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Thermal insulation — Determination of steady-state thermal resistance and related properties — Guarded hot plate apparatus

Isolation thermique — Détermination de la résistance thermique et des propriétés connexes en régime stationnaire — Méthode de la plaque chaude gardée

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Contents

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 8302 was prepared by Technical Committee ISO/TC 163, *Thermal insulation*.

Annex A forms an integral part of this International Standard. Annexes B, C and D are for information only.

Introduction

0.1 Document subdivision

This International Standard is divided into three sections, representing the most comprehensive assemblage of information required to use the guarded hot plate apparatus, i.e.

Section 1: General considerations

Section 2: Apparatus and error evaluation

Section 3: Test procedures

While the user of the method specified in this International Standard for test purposes may need to concentrate only on section 3, he must also be familiar with the other two sections in order to obtain accurate results. He must be particularly knowledgeable about the general requirements. Section 2 is directed towards the designer of the apparatus, but he also, in order to provide good apparatus, must be concerned with the other sections of this method. Thus, the method will serve its purpose well.

0.2 Heat transfer and measured properties

A large proportion of thermal testing is undertaken on light density porous materials. In such cases, the actual heat transfer within them can involve a complex combination of different contributions of

— radiation;

— conduction both in the solid and gas phase; and

— convection (in some operating conditions);

plus their interactions together with mass transfer, especially in moist materials. For such materials, the heat transfer property, very often wrongly called "thermal conductivity", calculated from a defined formula and the results of measurements of heat flow-rate, temperature difference and dimensions, for a specimen may be not an intrinsic property of the material itself. This property, in accordance with ISO 9288, should therefore be called "transfer factor" as it may depend on the test conditions (the transfer factor is often referred to elsewhere as apparent or effective thermal conductivity). Transfer factor may have a significant dependence on the thickness of the specimen and/or on the temperature difference for the same mean test temperature.

Heat transfer by radiation is the first source of dependence of transfer factor on specimen thickness. As a consequence, not only material properties influence results, but also the radiative characteristics of the surfaces adjoining those of the specimen. Heat transfer by radiation also

contributes to the dependence of transfer factor on temperature differences. This dependence can be experimentally detected for each type of material and for each mean test temperature when the temperature difference exceeds defined limits. Thermal resistance is therefore the property that better describes the thermal behaviour of the specimen, provided it is accompanied by information on the radiative characteristics of the adjoining surfaces. If there is the possibility of the onset of convection within the specimen (e.g. in light mineral wool for low temperatures), the apparatus orientation, the thickness and the temperature difference can influence both the transfer factor and the thermal resistance. In such cases, as a minimum it is required to fully specify the geometry and the boundary conditions of the specimen tested, even though information supplied in section 3 on test procedures does not cover these test conditions in detail. In addition, it will take considerable knowledge to evaluate the measurement, as such, especially when applying the measured values in practice.

The influence of moisture within a specimen on the heat transfer during a measurement is also a very complex matter. Therefore, dried specimens only shall be tested according to standard procedures. Measurements on moist materials need additional precautions not covered in detail in this International Standard.

The knowledge of the physical principles mentioned is also extremely important when a heat transfer property, determined by this test method, is used to predict the thermal behaviour of a specific material in a practical application even though other factors such as workmanship can influence this behaviour.

0.3 Background required

The design and subsequent correct operation of a guarded hot plate to obtain correct results and the interpretation of experimental results is a complex subject requiring great care. It is recommended that the designer, operator and the user of measured data of the guarded hot plate should have a thorough background of knowledge of heat transfer mechanism in the materials, products and systems being evaluated, coupled with experience of electrical and temperature measurements, particularly at low signal levels. Good laboratory practice in accordance with general test procedures should also be maintained.

The in-depth knowledge in each area mentioned may be different for the designer, operator and data user.

0.4 Design, size and national standards

Many different designs of guarded hot plate apparatus exist worldwide which conform to present national standards. Continuing research and development is in progress to improve the apparatus and measurement techniques. Thus, it is not practical to mandate a specific design or size of apparatus, especially as total requirements may vary quite widely.

0.5 Guidelines supplied

Considerable latitude both in the temperature range and in the geometry of the apparatus is given to the designer of new equipment since various forms have been found to give comparable results. It is recommended that designers of new apparatus read the comprehensive literature cited in annex D carefully. After completion of new apparatus, it is recommended that it be verified by undertaking tests on one or more of the various reference materials of different thermal resistance levels available.

This International Standard outlines just the mandatory requirements necessary to design and operate a guarded hot plate in order to provide correct results.

Limit values for the apparatus performance and testing conditions stated in this International Standard are given in annex A.

This International Standard also includes recommended procedures and practices plus suggested specimen dimensions which together should enhance general measurement levels and assist in improving inter-laboratory comparisons and collaborative measurement programmes.

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Thermal insulation — Determination of steady-state thermal resistance and related properties — Guarded hot plate apparatus

Section 1: General

1.1 Scope

This International Standard lays down a test method which defines the use of the guarded hot plate method to measure the steady-state heat transfer through flat slab specimens and the calculation of its heat transfer properties.

This is an absolute or primary method of measurement of heat transfer properties, since only measurements of length, temperature and electrical power are required.

Reports conforming to this standard test method shall never refer to specimens with thermal resistance lower than 0,1 $m^2 \cdot K/W$ provided that thickness limits given in 1.7.4 are not exceeded.

The limit for thermal resistance may be as low as 0,02 $m^2 \cdot K/W$ but the accuracy stated in 1.5.3 may not be achieved over the full range.

If the specimens satisfy only the requirements outlined in 1.8.1, the resultant properties shall be described as the thermal conductance and thermal resistance or transfer factor of the specimen.

If the specimens satisfy the requirements of 1.8.2, the resultant property may be described as the mean measurable thermal conductivity of the specimen being evaluated.

If the specimens satisfy the requirements of 1.8.3, the resultant property may be described as the thermal conductivity or transmissivity of the material being evaluated.

1) To be published.

1.2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 7345:1987, *Thermal insulation — Physical quantities and definitions.*

ISO 9229:—[1], *Thermal insulation — Materials, products and systems — Vocabulary.*

ISO 9251:1987, *Thermal insulation — Heat transfer conditions and properties of materials — Vocabulary.*

ISO 9288:1989, *Thermal insulation — Heat transfer by radiation — Physical quantities and definitions.*

ISO 9346:1987, *Thermal insulation — Mass transfer — Physical quantities and definitions.*

1.3 Definitions

For the purposes of this International Standard, the following definitions apply.

The following quantities are defined in ISO 7345 or in ISO 9251:

Quantity	Symbol	Units
Heat flow-rate	Φ	W
Density of heat flow-rate	q	W/m²
Thermal resistance[1]	R	m² ·K/W
Thermal conductance	Λ	W/(m² ·K)
Thermal conductivity[2]	λ	W/(m·K)
Thermal resistivity	r	m·K/W
Porosity	ξ	
Local porosity	ξ_p	

1) In some cases it may be necessary to consider also the temperature difference divided by the heat flow-rate; no special symbol is assigned to this quantity, sometimes also called resistance.

2) In the most general case $\vec{q}$ and grad T do not have the same orientation ($\vec{\vec{\lambda}}$ is not defined through a single constant λ but through a matrix of constants); moreover conductivity changes while changing position within the body, while changing the temperature and changes with time.

The following definitions related to material properties are given in ISO 9251:

porous medium
homogeneous medium
homogeneous porous medium
heterogeneous medium
isotropic medium
anisotropic medium
stable medium

Other terms not defined in ISO 7345 or ISO 9251:

1.3.1 thermally homogeneous medium: Is one in which thermal conductivity $[\vec{\vec{\lambda}}]$ is not a function of the position within the medium but may be a function of direction, time and temperature.

1.3.2 thermally isotropic medium: Is one in which thermal conductivity $[\vec{\vec{\lambda}}]$ is not a function of direction but may be a function of the position with the medium, of time and of the temperature ($[\vec{\vec{\lambda}}]$ is defined through a single value λ in each point).

1.3.3 thermally stable medium: Is one in which thermal conductivity λ or $[\vec{\vec{\lambda}}]$ is not a function of time, but may be a function of the co-ordinates, of the temperature and, when applicable, of the direction.

1.3.4 mean thermal conductivity of a specimen: Is the property defined in steady-state conditions in a body that has the form of a slab bounded by two parallel, flat isothermal faces and by adiabatic edges perpendicular to the faces, that is made of a material thermally homogeneous, isotropic (or anisotropic with a symmetry axis perpendicular to the faces), stable only within the precision of a measurement and the time required to execute it, and with thermal conductivity λ or $[\vec{\vec{\lambda}}]$ constant or a linear function of temperature.

1.3.5 transfer factor of a specimen: Is defined by

$$\mathcal{T} = \frac{qd}{\Delta T} = \frac{d}{R} \text{ W(m·K)}$$

It depends on experimental conditions and characterizes a **specimen** in relation with the combined conduction and radiation heat transfer. It is often referred to elsewhere as measured, equivalent, apparent or effective thermal conductivity of a **specimen**.

1.3.6 thermal transmissivity of a material: Is defined by

$$\lambda_t = \frac{\Delta d}{\Delta R} \text{ W/(m·K)}$$

when $\Delta d/\Delta R$ is independent of the thickness d. It is independent of experimental conditions and characterizes an insulating **material** in relation with combined conduction and radiation. Thermal transmissivity can be seen as the limit reached by the transfer factor in thick layers where combined conduction and radiation heat transfer takes place. It is often referred to elsewhere as equivalent, apparent or effective thermal conductivity of a **material**.

1.3.7 steady-state heat transfer property: Generic term to identify one of the following properties: thermal resistance, transfer factor, thermal conductivity, thermal resistivity, thermal transmissivity, thermal conductance, mean thermal conductivity.

1.3.8 room temperature: Generic term to identify a mean test temperature of a measurement such that a man in a room would regard it comfortable if it were the temperature of that room.

1.3.9 ambient temperature: Generic term to identify the temperature in the vicinity of the edge of the specimen or in the vicinity of the whole apparatus. This temperature is the temperature within the cabinet where the apparatus is enclosed or that of the laboratory for non-enclosed apparatus.

1.3.10 operator: Person responsible for carrying out the test and for the presentation through a report of the measured results.

1.3.11 data user: Person involved in the application and interpretation of measured results to judge material or system performance.

1.3.12 designer: Person who develops the constructional details of an apparatus in order to meet predefined performance limits for the apparatus in assigned test conditions and who identifies test procedures to verify the predicted apparatus accuracy.

1.4 Symbols and units

Symbol	Dimension	Unit
A	Metering area measured on a selected isothermal surface	m^2
A_g	Area of the gap	m^2
A_m	Area of the metering section	m^2
b	Guard width, starting from the gap centre-line	m
c	Imbalance coefficient	m
c_p	Specific heat capacity of the plate	J/(kg·K)
c_s	Specific heat capacity of the specimen	J/(kg·K)
d	Average thickness of a specimen	m
$d_1, d_2, ..., d_5$	Thicknesses of specimens designated $s_1, s_2, ..., s_5$	m
d_p	Metal plate thickness	m
e	Edge number	—
E_A	Error in the metering area value	—
E_d	Error in the thickness value	—
E_e	Error due to edge heat losses	—
E_E	Error in the electrical power value	—
E_g	Error due to imbalance	—
E_s	Error due to non-symmetrical conditions	—
E_T	Error in the temperature difference	—
E_ϕ	Error in the heat flow-rate	—
g	Gap width	m
h_t	Density of heat flow-rate per unit temperature difference	$W/(m^2 \cdot K)$
$2l$	Side length of the metering section from gap centre to gap centre	m
m_c	Relative mass change after conditioning	—
m_d	Relative mass change due to a conditioning after drying	—
m_r	Relative mass change after drying	—
m_w	Relative mass change after test	—
M_1	Mass as received	kg
M_2	Mass after drying	kg
M_3	Mass after conditioning	kg
M_4	Mass after test	kg
M_5	Mass before test	kg
P	Perimeter	m
q	Density of heat flow-rate	W/m^2
q_e	Edge density of heat flow-rate	W/m^2
r	Thermal resistivity	m·K/W
R	Thermal resistance	$m^2 \cdot K/W$
R_e	Thermal resistance of edge insulation	$m^2 \cdot K/W$
t	Time	s
$\mathcal{T}$	Transfer factor	W/(m·K)
T_1	Temperature of the warm surface of the specimen	K
T_2	Temperature of the cold surface of the specimen	K
T_a	Ambient temperature (temperature in the vicinity of the specimen)	K

Symbol	Dimension	Unit
T_e	Temperature on the edge of the specimen	K
T_m	Mean temperature (usually $(T_1 + T_2)/2$)	K
V	Volume	m^3
y	Heating unit thickness	m
Z_1	Error parameter for the edge configuration	—
Z_2	Error parameter for the surrounding temperature	—
Z_3	Error parameter for imbalance	—
Δd	Increment of thickness	m
ΔR	Increment of thermal resistance	$m^2 \cdot K/W$
ΔT	Temperature difference (usually $T_1 - T_2$)	K
ΔT_g	Temperature difference through the gap	K
Δt	Time interval	s
$\Delta\mathcal{T}$	Increment of transfer factor	W/(m·K)
ε	Emissivity	W/(m·K)
λ	Thermal conductivity	W/(m·K)
λ_g	Thermal conductivity of a material facing the gap	W/(m·K)
λ_t	Thermal transmissivity	W/(m·K)
Λ	Thermal conductance	$W/(m^2 \cdot K)$
ξ	Porosity	—
ξ_p	Local porosity	—
Φ	Heat flow-rate	W
Φ_e	Heat flow-rate due to edge heat losses	W
Φ_{el}	Heat flow-rate on the edge	W
Φ_g	Heat flow-rate due to imbalance	W
Φ_T	Heat flow-rate in a test	W
Φ_w	Heat flow-rate through the wires	W
Φ_o	Gap heat flow-rate per unit temperature imbalance	W/K
δ_d	Density of the dry specimen	kg/m^3
ρ_p	Density of the plate	kg/m^3
ρ_s	Density of the specimen after conditioning	kg/m^3
σ_n	Stefan-Boltzmann constant	$5{,}67\ W/(m^2 \cdot K^4)$

1.5 Significance

1.5.1 Factors influencing heat transfer properties

The heat transfer properties of a specimen of material may

— vary due to variability of composition of the material or samples of it;

— be affected by moisture or other factors;

— change with time;

— change with mean temperature; and

— depend upon the thermal history.

It must be recognized, therefore, that the selection of a typical value of heat transfer properties representative of a material in a particular application shall be based on a consideration of these factors and will not necessarily apply without modification to all service conditions.

As an example, this method provides that the heat transfer properties should be obtained on dried specimens, although in service such conditions may not be realized.

Even more basic is dependence of the heat transfer properties on variables such as mean temperature and the temperature difference. These dependencies should be measured or the tests made under conditions typical of use.

1.5.2 Sampling

Heat transfer properties need an adequate amount of test information to be considered representative of a material. A heat transfer property of a material can be determined by a single measurement only if the sample is typical of the material and the specimen(s) is (are) typical of the sample. The procedure for selecting the sample should normally be specified in the material specification. The selection of the specimen from the sample may be partly specified in the material specification. As sampling is beyond the scope of this test method, when the problem is not covered by a material specification, appropriate documents shall be considered.

1.5.3 Accuracy and reproducibility

The evaluation of the accuracy of the method is complex and is a function of the apparatus design, of the related instrumentation and of the type of specimen under test. However, apparatus constructed and operated in accordance with this method is capable of measuring heat transfer properties accurate to within $\pm$ 2 % when the mean temperature of the test is near the room temperature.

With adequate precautions in the design of the apparatus, and after extensive checking and cross-referencing of measurements with other similar apparatus, an accuracy of about $\pm$ 5 % should be obtainable anywhere in the full operating range of an apparatus. Such accuracy is normally easier to attain using separate apparatus for the extremes in the range. The reproducibility of subsequent measurements made by the apparatus on a specimen maintained within the apparatus without changes in test conditions is normally much better than 1 %. When measurements are made on the same reference specimen removed and then mounted again after long time intervals, the reproducibility of measurements is normally better than $\pm$ 1 %. This larger figure is due to minor changes in test conditions, such as the pressure of the plates on the specimen (that affect contact resistances), the relative humidity of the air around the specimen (that affects its moisture contents), etc.

These levels of reproducibility are required to identify errors in the method and is desirable in quality control applications.

1.6 Principle

1.6.1 Apparatus principle

The guarded hot plate apparatus is intended to establish within specimen(s), in the form of uniform slab(s) having flat parallel faces, a unidirectional uniform density of heat flow-rate at steady-state conditions as the one that would exist in an infinite slab bounded by two flat parallel isothermal surfaces.

1.6.2 Apparatus types

From this basic principle were derived two types of guarded hot plate apparatus:

a) with two specimens (and a central heating unit);

b) with a single specimen.

1.6.2.1 Two-specimen apparatus

In the two specimen apparatus [see figure 1a)], a central round or square flat plate assembly consisting of a heater and metal surface plates and called the heating unit is sandwiched between two nearly identical specimens. The heat flow-rate is transferred through the specimens to separate round or square isothermal flat assemblies called the cooling units.

1.6.2.2 Single-specimen apparatus

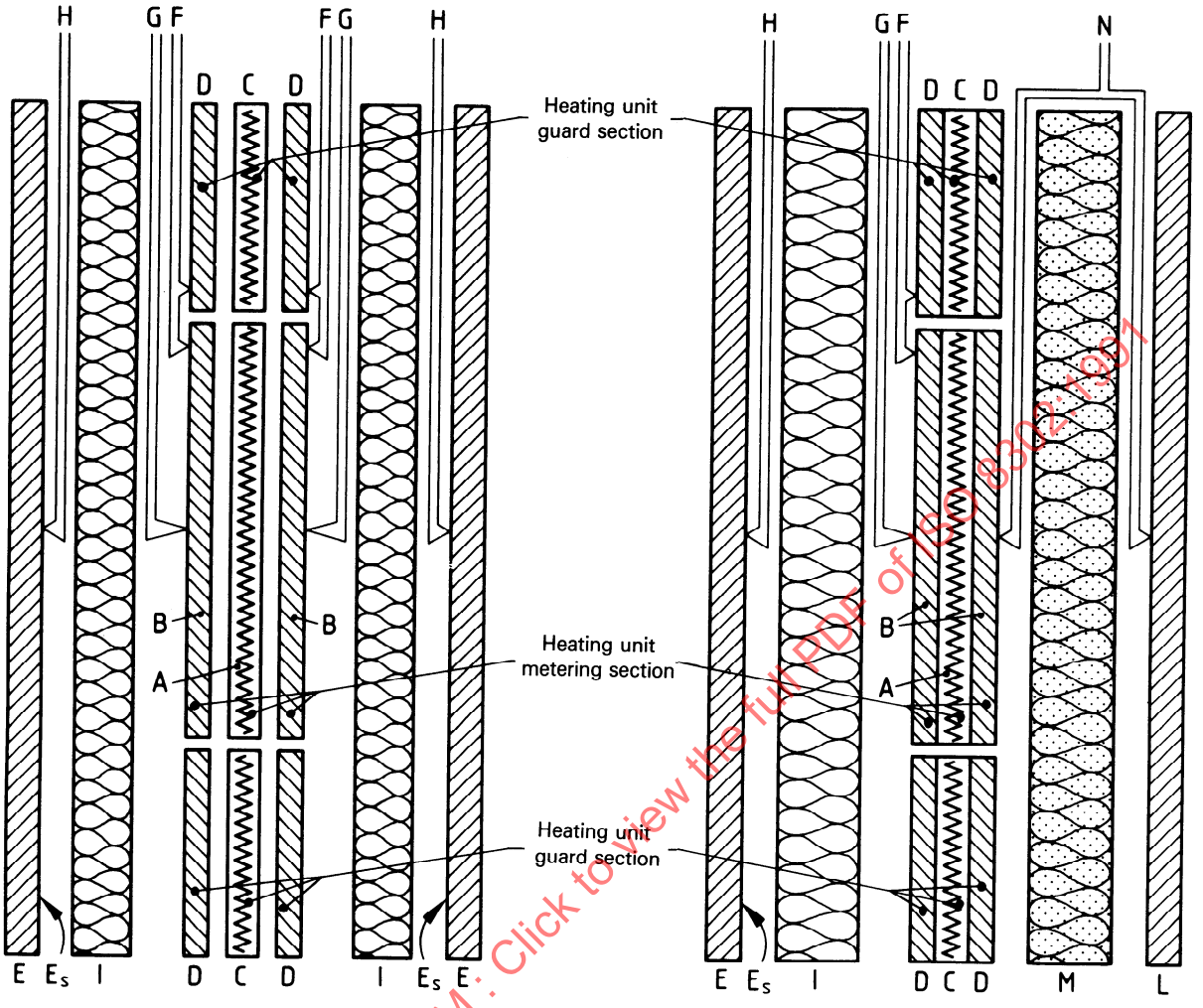
In the single specimen apparatus [see figure 1b)], the second specimen is replaced by a combination of a piece of insulation and a guard plate. A zero temperature-difference is then established across this combination. Providing all other applicable requirements of this Internation Standard are fulfilled, accurate measurements and reporting according to this method may be accomplished with this type of apparatus, but particular reference to the modification of the normal hot plate apparatus with two specimens should be made in the report.

1.6.3 Heating and cooling units

The heating unit consists of a separate metering section, where the unidirectional uniform and constant density of heat flow-rate can be established, surrounded by a guard section separated by a narrow gap. The cooling units may consist of a continuous flat plate assembly but it is preferable to have them in a similar form to the heating unit.

1.6.4 Edge insulation and auxiliary guarded sections

Additional edge insulation and/or auxiliary guard sections are required, especially when operating above or below room temperature.



a) Two-specimen apparatus

b) Single-specimen apparatus

Key

A Metering section heater
B Metering section surface plates
C Guard section heater
D Guard section surface plates
E Cooling unit
E_s Cooling unit surface plate
F Differential thermocouples
G Heating unit surface thermocouples
H Cooling unit surface thermocouples
I Test specimen
L Guard plate
M Guard plate insulation
N Guard plate differential thermocouples

Figure 1 — General features of two-specimen and single-specimen guarded hot plate apparatus

1.6.5 Definition of the guarded hot plate apparatus

The term "guarded hot plate" applies to the entire assembled apparatus, that, hence, is called "guarded hot plate apparatus". The general features of the apparatus with specimens installed are shown in figure 1.

1.6.6 Measuring the density of heat flow-rate

With the establishment of steady-state in the metering section, the density of heat flow-rate, q, is determined from measurement of the heat-flow-rate, Φ, and the metering area, A, that Φ crosses.

1.6.7 Measuring the temperature difference

The temperature difference across the specimen, ΔT, is measured by temperature sensors fixed at the surfaces of the metal plates and/or those of the specimens where appropriate.

1.6.8 Measuring the thermal resistance or transfer factor

The thermal resistance, R, is calculated from a knowledge of q, A and ΔT if the appropriate conditions given in 1.8.1 are realized. If the thickness, d, of the specimen is measured, the transfer factor, $\mathcal{T}$, may be computed.

1.6.9 Computing thermal conductivity

The mean thermal conductivity, λ, of the specimen may also be computed if the appropriate conditions given in 1.8.2 are realized and the thickness, d, of the specimen is measured.

1.6.10 Apparatus limits

The application of the method is limited by the capability of the apparatus to maintain the unidirectional uniform and constant density of heat flow-rate in the specimen coupled with the ability to measure power, temperature and dimensions to the limit of accuracy required.

1.6.11 Specimen limits

The application of the method is also limited by the form of the specimen(s) and the degree to which they are identical in thickness and uniformity of structure (in the case of two-specimen apparatus) and whether their surfaces are flat or parallel.

1.7 Limitations due to apparatus

1.7.1 Limitations due to contact resistances

When testing a specimen of high thermal conductance and rigid (i.e. specimens of a material too hard and unyielding to be appreciably altered in shape by the pressure of the heating and cooling units), even small non-uniformities of the surface of both specimen and the apparatus (surfaces not perfectly flat) will allow contact resistances not uniformly distributed between the specimens and the plates of the heating and of the cooling units.

These will cause non-uniform heat flow-rate distribution and thermal field distortions within the specimens; moreover, they will make accurate surface temperature measurements difficult to undertake. For specimens having thermal resistances less than 0,1 $m^2 \cdot K/W$, special techniques for measuring surface temperatures will be required. Metal surfaces should be machined or cut flat and parallel and stress-relieved.

1.7.2 Upper limits for the thermal resistance

The upper limit of thermal resistance that can be measured is limited by the stability of the power supplied to the heating unit, the ability of the instrumentation to measure power level and the extent of the heat losses or gains due to temperature imbalance errors (analysed later) between the central metering and guard sections of the specimens and of the heating unit.

1.7.3 Limits to temperature difference

Provided that uniformity and stability of the temperature of the surfaces of the heating and cooling unit plates, the noise, resolution and accuracy of the instrumentation and the restrictions on temperature measurements can be maintained within the limits outlined in sections 2 and 3, temperature differences as low as 5 K, when measured differentially, can be used in the measurements, provided the requirements described in 2.1.4.1.2 to 2.1.4.1.4 are met. Lower temperature differences shall be reported as non-compliance with this International Standard.

If temperature measurements of each plate are made by means of thermocouples with independent reference junctions, the accuracy of the calibration of each thermocouple may be the limiting factor in the accuracy of measured temperature differences. In this case, it is recommended that temperature differences of at least 10 K to 20 K are used in order to minimize temperature-difference measurement errors.

Higher temperature differences are limited only by the capability of the apparatus to deliver enough

power while maintaining required temperature uniformity.

1.7.4 Maximum specimen thickness

The boundary conditions at the edges of the specimens due to the effects of edge insulation, of auxiliary guard heaters and of surrounding ambient temperature will limit the maximum thickness of specimen for any one configuration, as described in section 2 (see also 3.2.1). For inhomogeneous, composite or layered specimens, the mean thermal conductivity of each layer should be less than twice that of any other layer.

This shall be regarded as a rough rule of thumb asking only for an estimate made by the operator that does not necessarily imply the measurement of conductivity of each layer. it is expected that in this situation the accuracy will remain close to the one predictable for tests on homogeneous specimens. No guidelines can be supplied to assess measurement accuracy when this requirement is not met.

1.7.5 Minimum specimen thickness

The minimum specimen thickness is limited by contact resistances given in 1.7.1. Where thermal conductivity or thermal resistivity or thermal transmissivity or transfer factor is required, the minimum specimen thickness is also limited by the accuracy of the instrumentation for measuring the thickness.

1.7.6 Metering area definition

Theoretical investigations show that the metering area, i.e. the area of the specimen traversed by the heat flow-rate fed by the central metering section, is related to the specimen thickness and to the gap width. As the thickness tends to zero, the metering area tends to the area of the central metering section, while for thick specimens the metering area is bounded by the line defining the centre of the gap (2.1.1.3). To avoid complex corrections, this definition can be retained, provided the thickness of the specimen is at least ten times the width of the gap. For some special applications see also 3.1c).

1.7.7 Maximum operating temperature

The maximum operating temperature of the heating and cooling units may be limited by oxidation, thermal stress or other factors which degrade the flatness and uniformity of the surface plate and by changes of electrical resistivity of electrical insulations which may affect accuracy of all electrical measurements.

1.7.8 Vacuum conditions

Particular care must be taken if a guarded hot plate apparatus is used for measurements under vacuum conditions. If a high vacuum is desired, the materials of the apparatus must be carefully selected to avoid excessive outgassing. Under vacuum conditions, especially at lower temperatures, serious errors can arise if due care is not taken when installing heater and temperature sensor leads so as to minimize extraneous heat flow-rates and temperature measurement errors.

1.7.9 Apparatus size

The overall size of a guarded hot plate apparatus will be governed by the specimen dimensions which range normally within the limits of 0,2 m to 1 m diameter or square. Samples smaller than 0,3 m may not be representative of the bulk material, while specimens larger than 0,5 m may create considerable problems in maintaining the flatness of the specimens and plates, temperature uniformity, equilibrium time and total cost within acceptable limits.

For ease of inter-laboratory comparisons and for general improvement in collaborative measurements, it is recommended that the design of future guarded hot plate apparatus be based upon one of the following suggested standard dimensions:

— 0,3 m diameter or square;

— 0,5 m diameter or square;

and in addition:

— 0,2 m diameter or square if only homogeneous materials are tested;

— 1 m diameter or square if specimens are to be measured at a thickness that exceeds the limits permitted for an 0,5 m apparatus.

1.8 Limitations due to specimen

1.8.1 Thermal resistance, thermal conductance or transfer factor

1.8.1.1 Specimen homogeneity

When making measurements of thermal resistance or thermal conductance in inhomogeneous specimens, the density of heat flow-rate both within the specimen and over the faces of the metering area may be neither unidirectional nor uniform. Thermal field distortions will be present within the specimen and can give rise to serious errors. The region in the specimen contiguous to the metering area and especially near the edges of this area is most critical.

It is hard to give reliable guidelines on the applicability of the method in such cases. The major risk is that the imbalance errors, edge heat loss errors, etc., now unpredictable, can vary in an unpredictable way when inhomogeneities take different relative positions within the specimen. The result is that all the checks proposed in 3.4 can be affected by systematic errors masking the true differences related to the different tests.

In some specimens the variation in structure may occur over small distances. This is true for many thermal insulations.

In other specimens direct thermal short circuits may exist between the surfaces of the specimens in contact with the plate of the heating and cooling units. The largest effect occurs when sections of material which conduct heat readily, with extended surface area on each side of the specimen, are connected by a path of low thermal resistance relative to other paths.

1.8.1.2 Temperature-difference correlation

Thermal resistance or thermal conductance are often a function of temperature differences across the specimen. In the report, the range of temperature differences that apply to the reported values of the two properties must be defined, or it must be clearly stated that the reported value was determined at a single temperature difference.

1.8.2 Mean thermal conductivity of a specimen

In order to determine the mean thermal conductivity (or thermal transmissivity) of a specimen (see 1.3.4), the criteria of 1.8.1 shall be fulfilled. The specimen shall be homogeneous or homogeneous porous as defined in ISO 9251. Homogeneous porous specimens shall be such that any inhomogeneity has dimensions smaller than one-tenth of the specimen thickness. In addition, at any one mean temperature, the thermal resistance shall also be independent of the temperature difference established across the specimen.

The thermal resistance of a material is known to depend on the relative magnitude of the heat transfer process involved. Heat conduction, radiation and convection are the primary mechanisms. However, the mechanisms can combine or couple to produce non-linear effects that are difficult to analyse or measure even though the basic mechanisms are well researched and understood.

The magnitude of all heat transfer processes depends upon the temperature difference established across the specimen. For many materials, products and systems, a complex dependence may occur at temperature differences which are typical of use. In these cases, it is preferable to use a temperature difference typical of use and then to determine an approximate relationship for a range of temperature differences. The dependence can be linear for a wide range of temperature differences.

Some specimens, while meeting the homogeneity criteria, are anisotropic in that the component of thermal conductivity measured in a direction parallel to the surfaces is different to that measured in a direction normal to the surfaces. For such specimens, this can result in larger imbalance and edge loss errors. If the ratio between these two measurable values is lower than two, reporting according to this method is still possible if imbalance and edge heat loss errors are determined separately with anisotropic specimens mounted in the apparatus.

1.8.3 Thermal conductivity, thermal transmissivity or thermal resistivity of a material

1.8.3.1 General

In order to determine the thermal conductivity or thermal resistivity of a material, the criteria of 1.8.2 shall be fulfilled. In addition, adequate sampling must be performed to ensure that the material is homogeneous or homogeneous porous, and that the measurements are representative of the whole material, product or system. The thickness of the specimens must be greater than that for which the transfer factor of the material, product or system does not change by more than 2 % with further increase in thickness.

1.8.3.2 Dependence on specimen thickness

Of the processes involved, only conduction produces a thermal resistance that is directly proportional to the thickness of a specimen. The others result in a more complex relationship. The thinner and less dense the material, the more likely that the resistance depends on processes other than conduction. The result is a condition that does not satisfy the requirements of the definitions for thermal conductivity and thermal resistivity — both of which are intrinsic properties — since the transfer factor shows a dependence on the specimen thickness. For such materials, it may be desirable to determine the thermal resistance at conditions applicable to their use. There is believed to be a lower limiting thickness for all materials below which such a dependence occurs. Below this thickness, the specimen may have unique thermal heat transfer properties, but not the material. It remains, therefore, to establish this minimum thickness by measurements.

1.8.3.3 Determination of minimum thickness for which heat transfer properties of the material may be defined

If the minimum thickness for which the thermal transmissivity can be defined is not known, it is necessary to estimate this thickness.

In the absence of an established method, the somewhat crude procedure outlined in 3.4.2 may be used for determining the thickness and whether it occurs in the range of thicknesses in which a material is likely to be used.

It is important to differentiate between added thermal resistance in measurements caused by the placement of the temperature sensors below the surfaces of the plates, added resistance caused by poor specimen surfaces, and added resistance caused by the coupling of the conduction and radiation modes of heat transfer in the specimens. All three can affect the measurements in the same way, and often the three may be additive.

1.8.4 Warping

Special care should be exercised with specimens with large coefficients of thermal expansion that warp excessively when subjected to a temperature gradient. The warping may damage the apparatus or may cause additional contact resistance that may lead to serious errors in the measurement. Specially designed apparatus may be necessary to measure such materials.

Section 2: Apparatus and error evaluation

2.1 Apparatus description and design requirements

Throughout the majority of this section aparatus requiring the use of a pair of specimens is described; the requirements that apply to the single-specimen apparatus can be readily identified.

2.1.1 Heating unit

2.1.1.1 General description

The heating unit consists of a central metering section and a guard section. The metering section consists of a metering section heater and metering section surface plates. The guard section consists of one or more guard heaters and guard surface plates. The surface plates are usually made of metal of high thermal conductivity.

The working surfaces of the heating unit and cooling unit plates shall not chemically react with the specimen and the environment, shall be smoothly finished to conform to a true plane, and should be checked periodically.

The maximum departure of a surface from a plane should not exceed 0,025 % in all operating conditions, i.e. with reference to figure 2, assuming an ideal plane to be in contact with the surface at P, then at any other point B of the surface the ratio between the distance AB from the plane and the distance AP from the reference contact point shall be smaller than 0,025/100.

2.1.1.2 Materials

The materials used in the construction of the heating unit must be chosen carefully giving adequate consideration to their performance at the temperatures at which the heating unit is to be operated.

The heating unit shall be designed to ensure adequate density of heat flow-rate and suitable characteristics for the intended use. The heating unit shall be designed and constructed so that when in operation, deviations from temperature uniformity for each face are not greater than 2 % of the temperature difference across the specimen.

For the two-specimen apparatus the two faces of the metering section and of the guard section should be within 0,2 K of their average temperature, at least for specimens having a thermal resistance greater than 0,1 $m^2 \cdot K/W$ and tested at a mean test temperature close to room temperature.

The heating unit shall also be designed and constructed so that the two faces do not warp or depart from planeness at the operating temperatures.

The surfaces of all plates shall have and maintain a total hemispherical emittance greater than 0,8 at the operating temperatures.

2.1.1.3 Gap and metering area

The heating unit shall have a definite separation or gap between the surface plates of the metering and the guard section. The area of the gap in the plane of the surface plate shall not be more than 5 % of the metering section area.

The separation between the heater windings and the gap between the metering section and the adjacent guard section shall be designed so as to distribute heat to the surface plates uniformly according to the temperature uniformity criteria in 2.1.1.2.

The dimension of the metering area shall be determined by measurements to the centre of the gap that surrounds this area, unless calculations or tests are used to define the area more precisely. For some special applications, see 3.1 c).

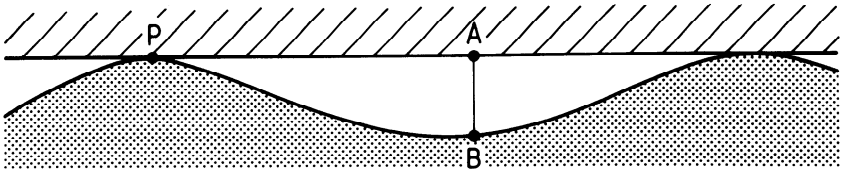


Figure 2 — Surface departure from a true plane

2.1.1.4 Imbalance through the gap

A suitable means such as a multijunction thermopile shall be provided to detect the average temperature imbalance between the surface plates of the metering and guard sections.

When a temperature imbalance exists between the metering and guard sections, an amount of heat will flow between the two elements, partly through the specimen (heat flow-rate dependent on temperature imbalance and specimen thermal conductivity) and partly through the gap itself (heat flow-rate dependent on temperature imbalance only). This heat flow-rate crossing the gap under thermal imbalance is the most severe limit when measuring high resistance specimens.

In a square guarded hot plate apparatus, it is known that temperature imbalance is not perfectly uniform along the whole gap, though little quantitative information is available on this subject. When only a limited number of imbalance sensors is used, it is suggested that the most representative positions to detect the average balance will be those at a distance from the corners of the metering section equal to one-quarter of the side of the metering section along the gap.

The corners and the axes shall be avoided (see figure 3 and reference [5]).

2.1.1.5 Imbalance sensors

If the imbalance sensors are installed in supporting sheets placed between the metal plates and the specimen(s) [see figure 4a)], or within grooves in the metal plates on the side in contact with the specimen(s), thermal resistances can exist between the sensor and the metal plates, between the sensors and the surface of the specimen(s), and between the sensors mounted on the metering section and those mounted on the guard section. The effect of thermal resistance between imbalance sensors and metal plates or specimen(s), as shown in figure 4a), in principle applies to all these situations.

When the apparatus is in operation, the temperature of the sensors is due to the combined effect of thermal balance between the metal plates of the metering section and of the guard section and of the density of heat flow-rate flowing from the metal plates to the specimen. Correct balance is obtained only if the resistance between the metal plates and the sensors is made negligible with respect to the other resistances mentioned or when the heat flow-rate flowing from the plates to the specimens does not cross the sensors, as in figure 4b) or 4c). Similar considerations are true when the sensors are installed between the metal plates and the electrical heaters of the heating unit.

As a result, the installation of the sensors in grooves in the metal plates either facing the specimens or the heaters, the use of thin sheets supporting the imbalance sensors, or similar solutions, should be avoided unless a careful experimental and analytical check under all operating conditions of the thermal resistances mentioned has been carried out.

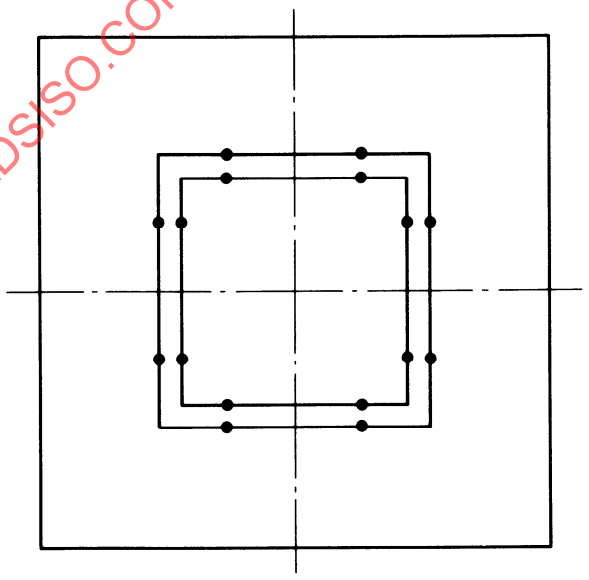
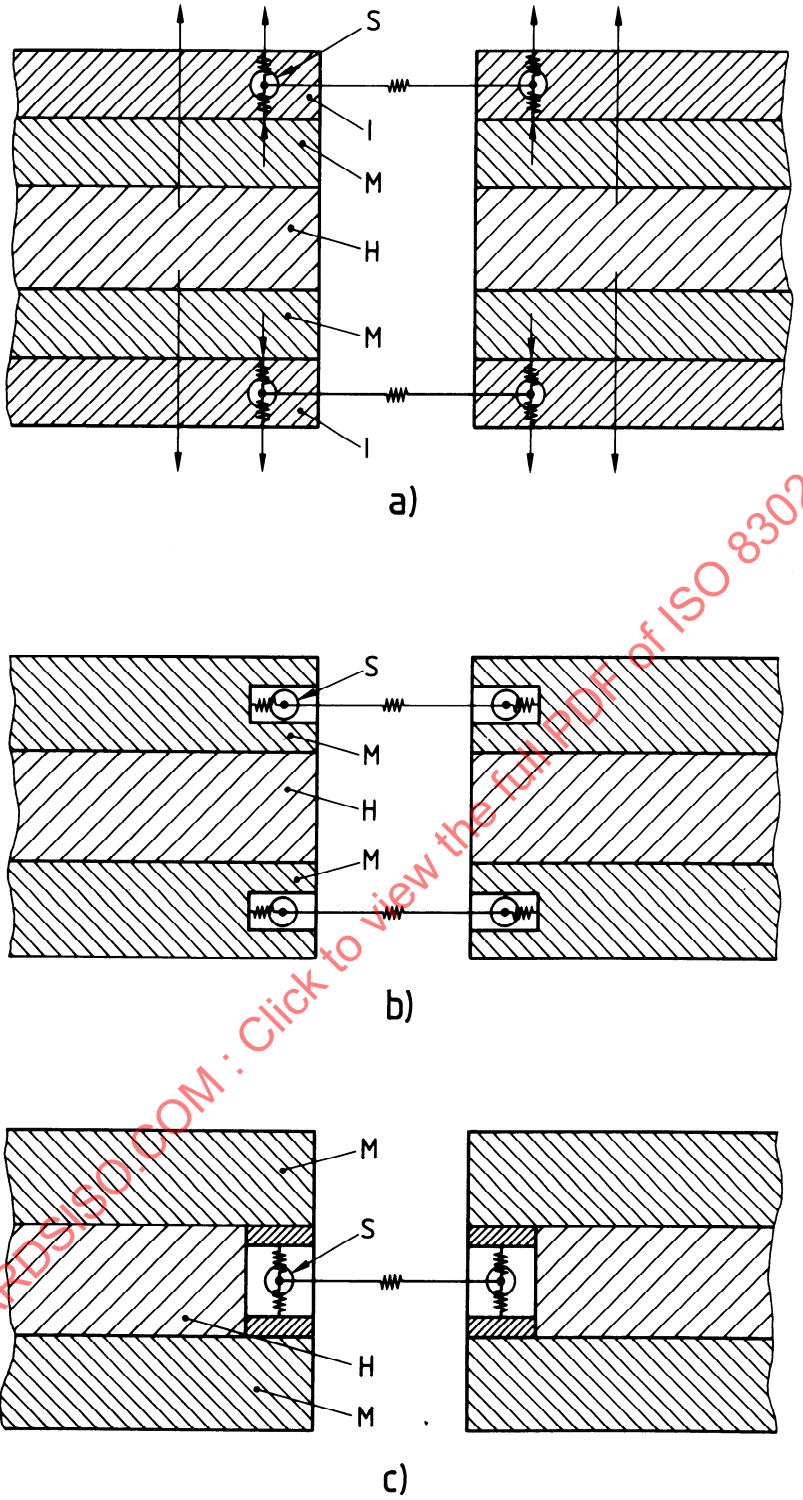


Figure 3 — Recommended positions for the imbalance sensors



Key

I Insulation due to the supporting sheets
H Heater
M Heating unit metal plate
S Imbalance sensor

Figure 4 — Imbalance sensing elements and related thermal resistances

The presence of the gap and of the mechanical connections through it create small temperature gradients in the metal plates of the heating unit in contact with the samples, and therefore the imbalance sensors shall be positioned to register the temperature imbalance existing along the edge of the gap and not the imbalance existing between some arbitrary points of the metal plates on the metering section and on the guard section. It is suggested that the distance between the gap edge and the sensor should be smaller than 5 % of the side or diameter of the metering section.

Since an uncertainty in the true temperature balance will always exist, the gap thermal resistance should be made as high as practically possible. Good general rules are that any mechanical connection between metering section and guard section should be made as small as possible, avoiding where possible, metal or continuous connections. Electrical wires should cross the gap through oblique paths and should be of thin diameter and of metal with low thermal conductivity. The use of copper should be limited to a minimum.

2.1.2 Cooling unit

The cooling units shall have surface dimensions at least as large as those of the heating unit, including the guard heater(s). They shall consist of metal plates maintained at a constant and uniform temperature, within 2 % of the temperature difference across the specimen and lower than that of the heating unit. This can be accomplished by the use of a constant-temperature fluid, by the use of electrical heaters, by the use of thermal insulation of uniform thermal resistance applied between the outermost surfaces of the heating units and appropriate auxiliary cooling plates, or by a combination of these, as suitable for the cooling unit temperature desired.

Fluid-cooled metal plates require particular care in their design in order to obtain temperature uniformity (see [5] and [24]). The temperature difference between the inlet and the outlet fluid should be evaluated for the situation of the highest thermal load in combination with a given flow-rate of the fluid. For most fluid-path layouts, this temperature difference is larger than any temperature non-uniformity of the plate. The best results will be obtained with helical counter-flow paths for the fluid. However, in this case the thermal resistance between the fluid and the metal plate should be sufficiently high (see [5] and [24]), otherwise plate temperature non-uniformity can be even larger than the temperature difference between the inlet and outlet fluid.

2.1.3 Edge insulation and edge heat losses

Deviation from one-dimensional heat flow in the specimen is due to non-adiabatic conditions at the edges of the heating unit and of the specimens. Moreover, heat losses from the edges of the heating unit and specimens will cause lateral temperature gradients in the surface plates of the guard section, thus creating additional deviation from the ideal one-dimensional heat-flow pattern intended.

Heat losses from the specimen edge cause edge loss errors which can be computed only for homogeneous isotropic opaque specimens under simplified boundary conditions. These errors will be minimal if the ambient temperature corresponds to the mean temperature of the specimens. For information on the computation, see 2.2.1 and [4], [5], [10], [11], [19], [28] and [37].

Little or no information is available on the other edge heat loss errors. The heat losses from the outer edges of the guard section and the specimen shall, therefore, be restricted. This can be accomplished by edge insulation, by controlling the ambient temperature, by an additional outer guard, by a linear gradient guard or by a combination of these methods; four possible configurations are shown in figure 5.

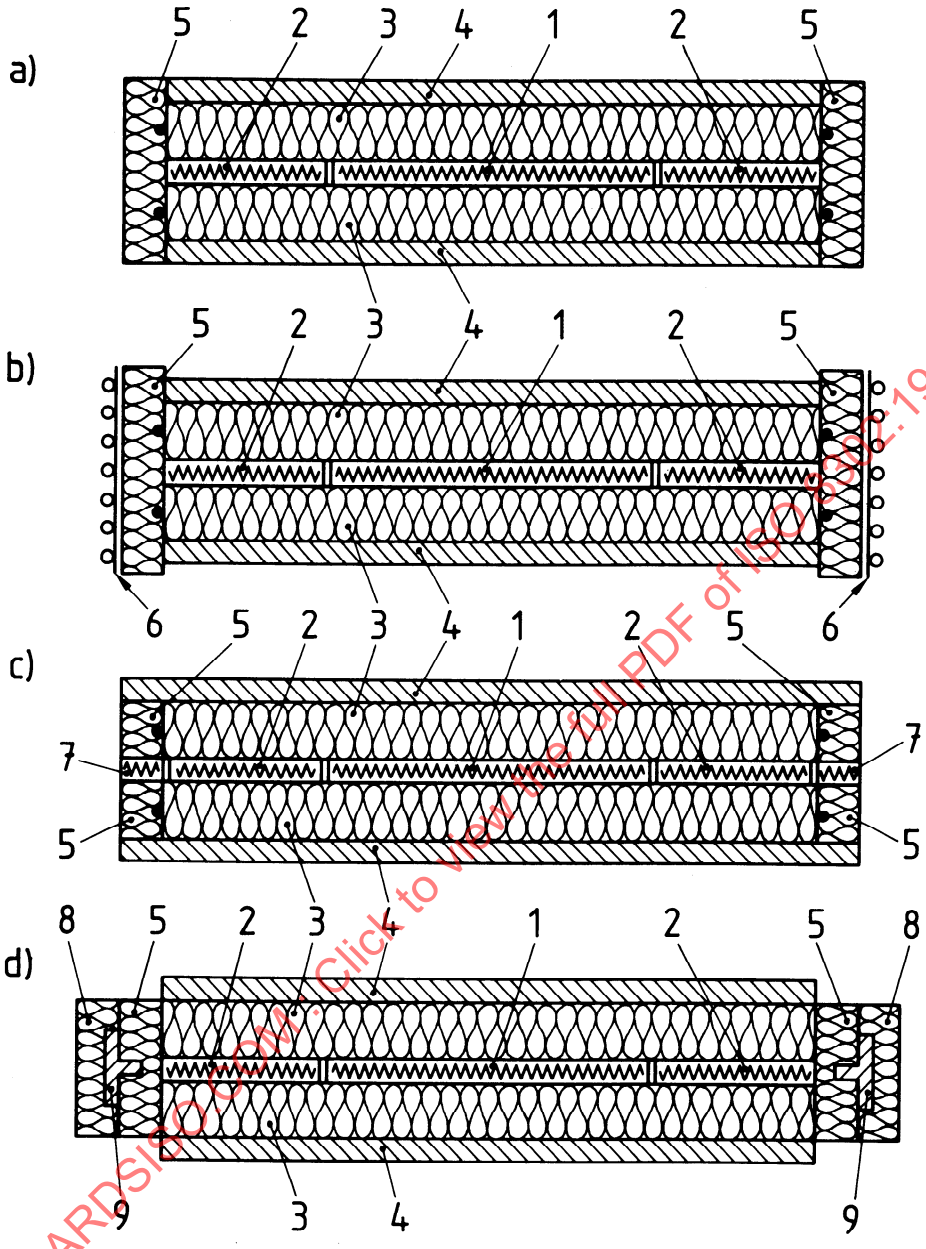
A very important heat flow path from the heating unit edges is along the wires of the heaters and temperature sensors. It is therefore necessary to provide an isothermal surface close to the heating unit and at the same temperature. All of the wires should be fastened securely to the surface. This isothermal surface may be an auxiliary guard or any other suitable surface. The level of thermal imbalance should be limited so that heat flow-rate exchanged through the wires will not exceed 10 % of the heat flow-rate crossing the samples in unidirectional ideal conditions.

2.1.4 Measuring devices

2.1.4.1 Temperature measurements

2.1.4.1.1 Imbalance detection

The temperature-imbalance sensors may be read individually and the temperature difference calculated, or, better, they may be connected differentially to indicate such temperature difference directly. Small diameter thermocouples, not greater than 0,3 mm, connected as a thermopile, are often used for this purpose. The detection system shall be sufficiently sensitive to ensure that the error in the measured property due to the gap temperature imbalance shall be restricted to 0,5 %, as determined experimentally or analytically. The sensitivity of many temperature sensors falls off drastically as the temperature is decreased. Particular care must



Key

1 Hot plate central metering section
2 Hot plate guard section
3 Specimen
4 Cooling unit
5 Edge insulation (dots are temperature sensors, when installed)
6 Additional outer isothermal guard or outer gradient guard
7 Additional outer plane guard
8 Additional outer guard insulation
9 Additional outer T-shaped guard

Figure 5 — Possible configurations to restrict edge heat losses

therefore be taken in designing thermopile measurement and control systems to operate under low temperature conditions.

2.1.4.1.2 Temperature differences in the apparatus

Any proven method capable of measuring the temperature difference between heating and cooling surfaces to an accuracy of 1 % may be used for the measurement of temperatures in the apparatus.

The surface temperatures are often measured by means of permanently mounted temperature sensors, such as thermocouples, set in grooves in the surface plates or placed just under the surface in contact with the specimen.

Other solutions, like thermocouples embedded in thin sheets, require particular care to reduce errors in the detection of surface temperatures, mainly with low resistance specimens. Some examples of thermocouple connections are shown in figure 6. Systematic errors associated with the thermocouple measurements are frequently due to the fact that their wires are not perfectly homogeneous, so that temperature differences along them generate small electromotive forces. The effect is usually larger in alloys than in pure metals. In figure 6a), each thermocouple has the reference junction in the bath R and can be read individually.

When high accuracy is required in temperature difference measurements but not in the absolute temperature of the heating and cooling units, the differential connections of figure 6b) or 6c) are two examples to reach this goal. When using differential connections, best results are obtained when the wires 1_c, 2_c, 1_h and 2_h in figure 6b) and 6c) are made of pure metals (for example: copper) and the wires connecting H_1 to C_1 or H_2 to C_2 in the same figures, remain within the cabinet A, at a temperature close to those of the heating and cooling units. In this situation, the temperature difference along the connections is kept to a minimum. On the contrary, most of the advantages are lost if the differential connection is made by clamping together leads 1 and 1′ of figure 6a).

Connections of the type shown in figure 6b) allow averaging of systematic errors of each differential measurement, while the connections illustrated in figure 6c) reduce to a minimum metallic linkages between heating and cooling units.

Temperature sensors can be either completely insulated electrically from the plates or grounded to them only in a single point of the whole circuit (as a consequence, in differential connections only one junction of a thermocouple can be grounded). The amount of electrical insulation required depends on whether the sensors are shielded by grounded metal plates of the heating or cooling units or they are only insulated from other electrical circuits; in the latter case, the insulation resistance should normally be larger than 100 MΩ. Computations and experimental verifications shall be made to be sure that other circuits do not affect the accuracy of the measurements of heat transfer properties.

The number of temperature sensors on each side shall be not less than $N\sqrt{A}$ or 2, whichever is greater, where $N = 10\ m^{-1}$ and A is the area in square metres of one face of the metering section plate.

It is recommended that one temperature sensor be placed in the centre of the metering area section and that a similar number of temperature sensors be permanently and similarly installed at corresponding positions in the facing cooling units.

2.1.4.1.3 Temperature difference across the specimen

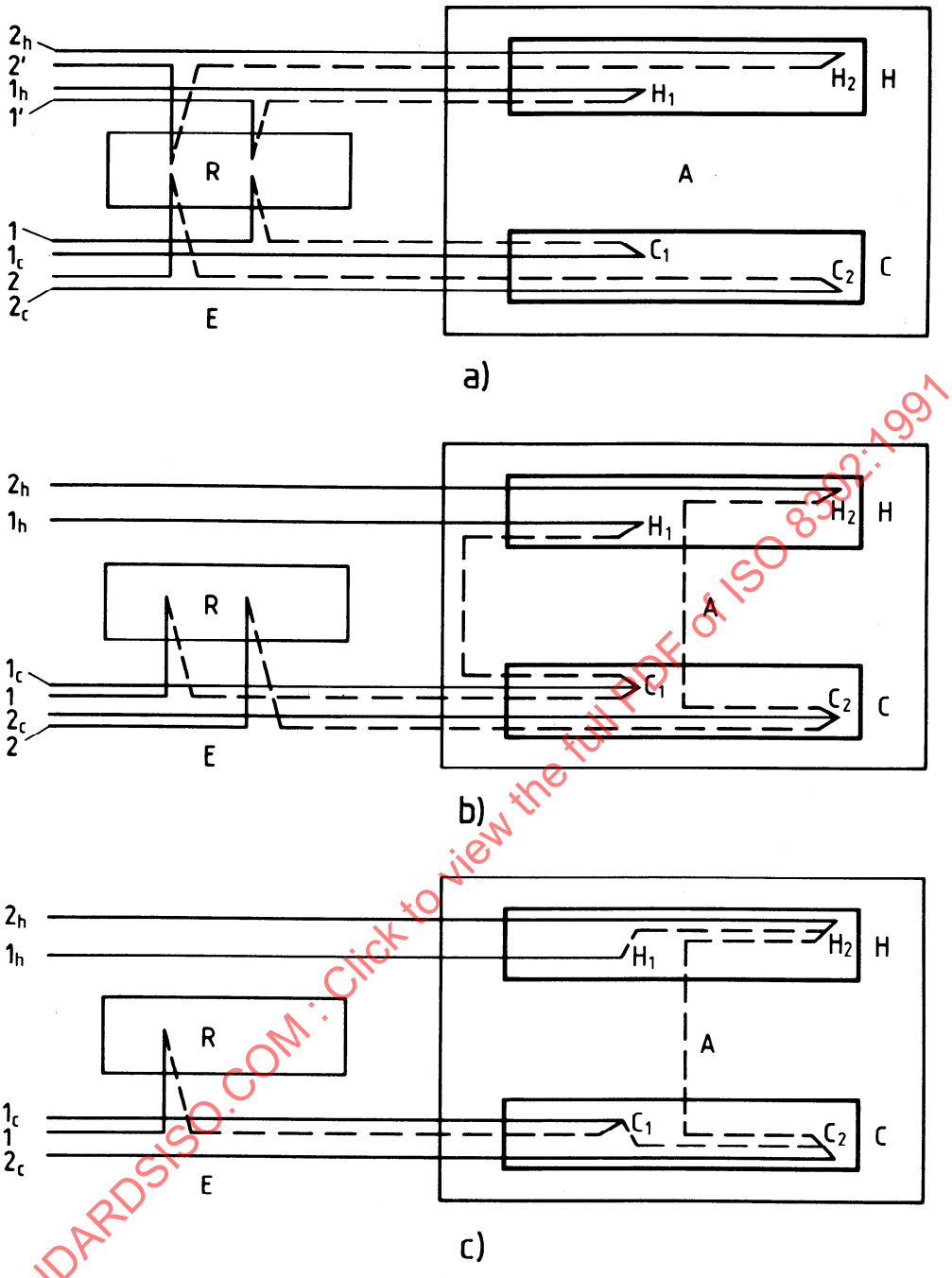
Due to possible influence of contact resistances between the specimen and the apparatus, different methods exist to determine the temperature difference across the specimen.

Some recommended techniques are as follows; errors inherent to some methods are described in [6], yet in some instances the choice of the method itself is left to the judgement of the operator.

a) For non-rigid specimens (see 1.7.1) with flat uniform surfaces that conform well to flat surfaces of the plates, and with thermal resistance greater than 0,5 $m^2 \cdot K/W$, the temperature difference across them is normally taken to be indicated by the temperature sensors, usually thermocouples, permanently mounted in the heating and cooling unit surfaces.

b) Rigid specimens (see 1.7.1) may be installed in the apparatus with thin sheets of suitable homogeneous material interposed between the specimen and each plate. The resistance of the composite sandwich (sheet/rigid specimen/sheet) is determined as in a) and the temperature difference across the specimen can be computed if the resistance of the sheets is known (see 3.2.2.2.1 for limitations of this method).

c) Another method used to determine the temperature difference across a rigid specimen is by means of separate temperature sensors, usually thermocouples, mounted flush with or interior to the surfaces of the specimen. This technique can also be used in conjunction with thin layers of low resistance material interposed between the specimen and the plates.



Key

H Heating unit
H_1, H_2 Thermocouple junctions on the heating unit
C Cooling unit
C_1, C_2 Thermocouple junctions on the cooling unit
R Reference isothermal bath, usually an ice bath
A Apparatus cabinet, usually conditioned close to the mean test temperature
E Environment, usually the laboratory air

Figure 6 — Some thermocouple layouts

2.1.4.1.4 Type and placement of temperature sensors

Thermocouples mounted in the surfaces of the plates shall be made of wire not larger than 0,6 mm in diameter and preferably not larger than 0,2 mm in diameter for smaller size apparatus. Thermocouples placed against or set into the surfaces of the specimens should be made of wire not larger than 0,2 mm in diameter. For low resistance specimens it is recommended that thermocouples be placed within the specimen surface whenever possible; otherwise thinner diameter thermocouples must be used.

The thermocouples that are used to measure the temperature of the hot and cold faces of the specimen should be fabricated from either calibrated thermocouple wire or from wire that has been certified by the supplier to be within the special limits of error given in table B.1. Thermocouples used to measure temperatures in the range from 21 K to 170 K should have a standard limit of error of $\pm$ 1 %. For information concerning the installation, sensitivity and accuracy of thermocouples in the cryogenic temperature range, see [7] to [9].

The resulting error in temperature differences due to distortion of the heat flow pattern around the temperature sensor, due to temperature sensor drift and due to other temperature sensor characteristics shall be less than 1 %.

Similar criteria shall be applied to temperature sensors different from thermocouples.

2.1.4.2 Thickness measurements

Means shall be provided for measuring the thickness of the specimen to within 0,5 %. Because of the changes of specimen thickness possible as a result of thermal expansion, or compression by the plates, it is recommended that, when possible, specimen thickness be measured in the apparatus at the existing test temperature and compression conditions. Gauging points, or measuring studs at the outer four corners of the cooling unit plates or along the axes perpendicular to the plates at their centres, will serve for these measurements. The effective specimen thickness is determined from the average difference in the distance between the gauging points when the specimen is in place in the apparatus and when it is not in place, and the same force is used to press the cooling unit plates towards each other.

2.1.4.3 Electrical measurement system

The design of the measuring system will depend on the heater design, on the type of temperature sensors used and on the temperature-difference sensing circuitry. The range of the outputs from these will vary according to the operating range of the apparatus. In all likelihood, it will vary by several orders of magnitude. This necessitates either highly linear, wide-range (multidigit) or less linear, multirange, measuring instruments. The choice will be governed by the general requirements of the user.

A measurement system having a sensitivity and accuracy of at least 0,2 % of the temperature difference across the specimen shall be used for measurement of the output of all temperature- and temperature-difference sensors. Measurement of the power to the heating unit shall be made to within 0,1 % over the full operating range.

2.1.5 Clamping force

A means shall be provided for imposing either a reproducible constant clamping force upon the system to promote good thermal contact or for maintaining accurate spacing between the plates of the apparatus.

A steady force, which will thrust the cooling units toward each other, can be imposed by means of constant-force springs, a system of levers and dead weights, or an equivalent method. It is unlikely that a pressure greater than 2,5 KPa will be required for the majority of insulating materials.

When compresible specimens are tested, it may be necessary to use stops of small cross-sectional area and low thermal conductivity between the corners of the cooling unit plates and the corners of the guard section. Other means may be used to impose the distance between heating unit and cooling unit plates; a constant pressure arrangement is not needed for such tests.

2.1.6 Enclosure

The guarded hot plate apparatus shall be placed in an enclosure equipped to maintain the desired interior environmental gas temperature and dew or condensation point when the cooling unit temperature is below room temperature or when the mean temperature is substantially above room temperature.

Means to control the environmental pressure and gas property should be provided if measurements are required in different gaseous environments.

2.2 Evaluation of errors

2.2.1 Imbalance and edge heat loss errors

Most of this error evaluation assumes conducting specimens opaque to ratiation. In case of low density materials semitransparent to radiation, some expressions may be inaccurate.

If ϕ is the heat flow-rate that in the ideal unidirectional condition would traverse the specimen (in a single-specimen apparatus) or both specimens (in a two-specimen apparatus) and ϕ_T is the actual value, the error in heat flow measurement, E_ϕ, is:

$$E_\phi = \frac{\phi_T - \phi}{\phi}$$

The heat flow-rate, ϕ, can be expressed as

$\phi = (\mathcal{T} A \Delta T)/d$ (see 3.5.2). For conducting

specimens opaque to radiation λ replaces $\mathcal{T}$.

Assuming that the metering section, the guard section and the cooling units are at the uniform temperatures, T_1, $T_1 - \Delta T_g$ and T_2 respectively, that the specimens are homogeneous and isotropic with conductivity λ and that their edges exchange heat towards a medium at the uniform temperature $T_e = T_2 + e\,(T_1 - T_2)$, where e is a dimensionless number, theoretical analysis (Bode, [28]) shows that

$$E_\phi = Z_1 + e\,Z_2 + \frac{\Delta T_g}{\Delta T} Z_3$$

where Z_1, Z_2 and Z_3 depend on the specimen dimensions, on the gap and guard section width, on the conductivity of the specimens, on the surface coefficients of heat transfer, on the edges of the specimens and on the thermal connections through the gap.

When the imbalance temperature difference through the gap, ΔT_g, is equal to zero, ϕ_T is affected only by a heat flow-rate, ϕ_e, that corresponds to edge heat loss errors E_e; as a consequence Z_3 is a parameter associated to the imbalance error E_g. The evaluation of Z_1, Z_2 and Z_3 requires very complex series expansion, yet, when the surface coefficient of heat transfer tends to infinity, the following approximate expression can be derived (see [11]):

$$E_e = \frac{\phi_e}{\phi} = Z_1 + e\,Z_2 =$$

$$= \left\{ \frac{d}{\pi l} \left[e \ln \frac{\cosh\left(\pi \frac{b+l}{d}\right) + 1}{\cosh\left(\pi \frac{b}{d}\right) + 1} + \right.\right.$$

$$\left.\left. + (1-e) \ln \frac{\cosh\left(\pi \frac{b+l}{d}\right) - 1}{\cosh\left(\pi \frac{b}{d}\right) - 1} \right] \right\}^2 - 1$$

This simplified theoretical expression gives correct results only when test conditions match the model: for example it does not work for specimens semitransparent to radiation or for non-isotropic or non-homogeneous specimens, either opaque or semitransparent to radiation. The use of the formula is suggested only to set limits on the design of the apparatus to minimize the effect of the edge heat losses but shall never be used to correct measured data.

The lowest values are obtained when the value of e is close to 0,5. However, it is difficult to maintain the specimen edges exactly at the mean test temperature which corresponds to $e = 0{,}5$, so computations should be carried out with e not greater than 0,25.

From the imbalance error $E_g = (\Delta T_g/\Delta T) Z_3$, an error heat flow-rate $\phi_g = E_g \phi$ can be expressed as

$$\phi_g = (\phi_o + \lambda c) \Delta T_g$$

where

$\phi_o \Delta T_g$ represents the heat flow-rate flowing directly across the gap due to conduction in the heater and temperature-sensor leads, in the mechanical connections, etc.;

$\lambda c\, \Delta T_g$ is the heat flow-rate through one specimen or both specimens in a two-specimen apparatus.

From the above expression, it is evident that

$$Z_3 = \frac{d}{A} \left(\frac{\phi_o}{\lambda} + c \right)$$

The coefficient c is not strictly a constant and was predicted theoretically (see [12]) as

$$c = \frac{16\,l}{\pi} \ln \frac{4}{1 - e^{-(\pi g/d)}}$$

considering, under approximate boundary conditions, the heat flow-rate through both specimens in a two-specimen apparatus.

The value of ϕ_o can be computed using elementary heat transfer formulae for any given design of the heating unit, provided the dimensions and the materials of construction are known. The values of ϕ_o and c can also be checked experimentally (see [1] and 2.4.4). In this discussion the difference in temperature imbalance around the guard-metering section gap for square apparatus as described in 2.1.1.4 has been assumed negligible; also assumed negligible are the problems due to the positioning of imbalance sensors described in 2.1.1.5. If these were not negligible, the corresponding errors should be added to E_g.

2.2.2 Errors due to non-symmetrical conditions

If the two specimens are not identical, the temperature differences may differ slightly. If it is assumed that each specimen has the same thermal conductivity and this is constant with temperature, the error $E_s = \Delta\lambda/\lambda$ due to non-symmetrical conditions can be written as follows:

$$E_s = \frac{\Delta\lambda}{\lambda} = \left(\frac{d_A - d_B}{2d} \right)^2 + \frac{(T_{1A} - T_{2A}) - (T_{1B} - T_{2B})}{2(T_1 - T_2)} \times \frac{d_A - d_B}{2d}$$

where subscripts A indicate quantities measured on the first specimen, subscripts B indicate quantities measured on the second specimen, and those without subscripts are average values.

If the thermal conductivity of each specimen is different or it is temperature-dependent, the formulae which define E_s are more complex. Similar expressions may be derived for the other heat transfer properties. E_s is negligible if the requirements of 3.2.1 and 3.3.6 are fulfilled.

2.2.3 Other errors

The measured properties are affected by other determinate sources of errors that must be considered both by the designer of apparatus and by the operator. These errors depend upon the accuracy of measurements of dimensions and electrical voltages at low levels. The major ones are

a) error in the measured electric power to the metering section, E_E;

b) error in the dimensions of the appropriate metering area for cut and uncut specimens and for the heating unit and gap dimensions, E_A;

c) errors in the value of temperature and temperature differences, E_T; these depend on the accuracy of the calibration of the temperature sensors, accuracy and noise of measuring instruments, uncertainty in the definition of the point where the temperature is measured by the sensors and uncertainties due to contact resistances between specimens and temperature sensors;

d) errors in thickness measurements, E_d; these depend on the accuracy of the instruments, on the uncertainty in defining the average thickness due to the lack of flatness of the specimens and the apparatus surfaces, and on the lack of agreement with test conditions if the thickness is not measured when the specimens are mounted in the apparatus.

2.2.4 Total error

The majority of the errors cited in 2.2.3 are systematic, and thus the total error is additive. However, the probability that they all act in one direction in the sense of increasing or decreasing the measured property (thermal conductivity thermal transmissivity or resistivity, thermal resistance, transfer factor or thermal conductance) is limited. The correct definition of the maximum probable error will require a complex statistical analysis, but if there is no one error which is far larger than all of the others, the maximum probable error ranges between 50 % and 75 % of the total error.

2.3 Apparatus design

2.3.1 Required performance

When a guarded hot plate apparatus is to be designed, preliminary decisions must be taken on the following parameters:

a) minimum and maximum specimen thickness to be tested in the apparatus;

b) minimum and maximum specimen thermal resistance;

c) minimum and maximum temperature difference across the specimen;

d) sensitivity of the balancing system of the guard section;

e) minimum cooling unit temperature;

f) maximum heating unit temperature;

g) overall apparatus accuracy as maximum acceptable error in measured property in a defined worst-case condition;

h) surrounding environment.

2.3.2 Tentative selection of apparatus dimensions

As a first trial take the side or diameter of the central metering section to be four times the maximum specimen thickness and the guard section external side or diameter to be eight times the maximum specimen thickness.

Compare these dimensions with those suggested in 1.7.9 and choose one of them.

2.3.3 Heating unit temperature uniformity

Define a tentative thickness for the metal plates of the heating unit. Large temperature non-uniformities exist in the guard section due to edge heat losses.

Compute edge heat losses ϕ_w through the wires, and edge heat losses ϕ_{el} through the side of the guard section and through the side of the specimen (see 2.1.3).

For the first case shown in figure 5, where no auxiliary guard section is used, assume surrounding insulation of uniform thermal resistance R_e where R_e is either due only to natural convection or is the insulation thermal resistance considered as a flat slab. A very rough estimation of edge heat losses ϕ_{el} can be computed as

$$\phi_{el} \approx \frac{P}{R_e}\left[\frac{y}{4}(T_1 - T_2) + \left(d + \frac{y}{2}\right)(T_m - T_a)\right]$$

where

d is the test specimen thickness, in metres;

$(T_1 - T_2)$ is the difference in temperature between the hot and cold surfaces of the specimen, in kelvins;

y is the thickness of the heating unit, in metres;

P is the perimeter of the guard section, in metres;

R_e is the minimum thermal resistance of the edge insulation, in kelvins square metre per watt;

T_m is the mean temperature of the specimens, in kelvins;

T_a is the temperature of the outer surface of the edge insulation (in practice it can be assumed as equal to the ambient temperature), in kelvins.

It should be noted that ϕ_{el} depends on both $(T_m - T_a)$ and $(T_1 - T_2)$; consequently, since it is desirable that the net heat flow-rate from the outer edges of the specimens be kept nearly equal to zero, $(T_m - T_a)$ should be kept small.

Evaluate then the deviation from isothermal conditions in the metal plates of the heating unit roughly computing the heat flow-rate $\phi_{el} + \phi_w$ in the guard section which exceeds the one that would flow in the unidirectional condition. Then, assuming that this heat flow-rate is transmitted uniformly by the guard section heater to the guard section metal plates as the density of heat flow-rate q_e and that it is exchanged only through the external guard section edges, calculate temperature non-uniformities on the metal plates (see figure 7).

Similar considerations may be used to evaluate temperature non-uniformities in the metering section and in the guard section due to the presence of the gap. This check must be carried out when the density of heat flow-rate through the heating unit is maximum; a proven design should be used when the designer has no prior experience in guarded hot plate apparatus design.

At the conclusion of the calculations, check whether the thickness of the metal plates of the heating unit is satisfactory; this thickness should not greatly exceed the minimum thickness required to reach temperature uniformity stated in 2.1.1.2, as thick plates will increase imbalance errors.

2.3.4 Cooling unit temperature uniformity

Compute the maximum heat flow-rate crossing the specimen when its thermal resistance is minimum and the temperature difference across the specimen is maximum. Add the heat flow-rate due to edge heat losses and the heat flow-rate exchanged from the cooling units towards the environment. Define the cooling system, the metal plate thickness and mass flow-rate of the cooling fluid (when pertinent) to reach the temperature uniformity stated in 2.1.2.

2.3.5 Imbalance and edge heat loss errors

Determine the maximum allowed value for $E_g + E_e$ and define a tentative gap width in accordance with 2.1.1.3.

A narrow gap increases imbalance errors while a wide gap increases uncertainties in the definition of the metering area.

Calculate the apparatus parameters ϕ_o and c as defined in 2.2.1.

Evaluate imbalance and edge heat loss errors as suggested in 2.2.1. They are maximum when the specimen thermal resistance and specimen thickness are maximum and when $(T_1 - T_2)$ is minimum.

If errors due to edge heat losses through the specimen and the guard section, as just outlined, cannot be evaluated, the heat flow-rate in the guard section must be calculated: the amount due to edge heat losses should not exceed 20 % of the heat flow-rate flowing in the specimen under ideal unidirectional conditions; see 2.3.3 for tentative calculations.

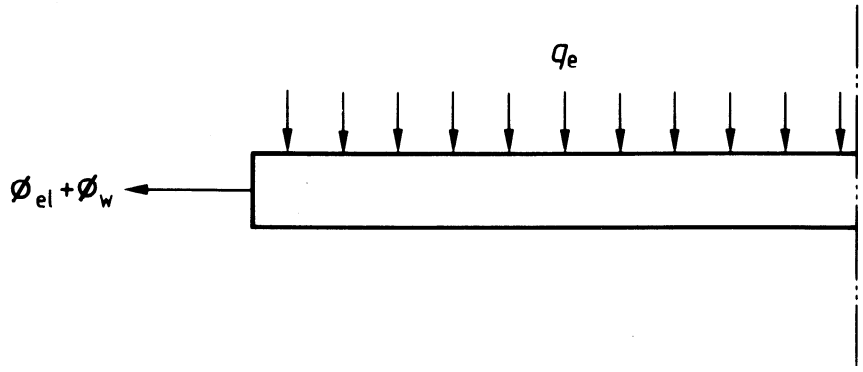


Figure 7 — Evaluating temperature uniformity in a metal plate

The imbalance error must be compatible with the sensitivity of the imbalance detecting system and should not be far larger or smaller than edge heat loss error. At this point the optimum guard section width and the maximum allowed specimen thickness should be verified (see [5]). Annex C contains a listing of a computer program that gives the maximum specimen thickness, once the sum $(E_g + E_e)$ has been defined. For a range of appropriate variables of the guarded hot plate apparatus this can be modified to evaluate the performance of any apparatus.

If the results are not satisfactory, different apparatus dimensions may be necessary or it may be necessary to add a second peripheral guard section; find a new approach and go back to the beginning of 2.3.

2.3.6 Detailed design

Once satisfactory values for the size and dimensions of the apparatus have been found

a) define surface tolerances according to minimum specimen thickness;

b) select surface finishing to reach an emissivity of 0,8 or greater (at room temperature, oxidized metallic surfaces and many paints meet this requirement);

c) define all apparatus details such as temperature sensor positions and mounting, heater layout, wiring, mechanical connections, thickness measurement devices, etc.;

d) select a cooling system according to minimum cooling unit temperature;

e) select a conditioning system according to the required surrounding environment and according to the needs of its stability and drifts to keep edge heat loss errors within the stated values;

f) select automatic temperature-control systems according to minimum temperature drifts and fluctuations acceptable by the apparatus;

g) choose the power supply for the central metering section according to the maximum power requirements stated in 2.3.1 and with drifts compatible with the minimum power corresponding to maximum specimen thermal resistance and minimum temperature difference;

h) select the electrical measuring system with a sensitivity and an accuracy in accordance with the minimum temperature difference on the apparatus.

2.3.7 Total error

Evaluate all the errors explained in 2.2, calculate the total error and compare it with the overall apparatus accuracy given in 2.3.1: the design will be successfully completed when the total error defined in 2.2.4 is smaller than the overall apparatus accuracy.

2.4 Performance check

When a new or modified design is evolved, a set of careful checks must be performed before starting regular testing.

2.4.1 Planeness

The planeness of the surface can be checked with a metal straightedge held against the surface and viewed at grazing incidence with a light behind the straightedge. Departures as small as 25 µm are readily visible, and large departures can be measured using shimstock or thin paper.

2.4.2 Electrical connections and automatic controllers

Thin, low thermal resistance specimen(s) shall be mounted in the apparatus, and the assembly allowed to reach thermal equilibrium with the air of the laboratory when the apparatus is installed. All temperature sensors shall indicate temperatures very close to the temperature of the air of the laboratory; check the noise of each sensor. Check insulation of all electrical circuits with an ohm-meter.

Compute the maximum expected voltage on the heaters of the heating unit; apply a voltage close to the one just computed between the heating unit metal plates and one lead of the heater of the metering section or of the guard section (no current should flow); if grounding, guarding and electrical insulation of the temperature sensors are correct, no change will be observed in their readings. Repeat the procedure also at the extremes of the operating temperatures of the apparatus; below room temperature, one frequent reason for the degradation of the electrical insulation is moisture. Also at high temperatures, electrical insulation can exhibit wide changes.

The next step is that of checking noise and drifts of all automatic controllers.

2.4.3 Temperature measurements

When the apparatus is enclosed in a conditioned cabinet, mount the specimen(s) in the apparatus, regulate the cooling unit temperature at some relevant value within its range. Control the ambient temperature within the cabinet at the same temperature.

Do not supply any electrical power to the heating unit central section and guard section. The heating unit temperature shall match the cooling unit temperature within the noise of the measuring system. Moreover, the guard section temperature shall also be balanced with the metering section, again within the noise of the imbalance-detection instrumentation (this isothermal configuration can also be used to check thermocouples). Wrong results can be due either to a poor design of the conditioning cabinet and of the insulation of the apparatus or to wrong wiring and connections of the temperature sensors.

2.4.4 Imbalance errors

Tests should be carried out on new equipment using different specimens and guard to metering section temperature-imbalances, to determine the maximum imbalance error for any kind of specimen (see 2.2.1; [1] to [5] and [12]). The parameter ϕ_o and c explained in 2.2.1 shall be determined as follows. A series of measurements should be carried out on a (couple of) specimen(s) of low thermal conductivity with a range of values for the temperature imbalance ΔT_g and the change of measured thermal conductivity recorded. The results should be fitted to a straight line to define $\Delta\lambda/\Delta T_g$.

The experiments and computation should be repeated for a (couple of) specimen(s) of high thermal conductivity. By means of the values $\Delta\lambda/\lambda = E_g$ and the two equations of the type $E_g = (\Delta T_g/\Delta T)Z_3$, the two unknowns ϕ_o and c can be obtained. Similar equations can be used for other measured properties.

The noise and drifts of the imbalance detection instrumentation must be smaller than the voltage corresponding to the minimum imbalance allowed in the worst test conditions.

2.4.5 Edge heat losses

Edge heat losses give rise to the greatest measurement inaccuracy when both the thickness and thermal resistance of the specimens are large and when the temperature difference across them is small.

Mount a (couple of) specimen(s) with thermal resistance and thickness close to the maximum design value. Then measure the power supplied to the guard section; it should not greatly exceed the one required by the specimen under perfect unidirectional conditions.

Checks must then be carried out to measure experimentally the effect on measured properties of the edge heat losses. The only direct method, where applicable, is to alter the ambient temperature and observe the corresponding change in guard section power and in measured thermal properties. This information is of considerable help in defining, for any type of specimen (homogeneous or not, isotropic or not, etc), the level of acceptable drifts in ambient temperature (see 2.2.1).

When it is impossible to change the ambient temperature, a useful method of determining whether or not sufficient edge guarding or insulation is present to reduce edge heat loss error in the specimen is to measure the temperature T_e using a thermocouple soldered or peened to a thin metal strip embedded into one specimen at the centre of an edge. Under these conditions, the following criterion should be met:

$$\frac{T_e - T_m}{\Delta T} < 0{,}1$$

where

T_m is the mean temperature of the specimen(s);

ΔT is the temperature difference across the specimen(s).

This method will only work for homogeneous specimens. For best accuracy, the factor should be less than 0,02.

2.4.6 Emissivity of apparatus working surfaces

If an air gap of thickness d, ranging from 5 mm to 30 mm, is created between heating and cooling unit surfaces, preventing the onset of natural convection, the density of heat flow-rate per unit temperature difference, h_t, is the sum of λ/d and $4\sigma_n T_m^3/(2/\varepsilon - 1)$ (λ is the conductivity of air and σ_n is Stefan-Boltzmann constant). The best fit of the plot of h_t versus $1/d$ supplies both the air conductivity and $4\sigma_n T_m^3/(2/\varepsilon - 1)$, hence the apparatus emissivity. When the onset of natural convection cannot be avoided, more complex procedures are required (see [21] and [38]).

2.4.7 Linearity test

When the equipment has met the design requirements throughout the checks 2.4.3 to 2.4.6, mount a specimen (or a couple of specimens) made of a thermally stable material the conductivity of which is a linear function of temperature. The Bureau Communautaire de Référence (BCR) reference material RM 64 (glass fibre board with a nominal density close to 90 kg/m^3) and the National Bureau of Standards (NBS) standard reference material SRM 1450 (glass fibre board with density ranging from 110 kg/m^3 to 170 kg/m^3) meet these requirements between 170 K and 370 K, and 255 K and 330 K respectively. Measure the conductivity at a given mean test temperature with widely different temperature differences, for example 10 K, 20 K and 40 K. The results must be independent of the temperature difference.

Repeat the check at some other relevant mean test temperatures. If the results are unsatisfactory, this is likely to be due to the combined effects of edge heat losses and bad placement of the imbalance sensors.

2.4.8 Proven performance checks

When all the other checks are successful, tests shall be made on at least two sets of material of known thermal stability which have been calibrated at a nationally recognized laboratory. Tests shall be made for each specimen at two mean temperatures typical of the operating range. All tests shall be conducted within 90 d of calibration, where possible. Any difference in results should be carefully studied to determine why they arise and how they can be removed. Appropriate action should be taken. Reports on materials tested according to this method shall be issued only after successful comparisons. No further checking will be necessary, though periodic checks are recommended.

Section 3: Test procedures

3.1 General

The measurement of the heat transfer properties of a low thermal conductance sample or of a thermal insulating material, product or system can be carried out in accordance with a set of specifications based upon this International Standard.

It is assumed that the operator is fully conversant with all of the foregoing basic principles of heat transmission and those of the design and operation of the guarded hot plate apparatus. It is further assumed that the operator can discuss their impact on the measurements with a person submitting a particular specimen or sample for test or requiring specific information on the heat transfer properties of the material, product or system.

Thus, before any measurements are undertaken, having ascertained that valid measurements are possible with guarded hot plate apparatus, a number of decisions have to be made, which relate to the specific property desired or needed as a result of any direct measurement (e.g. thermal conductivity or thermal resistance), or to any correlation desired or needed among measured properties (e.g. thermal conductivity as a function of temperature or thermal conductivity as a function of density at a given temperature).

In particular these decisions will be influenced by

a) The size and form of apparatus either available or necessary. A particular apparatus of one size may not be sufficient to carry out measurements on all specimen thicknesses to enable all of the requisite heat transfer properties to be determined directly or by interpolation from measurements on thicknesses up to its maximum limit, see 3.4.2. Similarly, the range of both temperature and environmental conditions either available or necessary may not be sufficient to yield the required information directly or by interpolation from measurements over the respective ranges available with the apparatus.

b) The size and number of specimens either provided or needed. This will depend on the ultimate requirement of a particular sample or material. If the material, product or system is highly anisotropic in nature, whether measurements are possible with the guarded hot plate apparatus should first be determined (see 3.4.1).

c) The need or desirability of interposing thin sheets of low thermal resistance between the specimen and the apparatus and the need or desirability of instrumenting the specimen with temperature sensors (thermocouples) see 2.1.4.1.3). These techniques are intended to make correct measurements of the temperature difference across the specimen of low thermal resistance and/or of rigid material. For materials, products or systems of high thermal conductance, especially when they are anisotropic in nature, in some laboratories the specimens are fabricated either in the form of central and annular sections of corresponding sizes to the metering and guard sections of the apparatus being used, or of the same size as the central section, replacing the gap and guard portions of the specimen with a suitable insulating material.

These techniques cannot be encouraged until a theoretical assessment of implied errors is available, yet in both situations the metering area, A, to be used in calculations shall be

$$A = A_m + A_g \frac{1}{2} \frac{\lambda_g}{\lambda}$$

where

A_m is the metering section area;

A_g is the gap area;

λ is the specimen conductivity;

λ_g is the conductivity of the insulating material or the conductivity of the material filling the region facing the gap.

d) The need or desirability of enclosing the specimen in thin water-vapour tight envelopes. These techniques are intended to prevent either moisture adsorption after drying or change in moisture content after conditioning.

e) The need for either specimen thickness spacers or applied pressure on the specimen.

The operator must also be conscious of the difference between a measurement the goal of which is to determine one of the steady-state heat transfer properties defined in section 1, and a measurement required by a material specification. The latter may be required by a sampling plan on specimens that do not conform to all the requirements stated in this International Standard. A typical situation is that of specimens not flat enough to ensure good contact with the apparatus, or not parallel, as required in 3.2.2.2.1, or tested at a thickness far from the end use. The numerical results of such tests must therefore be regarded merely as a convenient basis

for the acceptance or rejection of lots of a particular material, and not necessarily as a meaningful heat transfer property of the material or specimen.

3.2 Test specimens

3.2.1 Selection and size

One or two specimens shall be selected from each sample according to the type of equipment (see 1.6.2). When two specimens are required, they shall be as nearly identical as possible with thicknesses differing by less than 2 %. The specimen or specimens shall be of such size as to cover the heating unit surfaces completely, except in special applications described in 3.1 c). They shall either be of the actual thickness to be applied, or of sufficient thickness to give a true average representation of the material to be tested (see also 3.4.2). They shall also meet the general requirements outlined in 1.7 and 1.8. The relationship between the thickness of the test specimen used and the dimensions of the heating unit shall be restricted so as to limit the sum of the imbalance and edge heat loss errors to 0,5 % when using the formulae of 2.2.1, if applicable. Where the heating unit has other constructional details, a separate analysis shall be made to determine the point at which the sum of the imbalance and edge heat loss errors will be equal to 0,5 %.

3.2.2 Preparation and conditioning

3.2.2.1 Conformance to material specifications

Preparation and conditioning of the specimens shall be in accordance with the appropriate material specification. The following guidelines are given where no specification is available.

3.2.2.2 Guidelines for all specimens except loose fills

3.2.2.2.1 Preparation

The surface of the test specimens shall be made plane by appropriate means (sandpapering, face-cutting in a lathe, and grinding are often used), so that close contact between the specimens and the apparatus or interposed sheets can be effected.

For rigid materials, the faces of the specimens shall be made as flat as the heating unit (see 2.1.1.1) and shall be parallel over the total surface area within 2 % of the specimen thickness.

When the specimen is of rigid material and has a thermal resistance smaller than 0,1 $m^2 \cdot K/W$, either thin sheets [see 2.1.4.1.3 b)] or temperature sensors mounted on the specimen [see 2.1.4.1.3 c)] shall be used to determine the temperature difference across the specimen. When using the solution given in 2.1.4.1.3 b), the thermal resistance of the sheets shall not be larger than one-tenth of the thermal resistance of the specimen. The resistance of the composite sandwich (sheet/rigid specimen/sheet) will be determined using the temperature drop indicated by the permanent temperature sensors in the heating and cooling unit surface plates. The resistance of the interposed sheet alone will similarly be measured in a separate test made at the same mean temperature and with the same average thickness as when used on the surfaces of the specimens. The resistance of the rigid specimen will then be calculated from the two resistances obtained.

This procedure may allow severe errors if used without care, as the thermal resistance of the thin sheets include the contact resistances between the apparatus and the sheets, and therefore the sheet resistance cannot always be derived by the knowledge of thermal conductivity of the same material in thicker specimens. In addition, the thermal field within the sheets for the case when they are mounted within the apparatus and the specimen may be very different to that when tested alone. Thermal field differences may also be larger when the thermal conductivities of the specimens and sheets are similar and when the sheet thickness is comparable or smaller than the gap width (see thickness limits stated in 1.7.5 and 1.7.6).

When using the solution given in 2.1.4.1.3 c), the use of very thin wire or foil-type thermocouples is recommended. These must be installed either on or within the specimen surfaces and the measurement thickness must be adjusted to make appropriate allowance for the position of the thermocouples.

The method of measuring the specimen temperature difference may be subject to uncertainties difficult to evaluate, among them being the effect of distortion of heat flow lines in the immediate vicinity of the thermocouple due to its presence, the effect of imprecision in ascertaining the exact position of the effective thermocouple junctions, and the effect of local inhomogeneities in the surface of the specimen at the thermocouple junction, such as pores, voids or inclusions.

Comparison of results obtained by both methods will help to reduce measurement errors.

The number of uniformly distributed thermocouples on each side of the specimen in the area contiguous to the metering section should be not less than $N\sqrt{A}$, or 2, whichever is greater, where $N = 10\ m^{-1}$ and A is the area in square metres of one side of the metering section. If separate thermocouples are used, the effective thickness of the specimen shall be taken as the average distance, perpendicular to the faces of the specimen, between the centres of the thermocouples on the two sides.

For the type and placement of the thermocouples see 2.1.4.1.4. The use of the procedures described in 2.1.4.1.3 b) or 2.1.4.1.3 c) is recommended also when the thermal resistance of the specimen ranges between 0,5 $m^2 \cdot K/W$ and 0,1 $m^2 \cdot K/W$ or when the specimen is of a rigid material.

3.2.2.2.2 Conditioning

After the determination of the mass of the specimen(s), they shall be conditioned to constant mass in a desiccator or a ventilated oven at an appropriate temperature for the material. Thermally sensitive materials should not be exposed to temperatures that will change the specimens in an untypical manner. Where specimens are to be used in a given temperature range, they should be conditioned to constant mass at the upper limit of this range, in a non-stagnant, controlled environment.

The system may be closed if an absorber or adsorber is used. One example is a sealed desiccator at 330 K to 335 K with stirred air for conditioning of certain foam plastics.

A relative loss of mass is calculated from the mass determined before and after the drying. When the time required to carry out the measurements of the heat transfer properties is short compared with the time required by the specimen to absorb significant quantities of moisture from the laboratory air (for example, concrete specimens), it is suggested that the specimens be mounted quickly in the apparatus at the end of the drying period to prevent moisture pick-up. In the opposite situation (for example, when testing specimens of light density fibrous materials or of plastic foams), it is suggested that the conditioning be continued by leaving the specimens in a room at the standard laboratory atmosphere (temperature of 296 K $\pm$ 1 K, and relative humidity of 50 % $\pm$ 10 %) to reach equilibrium with the room air (constant mass). In intermediate situations (for example with some high density fibrous materials), the judgement of the conditioning procedure is left to the experience of the operator.

To reduce testing time, the specimen(s) may be conditioned to the mean test temperature immediately prior to being placed in the apparatus. To prevent moisture migration to or from the specimen during the test, the specimen itself may be enclosed in a vapour-tight envelope. If the presence of the envelope introduces significant thermal resistances between the specimen and the apparatus, the envelope must be treated as the thin sheets used to test rigid specimens, as described in 2.1.4.1.3.

3.2.2.3 Guidelines for loose-fill materials

3.2.2.3.1 General

When testing loose-fill materials it is recommended that the thickness of the specimen should be at least 10 times and whenever possible 20 times the mean dimension of the beads, grains, flakes, etc. of the loose-fill material. Most critical conditions are those when beads, grains, etc. are rigid. When the requirement cannot be fulfilled, alternate test methods such as the guarded or calibrated hot box should be considered. To prepare the specimen(s) it is recommended that a representative portion, slightly greater than the amount needed for the test, be taken from the sample and weighed before and after it has been conditioned as in 3.2.2.2.2, where applicable.

From these masses, the percentage mass loss is calculated. An amount of the conditioned material is weighed out such that it will produce one (two) specimen(s) of the desired as-tested density using the procedure described in the material specification; or, where no specification exists, either method A or B given below.

As the ultimate volume of the specimen is known, the required mass can be determined. The specimens are then quickly mounted in the apparatus or left to reach equilibrium with the standard laboratory atmosphere, according to the guidelines given earlier. When method A is used, or method B with covers of insignificant thermal resistance, the specimen surface temperatures should be taken as equal to those of the surface of the heating and cooling unit plates.

3.2.2.3.2 Method A

This method is suggested when operating the apparatus in the vertical position.

Set up the guarded hot plate apparatus with the required spacings between the heating unit and the cooling unit(s). Place low conductivity material that is suitable for confining the sample around or between the outer edges of the guard section and the cooling unit(s) in such a manner that it forms one (two) box(es) open on the top (one on either side of the heating unit).

Divide the weighed, conditioned material into four (eight) equal portions (four for each specimen). Place each portion in turn in (each of) the (two) specimen space(s), vibrating, packing, or tamping each portion in position until it occupies its appropriate one-quarter volume of the space, and taking care to produce specimen(s) of uniform density

3.2.2.3.3 Method B

This method is suggested when operating the apparatus in the horizontal position.

Use a (two) shallow box(es) of thin-walled low conductivity material having outside dimensions the same as those of the heating unit. The box edges shall be of such width as to make the depth of the box equal to the thickness of the specimen to be tested. Make covers for the open faces of the box(es) using either thin sheet plastic material not more than 50 µm thick or heat-resistant and non-reflective sheets (asbestos paper or other suitable uniform sheet material), these to be glued or otherwise fastened to the edges of the box(es).

The total hemispherical emittance of the surfaces seen from the specimen must be 0,8 or greater at operating temperatures. If the covers have significant thermal resistance, the method of determining the net specimen thermal resistance presented in 3.2.2.2 for rigid specimens can be used. (Divide the weighed conditioned material into two equal portions, one for each specimen). With one cover in place, and with the box(es) lying horizontally on a flat surface, place a (one) portion in the (each) box, taking care to produce a (two) specimen(s) of (equal and) uniform density throughout. Then apply the remaining cover(s), to make closed specimen(s) that can be put into position in the guarded hot plate apparatus.

Fluff compressible materials during placement so that the covers bulge slightly to make good contact with the plates of the apparatus at the desired density. For some materials, material loss during preparation of the specimen may necessitate reweighing before test, in which case determine the mass of the conditioned box and covers after the test to compute the as-tested density of the material.

3.3 Test method

3.3.1 Mass

Just before mounting the specimen(s) in the apparatus, determine its mass with an accuracy better than 0,5 %.

3.3.2 Thickness and density

The as-tested thickness (and consequently the as-tested volume) is either the thickness imposed by positioning the heating and the cooling unit or the thickness of the specimen(s) as measured at the beginning of the test.

Specimen(s) thickness can be measured either as indicated in 2.1.4.2 or outside the apparatus with instrumentation that will reproduce the pressure on the specimen during the test. From these data and the mass of the conditioned specimen determined as in 3.3.1, the as-tested density can be computed.

Blankett- or batt-type materials are usually tested at imposed thickness; material specifications define this thickness for many materials but sometimes the result of the test may not meaningfully describe the heat transfer property of the material, as pointed out in 3.1.

With some materials (for example light-density fibrous materials) it may be more accurate to measure the density of the portion of the specimen bounded by the metering area rather than the density of the full specimen; this helps to obtain a more correct correlation between density and measured heat transfer properties.

Whenever possible monitor the thickness during the test.

When the procedure of 2.1.4.1.3 c) is used, the thickness to be used to evaluate heat transfer properties must be adjusted making appropriate allowance for the position of the thermocouples.

3.3.3 Temperature difference selection

Select the temperature difference to be in accordance with one of the following:

a) The requirements of a particular material product or system specification.

b) The conditions of use for the particular specimen or sample being evaluated. [If this implies very low temperature differences, the accuracy required for measuring this quantity may be lowered. If this implies large temperature differences, it may be impossible to predict edge heat loss and imbalance errors, as theoretical evaluations assume specimens with thermal conductivity independent of temperature (see 2.2.1)].

c) As low as possible, for example 5 K to 10 K, when determining an unknown relationship between temperature and heat transfer properties.

d) The lowest temperature difference compatible with the accuracy required for the measurement of this quantity, when mass transfer within the specimen(s) is to be reduced to a minimum; this may imply non-compliance with this International Standard, as mentioned in 1.7.3.

3.3.4 Ambient conditions

3.3.4.1 Air relative humidity

When heat transfer properties are desired for the situation in which the specimen is immersed in air

(or some other gas) adjust the humidity of the atmosphere surrounding the guarded hot plate apparatus during a test to a dew-point temperature at least 5 K below the cooling unit temperature.

For inter-laboratory comparisons, it is suggested to use as standard atmosphere the one with the dew-point temperature between 5 K and 10 K below the cooling unit temperature.

When enclosing the specimen in a vapour-tight envelope to prevent moisture migration to or from the specimen, the test conditions must be such that no water condensation will take place on the portion of the envelope in contact with the cold side of the specimen.

3.3.4.2 Tests with other gases or in vacuum

For operation at cryogenic temperatures, the apparatus with the specimen(s) mounted should be purged with a dry gas prior to cooling. At temperatures between 77 K and 230 K, use dry gas rather than air as the atmosphere, and place the apparatus in a sealed cabinet. If nitrogen is used at cold unit temperatures below 125 K, take care to adjust nitrogen pressure so as to avoid condensation. At temperatures between 21 K and 77 K, gases with lower condensation temperatures such as helium are normally required as the atmosphere of the sealed cabinet. Sometimes dry hydrogen is used.

CAUTION — Hydrogen is a colourless, odourless and highly flammable gas and must be handled only by qualified and experienced personnel.

The thermal conductivity of air, nitrogen, hydrogen and helium are quite different and will therefore have a significant effect on the thermal transmission properties of the materials being tested. Care should be taken to record the type of ambient gas, its pressure and temperature, and to include this information in the report.

When heat transfer properties are desired for the situation in which the specimen is *in vacuo*, evacuate the system prior to cooling.

3.3.5 Heat flow-rate measurements

Measure the average electrical power supplied to the metering section to an accuracy of not less than 0,2 %; d.c. current is strongly recommended. With d.c. current, potentiometric four wire measurements of current and voltage are normally used.

Automatic regulation of the input power is recommended. Random fluctuations or changes in input power shall be less than that required to cause the temperature of the heating unit surfaces to fluctuate or to change in the test period by more than 0,3 % of the temperature difference between the heating and cooling units.

Adjust and maintain the power input to the guard section, preferably by automatic control, to obtain the degree of temperature balance between the metering and guard section that is required for conformance to 2.1.4.1.1.

3.3.6 Cold surface control

When a two-specimen apparatus is used, adjust the cooling units or cold surface heaters so that the temperature difference through the two specimens does not differ by more than 2 %.

3.3.7 Temperature difference detection

Determine the heating and cooling unit temperature, the specimen surfaces temperature [if the procedure in 2.1.4.1.3 c) is used] and the centre-to-guard temperature balance by proven methods having sufficient precision and accuracy to meet all the requirements given in this method.

The temperature difference across the specimen is determined by one of the procedures described in 2.1.4.1.3.

Procedure 2.1.4.1.3 b) requires also additional resistance determinations to be made on the thin sheets.

3.3.8 Settling time and measurement interval

As the principle of the method assumes steady-state conditions, to attain a correct value for properties it is essential to allow sufficient time for the apparatus and specimen to attain thermal equilibrium.

In measurements on good insulators having low thermal capacity and for cases where there is moisture absorption or desorption with consequent latent heat exchange, the internal specimen can require a very long time to attain thermal equilibrium.

The time required to reach equilibrium can vary from minutes to days and will depend on the apparatus, on the specimen, and on their interactions.

The following items must be critically considered to evaluate this time:

a) thermal capacities and control system of the cooling unit(s), of the heating unit metering section, and of the heating unit guard section;

b) insulation of the apparatus;

c) thermal diffusivity, water vapour permeability and thickness of the specimen;

d) test temperatures and environment during test;

e) temperature and moisture contents of the specimen(s) at the beginning of the test.

Operation in vacuum may also greatly increase the time required for the apparatus and specimen to reach thermal equilibrium (due to outgassing of the apparatus and specimen, and to the thermal diffusivity of the specimen being frequently low in such tests).

The effect of some of these are discussed in [18] and [20].

As a general guideline, control systems can strongly reduce the time to reach the thermal equilibrium, but little can be done to reduce the time to reach the moisture-content equilibrium.

Where a more accurate estimate of settling time is not possible, or where there is no test experience on similar specimens in the same apparatus at the same test conditions, compute the following time interval Δt:

$$\Delta t = (\rho_p c_p d_p + \rho_s c_s d) R$$

where

ρ_p is the density of the heating unit metal plate;

c_p is the specific heat of the heating unit metal plate;

d_p is the thickness of the heating unit metal plate;

ρ_s is the density of the specimen;

c_s is the specific heat of the specimen;

d is the thickness of the specimen;

R is the thermal resistance of the specimen.

Make observations as in 3.3.5 and 3.3.7 at intervals equal or larger than the time interval Δt until four successive sets of observations give thermal resistance values which do not differ by more than 1 % and are not changing monotonically in one direction. Continue these observations until at least 24 h have elapsed since the beginning of the steady-state conditions so defined whenever an accurate estimate of settling time is not possible or whenever there is no test experience on similar specimens in the same apparatus at the same test conditions.

It may be helpful, to check the attainment of steady-state conditions, to record temperature differences and/or voltage or current through the metering section heater when its temperature is automatically controlled.

3.3.9 Final mass and thickness measurements

Upon completion of the observations in 3.3.8, measure the mass of the specimen(s) immediately. It is also strongly recommended that the operator should repeat the thickness measurement and report any specimen volume change.

3.4 Procedures requiring multiple measurements

3.4.1 Procedures to assess specimen homogeneity

One way to try to estimate the error due to non-homogeneity is to compare the results for two specimens from the same sample, selected so that they have as widely different a structure near the edges of the metering area. If the two extremes cannot be identified, a number of specimens may have to be tested.

When the variations in structure occurs over small distances, it may be possible to use a single specimen cut larger than the apparatus. This over-size specimen is tested twice, in each case with the specimen carefully positioned so that the edges of the metering area are exposed to the two extremes in structure. The two results are then compared and the difference credited to distortion. The portion of the specimen(s) protruding from the apparatus should be well insulated in the two tests to reduce the possibility of the exposed section increasing edge losses. The size and thickness of the specimen affects the size of the variations in structure than can be accommodated. The larger the metering area, the smaller the effect on the results. The effect of distortion may either increase or decrease with specimen thickness.

When direct thermal short circuits exist between the surfaces of the specimens, the effect can best be identified by breaking the thermal paths, especially when the connection surfaces can be disconnected from the rest of the path. Sheets of thermally insulating materials can be used at the critical surfaces to provide the break.

Sheets made of finely ground cork, or a similar material 0,002 m or more thick, work well. The surfaces must be ground to the same degree of flatness as the heating unit (see 2.1.1.1). The thermal resistance of these sheets can be determined in separate measurements.

It is difficult to assess accuracy for these test conditions. It is not practical to assess homogeneity up to a level comparable to the accuracy of the method; detected differences shall have a physical meaning and shall not be just measurement errors.

The net change in thermal resistance of the specimen, due to thermal shorting, can thus be determined. If greater than 1 %, another measurement should be made with thicker sheets interposed.

It is also possible to evaluate the effect of thermal field distortion through the use of analysis and computation. Reference to the particular method or methods used to determine these effects should be given in the report. Differences in measurements of the heat transfer properties of less than 2 % may be considered insignificant for the purposes of this International Standard.

3.4.2 Procedure to determine the minimum thickness for which heat transfer properties of the material may be defined

Select a sample uniform in density and density distribution, with the thickness d_5 equal to the greatest thickness of the material to be characterized or equal to the maximum allowable thicknes for the test apparatus.

Cut five sets of specimens from the sample ranging in thickness from the smallest likely to be used in practice in approximately equal increments. The set of specimens shall be designated s_1 to s_5 according to their respective thicknesses d_1 to d_5.

For very low density materials, density gradients can exist, due to the mass of the specimen itself; check uniformity also with reference to this parameter.

For low density materials where heat is transferred by radiation and conduction mechanisms and where the absence of convection has been verified, the slope of a plot of thermal resistance versus thickness will very frequently diminish up to 1 cm to 2 cm and then will remain constant as the thickness increases. The reciprocal of this constant slope is the thermal transmissivity to be assigned to high thickness specimens.

Measure the thickness and thermal resistance of s_1, s_3 and s_5 at the same mean temperature and with the same temperature difference across the specimen. Plot the thermal resistance versus thickness. If these three values differ from a straight line relationship by less than $\pm$ 1 %, the slope of the straight line shall be computed. If the three values differ by more than 1 %, then similar measurements shall be made on s_2 and s_4 to check if there is a thickness above which the thermal resistance does not differ from a straight line by more than 1 %.

If this thickness exists, the slope of the straight line shall be determined to compute a thermal transmissivity $\lambda_t = \Delta d/\Delta R$ defined as the ratio between the increments of thickness, Δd, and increments of the thermal resistance, ΔR.

The thickness at which this occurs will vary according to the densities, types and forms of different materials, products and systems for different mean temperatures.

Thermal transmissivity then characterizes the material, product or system for thicknesses above which the transfer factor differs by less than 2 % from λ_t.

Allowance for experimental errors must be made in the interpretation of results. Least-square curve fitting of R versus d may also help. A larger number of specimens may be used where greater definition is required.

Thickness dependence may be a function of temperature difference across the specimens. For the purposes of this method, the above checks, if performed at typical operating temperature differences, shall be adequate to indicate the degree of thickness dependence.

3.4.3 Procedure to determine dependence on temperature difference

If the temperature-difference dependence of the heat transfer properties is not known for a material, a minimum of three measurements is necessary. These are made with widely differing temperature differences. A second-order dependence can be revealed by these measurements. When a simple linear relationship is known to occur, only two measurements, that is, one extra, need be made. This establishes the linear dependence for that particular specimen.

3.5 Calculations

3.5.1 Density and mass changes

3.5.1.1 Densities

Calculate the density ρ_d and/or ρ_s of the conditioned specimen as tested, as follows:

$$\rho_d = \frac{M_2}{V}$$

$$\rho_s = \frac{M_3}{V}$$

where

ρ_d is the density of the dry material as tested, in kilograms per cubic metre;

ρ_s is the density of the material after a more complex conditioning procedure (very frequently up to the equilibrium with the standard laboratory atmosphere), in kilograms per cubic metre;

M_2 is the mass of the material after drying, in kilograms;

M_3 is the mass of the material after a more complex conditioning procedure, in kilograms;

V is the volume occupied by the material after drying or conditioning, in cubic metres.

3.5.1.2 Mass changes

Calculate the relative mass change of the material as received due to the drying, m_r, or due to a more complex conditioning procedure, m_c:

$$m_r = \frac{M_1 - M_2}{M_2}$$

$$m_c = \frac{M_1 - M_3}{M_3}$$

where

M_1 is the mass of the material in as-received condition, in kilograms;

M_2 and M_3 are as defined in 3.5.1.1.

When required by the specifications, or when it is considered useful to evaluate the test conditions correctly, besides m_c, calculate the following relative mass change m_d due to the conditioning after the drying:

$$m_d = \frac{M_3 - M_2}{M_2}$$

Calculate the relative mass regain, m_w, of the specimen during the test, in relation to the mass immediately before the test, with the equation:

$$m_w = \frac{M_4 - M_5}{M_5}$$

where

M_4 is the mass of material in the specimen immediately after the test, in kilograms;

M_5 is the mass of dried or conditioned material in the specimen immediately before the test (it is either $M_5 = M_2$ or $M_5 = M_3$), in kilograms.

3.5.2 Heat transfer properties

To make all the computations, use average values of the observed steady-state data. The four sets of observations described in 3.3.8 shall be used in the computations; other sets of observations during the steady-state can be used as long as the heat transfer properties derived from each of these sets do not differ by more than 1 % from those derived from the four sets described in 3.3.8.

Compute the thermal resistance, R, in square metres kelvin per watt, using the following formula:

$$R = \frac{T_1 - T_2}{\phi} A$$

or the transfer factor, $\mathcal{T}$, in watts per metre kelvin, using the following formula:

$$\mathcal{T} = \frac{\phi d}{A (T_1 - T_2)}$$

where

ϕ is the average power supplied to the metering section of the heating unit, in watts;

T_1 is the average specimen(s) hot side temperature, in kelvins;

T_2 is the average specimen(s) cold side temperature, in kelvins;

A is the metering area as defined in 1.7.6 and 2.1.1.3 , in square metres. For two-specimen apparatus, the metering area defined in 1.7.6 and 2.1.1.3 must be multiplied by two;

d is the average specimen(s) thickness, in metres.

If conditions described in 1.8.2 and 1.8.3 are applicable, compute either thermal transmissivity, λ_t, or thermal conductivity, λ (or thermal resistivity, $r = 1/\lambda$), using the following formula:

$$\lambda_t \text{ or } \lambda = \frac{\phi d}{A (T_1 - T_2)}$$

where ϕ, A, T_1, T_2 and d are as defined above.

3.6 Test report

If results are to be reported as having been obtained by this method, then all requirements laid down for this method shall be met. Where such conditions are not met, a statement of compliance should be added, as required in 3.6.19.

The report of the results of each test shall include the following (the numerical values reported shall represent the average values for the two specimens as-tested or the value of a specimen for single-specimen apparatus).

3.6.1 Name and any other pertinent identification of the material, including a physical description supplied by the manufacturer.

3.6.2 Description of the specimen and its relationship to the sample, supplied by the operator. Conformance to a material specification where applicable. Method of specimen preparation for loose-fill materials.

3.6.3 Thickness of the specimens in metres, specifying if either imposed or measured. Criteria to define the imposed thickness.

3.6.4 Method and temperatures of conditioning.

3.6.5 Densities of the conditioned material as tested, in kilograms per cubic metre.

3.6.6 Relative mass changes during drying and/or conditioning (see 3.5.1).

3.6.7 Relative mass change during test (see 3.5.1). Observed thickness (and volume) changes during test (see 3.3.9).

3.6.8 Average temperature difference across the specimen(s) during the test and procedures for its determination (see 2.1.4.1.3), in kelvins or degrees Celsius.

3.6.9 Mean temperature of test, in kelvins or degrees Celsius.

3.6.10 Density of heat flow-rate through the specimen(s) during test, in watts per square metre.

3.6.11 Thermal resistance, in square metres kelvin per watt or transfer factor in watts per metre kelvin of the specimen(s). Where applicable, the thermal resistivity, in metres kelvin per watt, thermal conductivity or thermal transmissivity in watts per metre kelvin, and range of thicknesses for which these values have been measured or are known to apply (see 3.4.2).

3.6.12 Date of completion of the test; duration of the full test and of the steady-state part of the test if such information can help in interpreting results.

3.6.13 Orientation of the apparatus; vertical, horizontal, or any other orientation. In the case of single specimen apparatus, the position of the hot side of the specimen when not vertical: top, bottom or any other position.

3.6.14 For tests made using sheet material interposed between the specimen and the apparatus surfaces or for tests made using water-vapour tight envelopes, information shall be given on the nature and thickness of the sheet material or of the envelope.

Information shall be given on the type and arrangement of temperature sensors (when used) to determine the temperature difference across the specimen.

3.6.15 Type of guarded hot plate apparatus used, with one or two specimens. Method to reduce edge heat losses. Ambient temperature of the environment surrounding the apparatus during test.

3.6.16 Type and pressure of gas surrounding the specimen and type of gas used for purging, if any.

3.6.17 A graphical representation of the results in the reports shall be given when pertinent. This shall consist of a plot of each value of the thermal properties obtained versus the corresponding mean temperature of test, plotted as ordinates and abscissae respectively. Plots of thermal resistance or transfer factor as a function os specimen thickness are also very useful.

3.6.18 The inclusion within the report of a statement on the maximum expected error in a measured property is strongly recommended. When one or more of the requirements stated in this International Standard is not fulfilled (see also 3.6.19 on the statement of compliance), it is suggested that a complete estimation of the errors in measured property be included in the report.

3.6.19 Where circumstances or requirements preclude complete compliance with the procedure of the test described in this International Standard, agreed exceptions may be made, but must be specifically explained in the report. A suggested wording is: "This test conformed with all requirements of Standard Test Method ISO 8302 with the exception of ... (a complete list of the exceptions follows)."

Annex A
(normative)

Limit values for apparatus performance and testing conditions

Clause	Description	Value
1.1	Minimum measurable thermal resistance in a guarded hot plate apparatus	0,1 $m^2 \cdot K/W$
1.1	Minimum measurable thermal resistance in a guarded hot plate apparatus accepting derated accuracy	0,02 $m^2 \cdot K/W$
1.5.3	Expected guarded hot plate method accuracy (at room temperature)	2 %
1.5.3	Expected guarded hot plate method accuracy (full temperature range)	5 %
1.5.3	Expected reproducibility (specimen removed and mounted again)	1 %
1.7.1	Maximum thermal resistance for rigid specimens requiring special techniques to measure surface temperatures	0,1 $m^2 \cdot K/W$
1.7.3	Lower limit for temperature differences measured differentially	5 K
1.7.3	Lower recommended limit for temperature differences	10 K
1.7.6	Minimum specimen thickness related to gap width	10 times
1.7.9	Suggested apparatus sizes	0,3 m; 0,5 m
1.7.9	Suggested apparatus size (only for homogeneous materials)	0,2 m
1.7.9	Suggested apparatus size (only to assess thickness effect)	1 m
1.8.2	Maximum size for inhomogeneities related to specimen thickness	1/10
1.8.2	Maximum ratio of thermal conductivity in the directions perpendicular and parallel to specimen thickness in anisotropic specimens	2
1.8.3.1	Limit for transfer factor changes with thickness to assign thermal transmissivity to the material	2 %
2.1.1.1	Maximum departure from a plane of an apparatus surface or of the surfaces of rigid specimens	0,025 %
2.1.1.2	Required heating unit temperature uniformity related to temperature difference through the specimen	2 %
2.1.1.2	Maximum temperature difference between the average temperature of the opposite surfaces of the heating unit	0.2 K
2.1.1.2 ; 2.3.6; 3.2.2.3.3	Minimum total hemispherical emittance for any surface in contact with the specimen	0,8
2.1.1.3	Maximum gap area related to the metering section area	5 %
2.1.1.5	Maximum distance of imbalance sensors from the gap, related to the side or diameter of the metering section	5 %
2.1.2	Required uniformity and stability of the cooling unit temperature related to the temperature difference across the specimen	2 %
2.1.3	Maximum heat flow-rate through the wires, related to heat flow-rate through the specimen	10 %
2.1.4.1.1	Suggested maximum diameter for thermocouples to detect imbalance	0,3 mm
2.1.4.1.1	Maximum allowed imbalance error	0,5 %
2.1.4.1.2	Required accuracy in the measurement of temperature difference between heating and cooling unit	1 %
2.1.4.1.2	Minimum electrical resistance between unshielded temperature sensors and apparatus metal plates	100 MΩ
2.1.4.1.2	Minimum number of temperature sensors on each side of the metering section (whichever is greater)	$10\sqrt{A}$ or 2

Clause	Description	Value
2.1.4.1.3	Minimum thermal resistance for non-rigid specimens to use permanently mounted temperature sensors to measure the temperature difference across the specimen	0,5 $m^2 \cdot K/W$
2.1.4.1.4	Maximum thermocouple diameter when mounted in the surface of the plates to measure temperature differences between heating and cooling units	0,6 mm
2.1.4.1.4	Suggested maximum thermocouple diameter when mounted as above in the surface of small size plates	0,2 mm
2.1.4.1.4	Suggested standard errors for thermocouples	See table B.1
2.1.4.1.4	Suggested standard error for thermocouples between 21 K and 170 K	1 %
2.1.4.1.4	Maximum resulting error in the measurement of temperature differences	1 %
2.1.4.2	Required accuracy in the measurement of specimen thickness	0,5 %
2.1.4.3	Required accuracy of electrical measurements on temperature sensors, related to the temperature difference across the specimen	0,2 %
2.1.4.3	Required accuracy in the measurement of electrical power	0,1 %
2.1.5	Maximum suggested apparatus pressure on the specimen for most insulating materials	2,5 kPa
2.2.4	Maximum probable error as percentage of total error	50 % to 75 %
2.4.5	Maximum ratio between the edge to mean specimen temperature difference and temperature difference through the specimen (for best accuracy)	0,1 (0,02)
3.2.1	Maximum thickness difference for two specimens to be mounted in a two-specimen apparatus	2 %
3.2.1	Maximum value for the sum of imbalance and edge heat loss errors	0,5 %
3.2.2.2.1	Maximum deviation from parallel planes for specimen surfaces, related to specimen thickness	2 %
3.2.2.2.1	Maximum resistance of interposed sheets with respect to the specimen resistance	0,1
3.2.2.2.1	Minimum resistance for rigid specimens to measure temperature difference through apparatus thermocouples (recommended for specimen resistances between 0,1 $m^2 \cdot K/W$ and 0,5 $m^2 \cdot K/W$ and for rigid specimens)	0,1 $m^2 \cdot K/W$
3.2.2.2.1	Minimum number of thermocouples on each side of the specimen (whichever is greater of the two criteria)	$10\sqrt{A}$ or 2
3.2.2.3.1	Minimum suggested ratio between specimen thickness and mean dimension of beads, grains, flakes, etc.	10, better 20
3.2.2.3.3	Maximum specimen thickness for plastic sheets in method B for loose-fill materials	50 µm
3.3.1	Required accuracy in the determination of specimen mass	0,5 %
3.3.4.1	Minimum required difference between air dew point and cooling unit temperature	5 K
3.3.4.1	Suggested range for the above difference in inter-laboratory comparisons	5 K to 10 K
3.3.5	Accuracy in the measurement of average electrical power supplied to the metering section	0,2 %
3.3.5	Maximum allowed temperature fluctuations of the heating unit (related to the temperature difference between heating and cooling units) due to fluctuations of input power	0,3 %
3.3.6	Maximum difference between the temperature differences through the two specimens in a two-specimen apparatus	2 %
3.3.8	Maximum resistance change in four successive sets of observations to assess steady-state attainment	1 %
3.3.8	Minimum time elapsed since steady-state beginning, for unknown testing conditions, to complete observations	24 h
3.4.1	Change in thermal resistance in specimens containing short circuits requiring measurements with thicker sheets	1 %

Clause	Description	Value
3.4.1	Minimum difference in measured properties to consider a specimen as non-homogeneous	2 %
3.4.2	Maximum acceptable difference from a linear relationship versus thickness for thermal resistance to compute the interpolating line slope	1 %
3.4.2	Maximum difference for transfer factor at different thicknesses to be assumed as thermal transmissivity	2 %