participants	Standard Referenced	Material measured	Temperature range / °C	Maximum deviation / %
1965-75	4	ASTM C177	Aluminosilicate Fibre	100 to 1000	37 at 1000 °C
!980-85	7	ASTM C177	Calcium silicate and aluminosilicate fibre	100 to 500	16 to 18 at 500 °C
1990-2000	12	ASTM C177 and ISO 8302	Ceramic fibre	100 to 1000	24 at 900 °C
1995-2005	9	ISO8302	Mineral fibre	100 to 500	12 at 500 °C

method) in the first, ±16% to ±18% in the second and third, to ±12% to ±14 % for the most recent. During this long time period the uncertainty level has decreased, due probably to improvements in instrumentation, use of computer control and automation, but it is clear that there are still problems with the basic measurement such that the current level of uncertainty is totally unacceptable for existing and forthcoming requirements of both manufacturers and users of high temperature thermal insulations.

However, further examination of the results of the most recent study that involved only ISO 8302 apparatus provides some encouragement that further improvement is possible. The results of two of the nine participants were significantly and consistently greater than 10% higher or lower than the mean value curve of all results. If these are discarded as being consistent outliers the uncertainty levels fall immediately to below ±7% at the highest temperature. This suggests that the possibility of reducing the level to ±5% or lower does exist if improvements could be made for example, to the apparatus and measurement procedures.

This possibility is illustrated further by the results obtained in another study that involved the certification of the thermal properties of Pyroceram 9606 up to 1000°C (14). This investigation used the guarded hot plate and hot wire methods for direct property measurements and thermal diffusivity and specific heat methods to derive thermal conductivity values. While this hard relatively high thermal conductivity material (approximately 4W/m.K at 20°C) is not one that would normally be measured by the guarded hot plate, requirements for the certification were such that only “absolute” methods could be used to provide certified values. In the event there was promising agreement in the measured values by three guarded hot plates to better than ±5% over their common ranges of temperature up to 800°C and overall agreement of all values by all methods to the same order of uncertainty for the complete temperature range.

As a result of the urgent need for improvements to be made in existing guarded hot plate measurement technology coupled with suggestions that there is evidence that this may be possible, CEN TC 89 the technical committee responsible for thermal measurements of thermal insulations has established a small Working Group (WG11)

The objective is to review all aspects of the present Standard. If as a result of the review it is considered inadequate for attaining a required precision of ±5% or better when operating at temperatures above 100°C to 150°C then the group shall recommend that a new standard, or a revision to the existing standard, containing proposed means to minimize the effects of sources of error shall be prepared.

The present paper discusses the initial review to identify reasons and possible sources of error or uncertainty that could explain the differences in measurement uncertainty over the two ranges of temperature, the consequent need for a new standard for higher temperatures and the status of a draft standard. The review included a number of the most significant issues such as the historical development of the method, its standards and inter-comparisons, the apparatus principles, the heater design, heat loss or interchange, temperature measurement and control, specimen limitations, operation procedures, performance verification and need for reference materials. Potential sources of error or uncertainty are identified together with recommendations to minimise their effects including those requiring additional work. A protocol is suggested for undertaking measurements in the future.

DEVELOPMENT AND REVIEW OF THE METHOD

(a) History

It is a well-accepted adage that measurements at high temperatures are more difficult to make, and that the difficulty increases “exponentially” with increase in temperature. As a consequence they are more likely to have higher uncertainties than those at or near room temperature. This is due principally to the need to use different material types and components for apparatus and its operation coupled with adverse effects of prolonged exposure to high temperatures on material properties together with the fact that radiation heat exchange becomes a dominating factor with increase in temperature.

As discussed earlier the guarded-hot-plate has been shown to be an excellent example illustrating these factors. Some obvious indications that differences in performance could be obtained at different temperatures can be seen by reference to the original 1945 draft of the well-known and internationally used ASTM Standard Test Method C-177 for the guarded hot plate. At that time, based on their early experiences, the developers of the standard do not mandate specific design details for one form of apparatus for a broad temperature range. Instead the basic concept and essential principles of the method are provided but for <u>two forms</u>. One version is recommended for operation at “low” temperatures (up to 150°C to 200°C max) and the other for the higher temperature range.

Both adhere to the same basic concept and design but the latter is seen to be much more substantial in design and constructed with materials having quite different thermal and physical properties to those for the lower temperature version. Early anecdotal evidence concerning the different uncertainties for the higher temperature version has been confirmed independently (15, 16). When using a high temperature plate at temperatures below 100°C to 150°C values (considered acceptable by the authors at the time) of the order of 2 % to 5 % different from those measured with a low temperature plate were obtained and there was also indications of differences in the slope of the curve of variation with temperature.

Qualitatively some differences can be expected since the attainment of, the uniformity of and the control of temperature to the 0.1°C to 0.2°C levels which are possible at or near room temperature with a low temperature version are much more difficult to achieve at elevated temperatures. Furthermore the uniformity of the heat source for high temperatures is more difficult to attain while the presence of any air gaps between the plates, or in and around the test stack will accentuate effects of unwanted radiation heat transfer within the system. There is now a general consensus that a plate apparatus designed specifically for high temperatures should not normally be used for measurements in the lower temperature range although this is not stated explicitly in any standard.

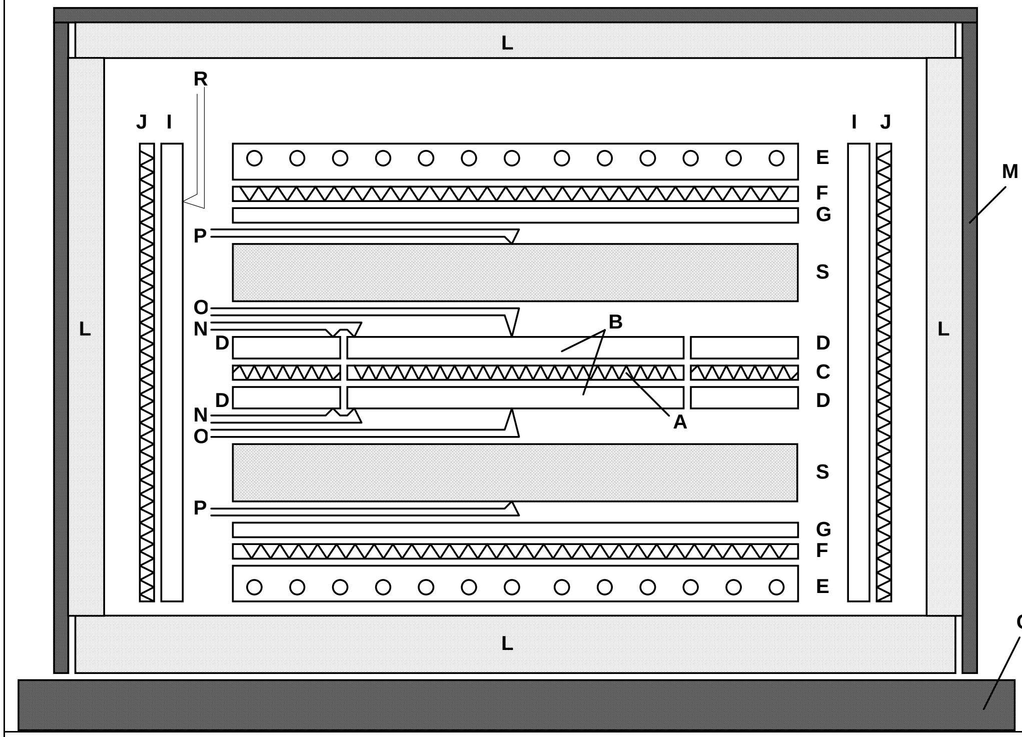
Thus the two forms of apparatus have been maintained through successive versions of both the ASTM and similar standards developed more recently. As a result there are a wide variety of different sizes and forms of standard guarded hot plates in current use for different temperatures and applications and providing different ranges of uncertainty. However the ISO 8302 Standard and the more recent editions of the C-177 Standard now contain some recommendations for verification of the performance of an apparatus or system based on accepted concepts and guidelines such that some estimate of uncertainty level is possible.

Furthermore, since the original Lees Disc unguarded method for flat slabs (17) was improved approximately a century ago by the addition of a guard by Poensgen (18) its predominant use has been directed to measurements on thermal insulations and other materials used for the building envelope and similar low temperature applications. As a result most of the many analytical and experimental studies that have been undertaken to estimate or quantify the effects of the governing parameters on the measurement uncertainty, such as the form of apparatus, the heater design, heat losses and the operational procedure, have, in general, related only to the low temperature version (19, 20). It has been assumed that the various relationships and results obtained for this regime apply equally to both forms. It is also pointed out that the analyses have usually been undertaken on “ideal” systems and specimens with the absence of radiation and convection effects.

Some attention has been directed more recently to address the high temperature version, due probably to the issue of differing uncertainty levels. Factors that have been addressed include errors due to guard gap unbalance (21), and effects of guard ring width and edge guarding (22). Effects, due especially to radiation and to use of inhomogeneous specimens, that may have more significance at high temperatures, are areas requiring further study.

(b) General Considerations

The basic concept and general features of the method shown in Fig.1 are well established. Essentially a heating plate consisting of a central uniformly heated “metering” section surrounded by a separately heated annular primary guard section with a small gap between them is sandwiched under an applied load between a “homogeneous” test specimen normally consisting of two identical (closely similar) pieces of material. The opposite faces of the specimen are in contact with separate heater/fluid cooling units and further linear or cylindrical secondary guards may be added as shown in the Figure. The whole stack is surrounded by powder or fibrous insulation and the complete system maintained in a constant temperature environment.



Key:

A Metering section heater	J Edge guard heater
B Metering section surface plates	K Test specimen
C Guard section heater	L Insulation
D Guard section surface plates	M Enclosure
E Cooling unit	N Differential thermocouples
F Cold plate heater	O Metering section surface thermocouples
G Cold surface plate	P Cold surface plate thermocouples
H Test specimen	Q Base plate
I Edge guard plate	R Edge guard plate thermocouples

Figure 1. Schematic diagram of a guarded hot plate apparatus

A temperature gradient is established in the system and the temperature difference across the gap adjusted to zero using the guard heater controlled by the output of a multi-junction differential thermocouple (or similar system) with alternate junctions attached to or in each heater side of the gap which is also filled with a loose fill insulation to eliminate convection. At equilibrium (steady-state) conditions, established in accordance with requirements given in the standard, the thermal conductivity is obtained using the standard Fourier equation involving the power to the central heater, the temperature difference across the specimen an be measured and the specimen area and the thickness (preferably measured *in situ*). In some cases the apparatus is operated in the "single-sided mode" where only one test specimen is used, usually on the lower side i.e. heat flow down, to avoid convection. A "dummy" piece of the same or similar thermal conductance as the specimen is placed on the upper side and a zero temperature difference established across this to ensure all power generated is applied to the test piece.

It should be recognized here that for a majority of thermal insulations the measured property value is an "*apparent*" one applicable only to the particular specimen unless it is known or shown to be homogeneous as defined by an applicable standard. The term thermal conductivity has been used throughout the paper.

The principle is thus simple in concept for the "ideal" case and for a homogeneous specimen but often difficult to realize in practice due to unknown and/or unquantifiable heat exchanges within the system. Based on practical limitations of apparatus size and form plus an acceptable uncertainty level, the standard method is limited, for certification purposes, to specimen thermal resistances in the approximate range

$$0.02\ \mathrm{m^2.K.W^{-1}} < R < 0.1\ \mathrm{m^2.K.W^{-1}} \tag{1}$$

This translates to a thermal conductivity range from less than 0.02 W/m.K to an upper level of less than 3 to 4 W/m.K for most material and thickness applications (not in vacuum) for most current or practical apparatus sizes and available material thickness ranges.

At these low levels very small heat inputs are involved, often of the order of tenths of a Watt in some cases depending on material and temperature, and thus any such heat loss or gain, unless accounted for, can cause a significant error in the measured value. Thus at higher temperatures where heat transfer effects do increase, due to the use of higher conductivity materials, possible convection within the system and especially greater radiation heat exchange, there are many possible sources for additional heat exchange and consequent errors to arise.

(c) Error sources

In an ideal apparatus the cold and hot plates are isothermal and the heat generated by the uniformly distributed guard and centre heater systems passes, perpendicular to the plates, through the specimens. However in the practical case the temperatures of the upper and lower hot and cold plate surfaces and their contacting specimen surfaces may not be uniform or identical due essentially to possible non-uniformity both of and in the heaters and the specimen pieces. Further sources of error arise due to heat transfer across the guard/centre gap caused by incorrect balance conditions or too many high conductivity wires crossing the gap and heat transfer from the edges of the specimen and the heater to the surrounding environment, this edge heat loss increases with increasing specimen and heater thickness.

RECOMMENDATIONS BASED ON A REVIEW OF THE STANDARD

The objective of a standard specifically for high temperature operation is to provide appropriate guidelines on the design, construction and operation of a measurement system, based on the existing document, such that additional effects due to the above issues are either eliminated or minimized and quantified. The following discussion is based on the two specimen apparatus, but also applies to the single specimen design; it highlights the main factors involved and means to provide a solution that will help to reduce their overall contribution to the measurement uncertainty.

(a) Main heater design and construction

An essential need is to provide a heat source that is very uniform and highly stable. Thus for its operational range the high temperature version needs a heater system that consists of plates and related components of suitable metals, alloys and ceramics that in general are thicker, more dense, having high thermal mass but have a sufficiently high thermal conductivity (copper and aluminium are not suitable) in relation to the plate thickness and the separation of the wires or strips forming the heating element to ensure that uniform temperatures without hot spots can be maintained at the working surfaces.

They should be chosen carefully to ensure adequate performance at the maximum operating temperatures. They need to be resistant to further oxidation or degradation once an initial high emittance film or coating has formed or been applied. The recent development of a stable high temperature paint having an emittance of 0.95 to 0.96 has been reported (23). This paint appears to be stable and robust and should be considered for use in this application particularly as a difference in emittance between 0.8 and 0.9 can introduce an error in thermal conductivity in excess of 1% to 2%.

The plates should not distort beyond their flatness requirements following repeated cycling from room temperature to the highest design temperature. This requirement is particularly important since distortion and non-parallelism of the plates will result in the formation of air gaps at the surfaces that can give rise to non-uniformity of the temperatures and the temperature differences. A heat treatment or anneal of all components after initial machining is one means of minimizing this problem. However the plate should be checked regularly after continued cyclic heating/cooling use to ensure that uniformity is maintained.

The plate is more complex than the low temperature version due essentially to the *need to have the gap completely separated but with only minimal direct contacts, including the thin heater leads and temperature sensor wires, to support the central unit and to minimize conduction across the gap. This factor also contributes to difficulties in ensuring that the whole plate maintains its uniform thickness and parallelism of surfaces especially on heating and cooling*. The gap should be filled with a suitable high temperature insulation to eliminate convection and minimize radiation heat transfer across the gap.

Most central and guard heaters now consist of sheathed wire usually wound in a uniform bifilar configuration and either directly attached to the plates or embedded in a ceramic former and sandwiched between the plates. With sheathed wire (and also with coiled wire fitted into grooves) it is quite difficult to ensure that the power to the heater in the metering area is measured directly at the guard-centre gap due to the uncertainty in the position of the voltage connections inside the sheathing. The position of these joints can be found by X-ray photography and a suitable allowance applied to the measured power during a measurement to account for the additional small lengths of wire involved. It is recommended that this correction should be less than 2%.

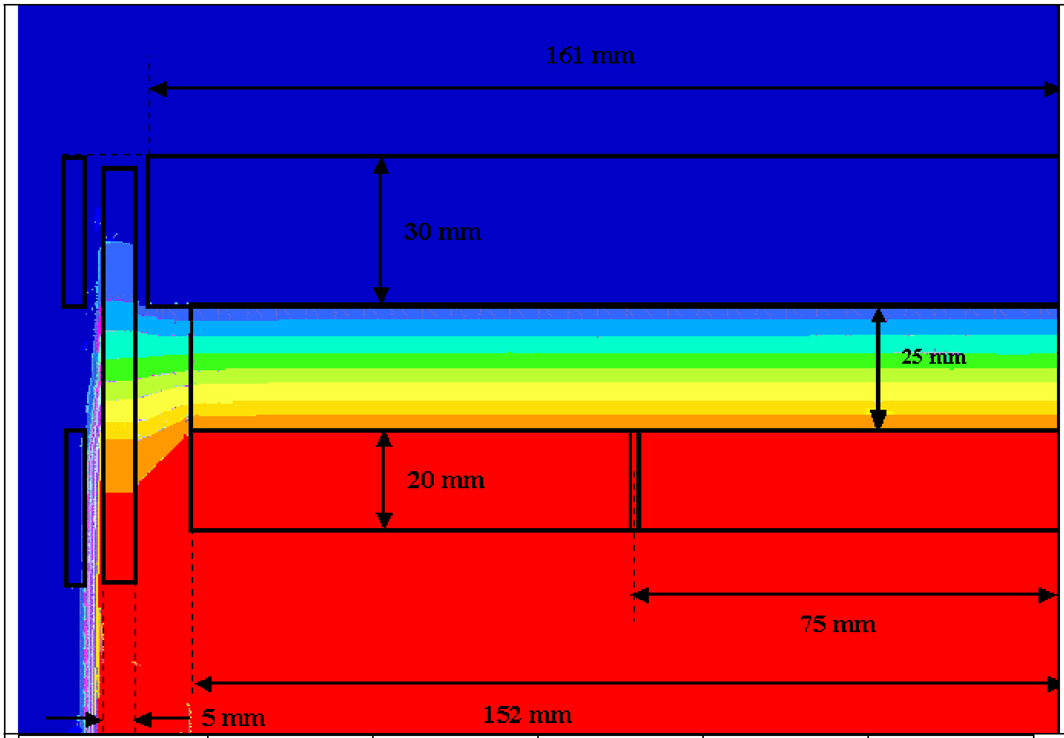
Currently both round and square plate configurations are used. Square plates are more simple in basic design and easier to construct. However for operating at high temperatures additional heaters (with individual control) are required at the corners of the guard area in order to maintain a uniform temperature across the whole plate. Round plates are simpler in design and easier to construct although greater care is required in the assembly and to ensure uniform heating per unit area. In either case it is essential that the temperature distributions in the components of the final system be modelled adequately using finite element analyses, analytical solutions, etc (24) and then experimentally verified. Fig. 2 shows a possible configuration and typical temperature distribution profile for a well-designed plate.

(b) Temperature measurement considerations

An important consideration for trying to attain improved uncertainty levels at higher temperatures is the need to reproduce the precision levels of measurement, control and stability of temperature that are possible close to ambient temperatures. In general, based on input from both members of the group and some participants of the various inter-laboratory comparisons a major problem has not only been the attainment of but also measurement of uniform temperatures to the required order of 0.1°C to 0.2°C. It would appear that in many cases the best uniformity, depending on the plate and the mean temperature, has been the order of 1°C and often larger.

In the past most high temperature plates have used Type K, or in a few cases Type N nickel based sheathed thermocouples due to their much higher sensitivity compared to the Type S platinum-platinum alloy based sensors. In addition, due to their increased fragility at high temperatures the thermocouples have tended to be larger than those used at lower temperatures and thus contribute to potentially higher heat losses. However following continued temperature cycling Type K can undergo transformations and change of calibration and is neither accurate nor stable enough to provide the required precision over continuous use.

Currently, an obvious and favoured solution to address these high precision and stability requirements is the use of small pre-calibrated platinum resistance thermometers. This is recommended and there should be **at least** one per plate fixed in the primary measurement position. In addition small diameter sheathed Type N thermocouples should be included both as additional sensors in the plates to evaluate temperature uniformity and also in the less sensitive areas such as the outer guard(s), specimen edges and control positions for the cold plates and overall temperature. In all cases regular calibration is a necessity.

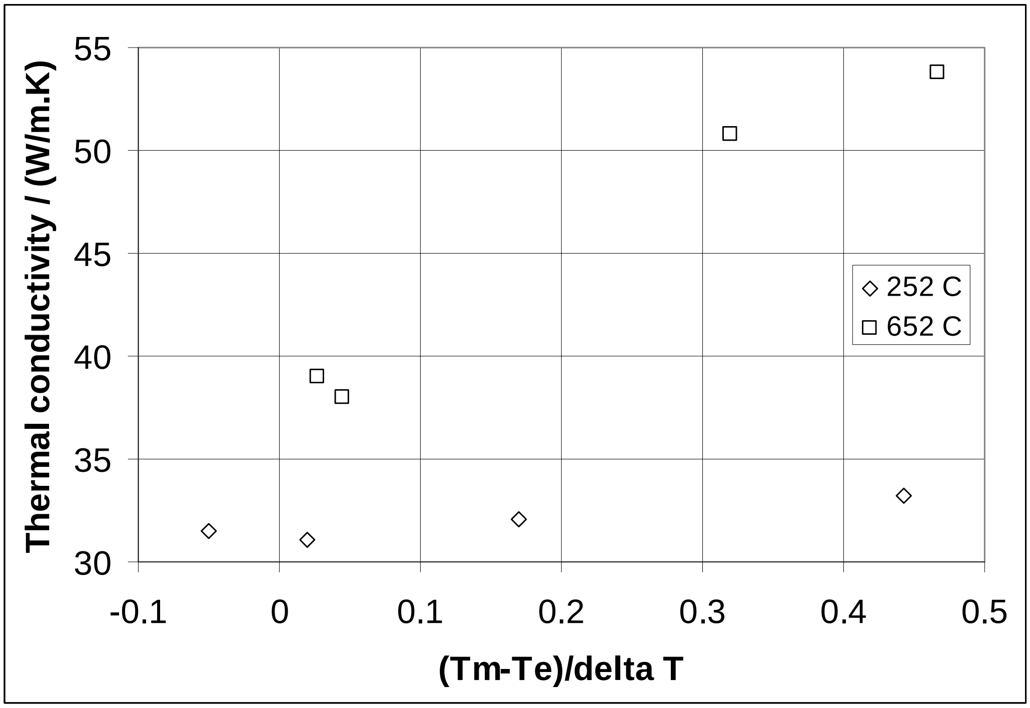


Temperature/°C	<825	825 - 836	836.1 – 841.6	841.7 – 847.1	847.2 – 852.7
Colour	🟦	🟦	🟦	🟦	🟩
Temperature/°C	852.8 – 858.2	858.3 – 863.8	863.9 – 869.3	869.4 – 874.9	>875
Colour	🟩	🟨	🟨	🟧	🟥

Figure 2. Temperature distribution in a high temperature guarded hot plate apparatus at a mean specimen temperature of 850°C

The guidelines in the current standard regarding the numbers and positions of sensors in the plates (and specimens if these have to be instrumented) should now be considered as a minimum for the high temperature version of a specific size and configuration especially for square configurations. Because of the greater effects of radiation heat exchange additional temperature sensors are necessary at the centre of the edges of each specimen piece, preferably at two diametrically opposite positions in order to evaluate corrections for edge heat loss.

While radiation was considered negligible in measurements at or near room temperature the presence of heat exchange (losses) from the edges of the specimen due to conduction has long been recognised. However it was demonstrated that for these conditions their combined effects could be minimised to an acceptable level (25) providing the hot plate was operated within a set of temperature conditions also involving the specimen edge temperature as follows:



$(T_{edge} - T_{mean})$/delta T< |0.2|

Figure 3. Variation of thermal conductivity as a function of $(T_m-T_e)/\Delta T$ for two elevated temperatures.

At ambient temperatures and with the small temperature differences (<30C) involved this condition was quite easy to maintain and as a result the edge temperature was not considered to be an important parameter in the measurement. However as the temperature increases the influence of radiation and convection becomes significant especially as larger temperature differences can be involved. One study in the past (26) indicated that this factor could be important. The results, shown in Fig.3, indicate that above 300 °C to 400 °C corrections for this factor can range from the order of 2% to over 5% depending on the mean temperature and/or the thermal conductivity of the specimen and that the relationship should be kept to:

$(T_{edge} - T_{mean})$/delta T< |0.1|

This effect of edge heat loss does appear to require more detailed study to verify its applicability at high temperatures

The multi-junction thermopile used across the gap needs to have as high sensitivity as possible and be robust while also ensuring that all heat conduction paths, including heater wires, across the gap are kept as small as possible. A recent suggestion to address sensitivity and robustness is the use of a thermopile sensor consisting of wires of Type KP (nickel-10% chromium) for the positive arm and a gold-35% palladium alloy which has a sensitivity similar to Type E thermocouples but is less susceptible to degradation at the higher temperatures (27). The thermopile needs to be installed in the heater with sufficient junctions uniformly attached opposite each other (no greater than 5mm in from the gap edges) to ensure that it can provide and maintain a representative zero or close to zero temperature difference.

(c)Test specimen considerations

In general the uniformity of thickness, flatness and overall homogeneity requirements for the test specimen are the same or more stringent as those for low temperatures. Where possible the density and homogeneity of a specimen pair should be more closely matched since the thermal conductivity tends to vary more strongly with density on increase of temperature. As the “thickness” (boundary) effect (28) will also occur at increasing higher density levels as the temperature increases (29, 30), for fibrous and cellular materials in particular care has to be taken to ensure that the thickness of the test specimen is sufficient to satisfy the standard criteria for a thermal conductivity value.

Based on experience gained through testing at low temperatures the existing standard provides criteria to establish the maximum thickness of a specimen that can be tested in an apparatus of a given size. Since current circular and square apparatus ranges from 0.3 m to 1 m the maximum thickness can range from the order of 50 mm up to 200 mm. As the size of most corresponding high temperature apparatus is only of the order of 0.2 m to 0.5 m and there is more opportunity for heat loss, including from the edges, it is suggested initially that the relevant maximum thickness levels be reduced to 30 mm to 750 mm until these can be quantified following further study.

At elevated temperatures a majority of materials have a significantly higher thermal conductivity value compared with that at room temperature. This implies that for a specimen in one piece the opportunity for lateral heat loss is greater, especially for anisotropic fibrous and layered materials. To minimize this problem it is also recommended that each specimen, especially if it is a hard or low-compressible material having an expected thermal conductivity above 0.5 to 1 W/m.K, be cut into two pieces covering the central and guard areas respectively with the same width of gap between them filled with loose fill insulation (31).

For hard and high thermal conductivity materials separate instrumentation is necessary to monitor the temperatures of the surfaces at similar positions to those in the plates. For high temperatures the sensors should normally be attached directly in small grooves cut into the surfaces due to the fragility of the technique using thin foil type sensors either attached directly or using compressible surface sheets (32) that has been used at lower temperatures.

For single specimen operation the choice of a “dummy” specimen or whatever system is used to ensure unidirectional heat flow in the main heater becomes critical. It may be difficult to adjust the temperature difference across the “ dummy” specimen to a zero value due to non-uniformity in plate temperatures caused by the difference in thermal conductance of the specimen and “dummy”. Where possible, the thickness of each should be similar but it is more important to match the thermal conductance values.

(d) Testing Procedure

From discussions with workers involved in guarded hot plate measurements it appears that where specific instructions are not supplied a test can be carried out quite differently depending on the particular interpretation of the standard by the user. This could be a significant issue in the cause of the large differences in measurement uncertainty that have occurred. As a result it is recommended that a specific test procedure be developed.

As a few examples, means to provide for constant loading of the test stack available and thickness and any changes should be measured *in situ* and checked before and after manually; unless changes in thermal conductivity during a measurement are being investigated a specimen should be pre-heated to the maximum temperature of testing prior to test; measurements should be carried out at increasing mean temperatures and one or more repeat values taken on cooling; unless a thermal conductance is required for a specific a measurement of thermal conductivity shall be made with a temperature difference of 30 to 50C; at any mean temperature a test should be made with positive and negative values of

$$(T_{edge} - T_{mean})/\text{delta T} < |0.1|$$

and a true value obtained at the zero value; record uniformity of plate temperatures, this should be better than 0.5C or 1% of temperature difference; calculation of heat exchange across the gap if the temperature difference is not zero; etc, etc.

(e) Performance checks

Various analytical and experimental criteria are provided in the current standard to enable an estimate of precision of an apparatus to be carried out. A major item is the use of reference materials or transfer standards (having accepted property values to ±3% or ±1% in some cases) that have been made available for the lower temperature range. However the major issue for high temperatures is the lack of such materials and artefacts. The need for reference materials to cover the property and temperature ranges is a matter of utmost urgency. However until there is more certainty in the efficacy of high temperature guarded hot plates it will be difficult to provide references having values with an acceptable level of uncertainty. Thus it is absolutely vital to produce apparatus to an acceptable improved standard as rapidly as possible in order to undertake the necessary measurements programmes to provide these materials.

A suggested investigation is to run a series of tests on a selected reference material(s) before and after changes are made to the standard and its modified or new apparatus. In the light of changes to the standards look for improvements in the spread between partners in order to verify the precision as well as providing acceptable references.

Candidate reference materials suggested in the past (32) have included

- A high density calcium silicate – stable to 700°C to 800°C but can be susceptible to cracking
- A blown stabilized aluminosilicate low density fibre – stable to above 1000°C and easily compressed to a reproducible specific high density
- A microporous material- NIST in USA has worked on this; it has a very low value dependent on atmospheric pressure (1% to 2% extreme), quite fragile
- A foamed glass- stable to 450C, very stable and reproducible

- There is also an urgent need for a powder- for both the guarded hot plate and the pipe insulation testing apparatus. However there are doubts about being able to reproduce uniform specimen density for both plate and pipe samples

SUMMARY

Current problems in and reasons for the measurement uncertainties of thermal conductivity at high temperatures using the guarded hot plate method have been reviewed prior to the development of a European standard for this application.

The review included various significant issues including history, standards and inter-comparisons, principles, heater design, heat losses, temperature measurement and control, use of a specific test procedure, performance verification and need for reference materials and identification of and solutions to potential sources of error. A protocol is suggested for undertaking standard measurement

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