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Thermal insulation — Determination of steady-state thermal resistance and related properties — Heat flow meter apparatus

Isolation thermique — Détermination de la résistance thermique et des propriétés connexes en régime stationnaire — Méthode fluxmétrique

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Contents

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Annexes

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 8301 was prepared by Technical Committee ISO/TC 163, *Thermal insulation*.

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

Introduction

0.1 Document subdivision

This International Standard is divided into three sections representing the most comprehensive assembly of information required to use the heat flow meter apparatus:

Section 1: General considerations

Section 2: Apparatus and calibration

Section 3: Test procedures

While the user of the method may need to concentrate only on section 3 for test purposes, he must also be familiar with the other two in order to obtain accurate and precise results. He must be particularly knowledgeable about the general requirements. Section 2 is directed towards the constructor of the apparatus, but he also, in order to build good apparatus, must be familiar with the other sections.

0.2 Heat transfer and measured properties

A large number of tests are run 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 in the gas phase;

— convection (in some operating conditions);

plus their interaction, together with mass transfer, especially in moist materials. Therefore, the heat transfer property, very often improperly called "thermal conductivity", calculated from a defined formula and the results of measurements of heat transfer 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 the "transfer factor" as it may depend on the test conditions (the transfer factor is often referred to elsewhere as apparent or effective thermal conductivity). The 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 the transfer factor on specimen thickness. As a consequence, not only the material properties but also the radiative characteristics of the surfaces bounding the specimen influence results. Thermal resistance is therefore the property that better describes the thermal behaviour of the

specimen, provided that it is accompanied by information on the bounding surfaces.

If there is any 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 that the geometry and the boundary conditions of the specimen tested be fully specified, even though information supplied in the 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. Dried specimens only therefore ought to 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 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 heat flow meter (HFM) apparatus (see 1.6.1 and 2.2.2) 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 user of measured data of the HFM apparatus should have a thorough background of knowledge of heat transfer mechanisms 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 cited may be different for the designer, operator, and data user.

0.4 Design, size, and national standards

Many different designs of heat flow meter apparatus exist worldwide to 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 carefully read the comprehensive literature cited in annex E. After completion of new apparatus it is recommended that it should be checked by undertaking tests on one or more of the various reference materials of different thermal resistance levels now available. This International Standard outlines only the mandatory requirements necessary to design and operate heat flow meter apparatus in order to provide correct results. A table summarizing limit values for the apparatus performance and testing conditions

stated in this International Standard is supplied in annex A. It also includes recommended procedures and practices plus suggested specimen dimensions which together should enhance general measurement levels and assist in improving inter-laboratory comparison and collaborative measurement programmes.

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Thermal insulation — Determination of steady-state thermal resistance and related properties — Heat flow meter apparatus

Section 1: General

1.1 Scope

1.1.1 This International Standard defines the use of the heat flow meter method (see 2.2.2) to measure the steady-state heat transfer through flat slab specimens and the calculation of the heat transfer properties of specimens.

This is a secondary or relative method since the ratio of the thermal resistance of the specimen(s) to that of a standard specimen(s) is measured.

Reports conforming to this standard test method shall refer to specimens with thermal resistance greater than 0,1 $m^2 \cdot K/W$ provided that thickness limits given in 1.7.2 are not exceeded.

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

1.1.3 If the specimens satisfy the requirements of 1.8.2, the resultant properties shall be described as the mean thermal conductivity of the specimen being evaluated.

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

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 8302:1991, *Thermal insulation — Determination of steady-state thermal resistance and related properties — Guarded hot plate apparatus.*

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 ISO 9251:

1) To be published.

Quantity	Symbol	Units
Heat flow-rate	Φ	W
Density of heat flow-rate	q	W/m^2
Thermal resistance[1)]	R	m^2·K/W
Thermal conductance	Λ	W/(m^2·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: It 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 on a heat flow meter apparatus and for the presentation through a report of 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	Quantity	Units
A	Area measured on a selected isothermal surface, or metering area	m^2
c_s	Specific heat capacity	J/(kg·K)
d	Thickness of specimen measured along a path normal to isothermal surfaces	m
d, d''	Thickness for each specimen in a two-specimen configuration HFM apparatus	m
d_m	Mean thickness of a pair of two specimens	m
d_1 , d_2 , ..., d_5	Thickness of specimens designated s_1 , s_2 , ..., s_5	m
D_t	Maximum allowable distance between hot and cold plates during the test	m
e	Heat flow meter output	mV
f	Calibration factor of the heat flow meter	W/(mV·m^2)
L	Length of the side of heat flow meter(s)	m
L_m	Length of the side of heat flow meter metering area	m
m_c	Relative mass change after conditioning	—
m_d	Relative mass change due to conditioning after drying	—
m_r	Relative mass change after drying	—
m_w	Relative mass change of a specimen during the test	—
M_1	Mass in as-received condition	kg
M_2	Mass after drying	kg
M_3	Mass after conditioning	kg
M_4	Mass after test	kg
M_5	Mass of dried or conditioned material, immediately before test	kg
q	Density of heat flow-rate	W/m^2
q' , q''	Density of heat flow-rate for each specimen in a two-specimen configuration HFM apparatus	W/m^2
r	Thermal resistivity	m·K/W
r_{avg}	Average thermal resistivity in a two-specimen configuration HFM apparatus	m·K/W
R	Thermal resistance	m^2 ·K/W
R_s	Thermal resistance of the standard specimen	m^2 ·K/W
R_u	Thermal resistance of the unknown specimen	m^2 ·K/W
R_t	Total thermal resistance in a two-specimen configuration HFM apparatus	m^2 ·K/W
s_1 , s_2 , ..., s_5	Set of specimens of different thicknesses	—
$\mathscr{T}$	Transfer factor of a specimen	W/(m·K)
$T_m = (T_1 + T_2)/2$	Mean temperature	K
T'_1 , T''_1	Hot side temperatures for each specimen in a two-specimen configuration HFM apparatus	K
T'_2 , T''_2	Cold side temperature for each specimen in a two-specimen configuration HFM apparatus	K
T'_m	Mean temperature of specimen (′) in a two-specimen configuration HFM apparatus	K
T''_m	Mean temperature of specimen (″) in a two-specimen configuration HFM apparatus	K

Symbol	Quantity	Units
V	Volume	m^3
Δd	Increment of thickness	m
$\delta d = (d' - d'')/2$	Mean thickness difference of specimens (′) and (″) in a two-specimen configuration HFM apparatus	m
$\delta\lambda$	Deviation of thermal conductivity at mean temperature T_m of specimens (′) and (″)	W/(m·K)
$\delta T_m = (T'_m - T''_m)/2$	Mean deviation between the mean temperature of specimen (′) and (″)	K
$\delta T = (\Delta T' - \Delta T'')/2$	Mean deviation between the temperature differences of specimens (′) and (″)	K
ΔR	Increment of thermal resistance	$m^2 \cdot K/W$
$\Delta T = T_1 - T_2$	Temperature difference	K
$\Delta T'$, $\Delta T''$	Temperature differences for each specimen (′) and (″) in a two-specimen configuration HFM apparatus	K
$\frac{\Delta e}{\Delta q}$	Sensitivity coefficient of the HFM	$mV/(W \cdot m^2)$
Φ	Heat flow-rate	W
Φ_u	Heat flow-rate with unknown specimen	W
Φ_s	Heat flow rate with "standard" or "reference" specimen	W
λ	Thermal conductivity	W/(m·K)
λ' , λ''	Thermal conductivity for each specimen (′) and (″) in a two-specimen configuration HFM apparatus	W/(m·K)
$\dot{\lambda}(T)$	First order temperature derivative of λ (T)	$W/(m \cdot K^2)$
$\ddot{\lambda}(T)$	Second order temperature derivative of λ (T)	$W/(m \cdot K^3)$
λ_{avg}	Average thermal conductivity in a two-specimen configuration HFM apparatus	W/(m·K)
λ_m	Mean thermal conductivity of a specimen or thermal conductivity at the mean temperature T_m	W/(m·K)
λ_M	Mean thermal conductivity of specimens (′) and (″) measured in a guarded hot plate apparatus	W/(m·K)
λ_t	Thermal transmissivity of a material	W/(m·K)
Λ	Thermal conductance	$W/(m^2 \cdot K)$
ρ_d	Density of the dry material as tested	kg/m^3
ρ_s	Density of the material after conditioning	kg/m^3
$\rho \cdot c_s$	Product of as-tested density and specific heat of the specimen	$J/(m^3 \cdot K)$
ξ	Porosity	—
ξ_p	Local porosity	—
(′), (″)	Indexes used to refer to properties of the first and the second specimen in a two-specimen configuration HFM apparatus	—

1.5 Significance

1.5.1 Factors influencing thermal properties

The thermal transmission properties of a specimen of material may

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

— be affected by moisture or other factors;

— change with time;

— change with mean temperature;

— depend upon the prior 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 shall be obtained on dried specimens, although in service such conditions may not be realized. Even more basic is the dependence of the heat transfer properties on variables such as mean temperature and temperature difference. Such dependence should be measured or the test made under conditions typical of use.

1.5.2 Sampling

Heat transfer properties need adequate 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 method, when the problem is not covered by a material specification, reference shall be made to appropriate documents.

1.5.3 Accuracy and reproducibility

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 specimens under test. The accuracy and the calibration should be a function of the reference material.

1.5.3.1 The reproducibility of subsequent measurements made by the apparatus on a specimen maintained within the apparatus without change 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 and heat flow meter on the specimen (that affects contact resistances), and the relative humidity of the air around the specimen (that affects its moisture content). These levels of reproducibility are required to identify errors in the method and are desirable in quality control application.

1.5.3.2 The accuracy of the calibration of heat flow meter apparatus is normally within $\pm$ 2 % when the mean temperature of the test is near the room temperature.

The accuracy of calibration is mainly due to the accuracy of the guarded hot plate method when measuring the properties of reference specimens.

1.5.3.3 As a consequence this method is capable of determining the heat transfer properties within $\pm$ 3 % when the mean temperature of the test is near the room temperature.

1.5.4 Calibration procedure

One of the following procedures shall be followed.

1.5.4.1 The test-laboratory apparatus shall be calibrated (see 2.4) within 24 h before or after the test using calibration standards that have been issued by a recognized standard laboratory. Stability of calibration standards depends upon the type of material; some calibration standards have been successfully used over 20 years but it is suggested to check them at least each 5 years. The reported test and the apparatus calibration test shall be carried out using approximately the same hot- and cold-side temperatures as were used in the official calibration of the standards.

1.5.4.2 Where both short- and long-term stabilities of the heat flow meter have been proved to be better than $\pm$ 1 % of the reading, the heat flow meter apparatus may be calibrated at less frequent intervals, for example 15 d to 30 d. The specimens so tested cannot be reported until after the calibration following the test and then only if the change in calibration from the previous test is less than 1 %.

The average of the two calibrations shall be used as the calibration factor and the specimens tested with this value. When the change in calibration is greater than $\pm$ 1 %, test results from this interval

shall be considered void and tests repeated in accordance with 1.5.4.1.

1.6 Principle

1.6.1 The heat flow meter apparatus tends to establish a unidirectional uniform density of heat flow-rate which simultaneously crosses the central metering area of one (or two) heat flow meter(s) and the central area of one (or two nearly identical) specimen(s) in the form of slab(s) when tested in steady-state conditions of constant mean temperature and constant temperature difference between one heating unit and one cooling unit bordering the assembly of the specimen(s) and heat flow meter(s).

1.6.2 This is a secondary or relative method, since the ratio of the thermal resistance of the specimen(s) to that of a standard specimen(s) is measured. The thermal resistance of standard specimen(s) must be determined separately in accordance with ISO 8302 on the guarded hot plate apparatus.

1.6.3 The ideal condition of unidirectional density of heat flow-rate cannot be obtained in the whole area of the specimen and heat flow meter. This implies that special attention must be given to

a) the problem of heat losses by the edges of the specimen(s) and heat flow meter(s);

b) the differences between the geometrical (thickness) and thermal properties of the standard specimen(s) and of specimen(s) to be measured;

c) the differences in temperature boundary conditions (if any) between determination of thermal resistance of standard specimen(s) in the guarded hot plate apparatus and calibration procedure of the heat flow meter apparatus by means of the standard specimen(s).

1.6.4 From the measurement of the heat flow rate Φ_s with standard specimen(s) and Φ_u with unknown specimen(s) to be measured, the assumption of a constant density of heat flow-rate of the metering section and the assumption of the stability of temperature difference ΔT and mean temperature T_m gives the ratio between thermal resistance R_s of the standard specimen(s) and R_u of the unknown specimen(s) as follows:

$$\frac{R_u}{R_s} = \frac{\Phi_s}{\Phi_u}$$

From this R_u is calculated.

1.6.5 The thermal conductivity of the specimen(s) may be also computed if the conditions of the definition are met and the thickness d of the specimens is known.

1.6.6 The application of the method is limited by the capability of the apparatus of producing unidirectional constant density of heat flow-rate in the specimens and by the accuracy in the measure of temperature, thickness, emf produced by heat flow meter, etc.

1.6.7 Another set of limits is due to the specimens, as they are not exactly of the same thickness (in the case of two-specimen apparatus); nor is the larger surface ever perfectly flat, or perfectly parallel.

1.7 Limitations due to apparatus

The use of apparatus shall be limited to a number of factors related to the calibration and to the limitations on specimen thickness.

1.7.1 Limitations related to calibration

The apparatus shall not be used at temperatures other than those applied to the calibration. If a calibration curve has been established in a temperature range, extrapolation is not allowed.

Particular attention shall be paid to use the apparatus for densities of heat flow rate comparable to those applied at calibration. This is related to the type of material to be tested, to the specimen thickness and to the temperature difference during the test.

1.7.2 Limitations related to specimen(s) thickness

1.7.2.1 General

The combined thickness of the specimen(s), the heat flow meter(s) and any damping materials, which in total equals the distance between the cold and hot plates, shall be restricted in order to limit the effect of edge losses on the measurement of heat flow-rate. A limiting geometry (see 1.7.2.2) must be chosen that corresponds to the limiting geometry of a specimen used in guarded hot plate apparatus, for which the edge losses have been estimated. The edge losses are affected by the edge insulation and the temperature of the ambient surrounding the edge of the specimen.

1.7.2.2 Maximum spacing between hot and cold plates

The maximum allowable distance between the hot and cold plates during the test, D_t, is related to the length of the side of the heat flow meter, L, the

length of the side of the heat flow meter metering area, L_m, the width of the non-metering area ($L - L_m$), the construction of the heat meter, and the properties of the test specimen. No suitable theoretical analysis is available to predict the maximum allowable thickness of specimens. It is necessary to use the results of the analysis for the guarded hot plate as a guide.

Documents [19] and [23] on guarded hot plate analysis and annex C can provide some elements for this estimation.

In the single specimen symmetrical configuration (see 2.1 and figure 1), the maximum value of the specimen thickness is increased by 50 % to that corresponding to the two specimens in symmetrical configuration.

If a specimen thickness is beyond the limits of the apparatus, tests should be performed using apparatus with larger plates or the guarded hot box.

1.7.2.3 Minimum thickness

The minimum specimen thickness is determined by contact resistances as in 1.7.3. Where thermal conductivity or thermal resistivity is required, the minimum specimen thickness is also limited by the accuracy of the instrumentation for measuring the thickness.

1.7.3 Limitations due to contact resistances

When testing rigid specimen(s), 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 the specimen and the apparatus (surfaces not perfectly flat) will cause contact resistances not uniformly distributed between the specimen(s) and the working surfaces of the heating and cooling units and heat flow meter(s).

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 without the help of special techniques.

1.8 Limitations due to the specimens

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 of non-homogeneous specimens, the density of heat flow-rate both within the specimen and over the faces of the metering area may neither be unidirectional nor constant. 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 edge heat loss errors, errors due to a non-uniform temperature distribution within the heat flow meter etc., that are now unpredictable, can vary in an unpredictable way when non-homogeneities 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 unit(s) and heat flow meter(s). 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 Influence of temperature difference

Thermal resistance or thermal conductance may be 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 resistivity of a specimen (see 1.3.4), the criteria of 1.8.1 shall be fulfilled. The specimen shall be thermally homogeneous or homogeneous porous as defined in ISO 9251. Homogeneous porous specimens shall be such that any non-homogeneity have 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 differences established across the specimen.

The thermal resistance of a material is known to depend on the relative magnitude of the heat transfer processes involved. Heat conduction, radiation and convection are the primary mechanisms. However, the mechanism 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, the complex dependence will occur at temperature differences which are typical of use. In these cases it is preferable to use such a value and 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.

1.8.3 Thermal conductivity, thermal resistivity or thermal transmissivity 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 requirement of the definitions for thermal conductivity and thermal resistivity, both defining intrinsic properties, since the transfer factors show 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. There is no established procedure for determination. The somewhat crude procedure outlined in 3.4.2, may be used for determining the thickness and whether it occurs in the range of thickness 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 thermocouples below the surfaces of the plates or heat flow meter, added resistance caused by poor specimen surfaces, and added thermal resistance caused by the coupling of the conduction and radiation modes of heat transfer in the specimen(s). All three can affect the measurements in the same way, and often the three may be additive.

Section 2: Apparatus and calibration

2.1 General

The construction guidelines given in this section must be fully understood by the user of this test method. While it is mandatory that these details be carefully followed when constructing an apparatus, the user should also verify that the equipment was built as specified. Serious errors of measurement could result from ignoring this.

As stated in 1.6, the general features of a heat flow meter apparatus with the specimen or the specimens installed are shown in figure 1; they shall consist of a heating unit, one or two heat flow meters, one or two specimens and a cooling unit.

The configuration a) in figure 1 is called "single-specimen asymmetrical"; the heat flow meter may be placed against either unit. The configuration b) is called "single-specimen symmetrical". The configuration c) is called "two-specimen symmetrical"; in this case, the specimens should be substantially identical and cut from the same sample of material.

Each configuration will yield equivalent results if used within the limitations stated in this method. There are distinct advantages for each method in practice. A brief discussion is included in annex B. When more than one heat flow meter apparatus is desired, a double apparatus can be constructed by using the other side of the heating unit and adding another heat flow meter and a cooling unit. Examples of both single and double apparatus are described in [2] to [7] and [16]; see also figure 1d) and figure 1e).

2.2 Apparatus

The working surfaces of the heating and cooling units and the heat flow meter(s) (i.e. the surfaces making contact with the specimens) shall be painted or otherwise treated to have a total hemispherical emittance of greater than 0,8 at operating temperatures.

2.2.1 Heating and cooling units

2.2.1.1 General description

The heating and cooling units shall be constructed so that isothermal working surfaces will be obtained. This may be achieved by placing an electrical winding of uniform specific power between two metal plates or by circulating a constant-temperature liquid between the plates or by a combination of both or by other adequate means; (see [2]). Liquid-heated metal plates need particular care in their design. The worst case thermal load should be first defined, then the liquid flow rate should be tentatively assigned and in this situation the fluid temperature difference between plate inlet and outlet should be evaluated to check whether the liquid flow rate is correct. 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 (see figure 2). However, in this case the thermal resistance between the fluid and the metal plate should be sufficiently high, otherwise plate temperature non-uniformity can be even larger than the temperature difference between the inlet and outlet fluid. For information on the correct design of a liquid-cooled or liquid-heated metal plate, see [9] and [14]. The temperature uniformity of the working surfaces of a heating or cooling unit in a heat flow meter apparatus may be even more critical than in a guarded hot plate apparatus, as some heat flow meters may be sensitive to temperature differences along their main surfaces; (see 2.2.2.3).

The working surface of the heating and cooling units shall consist of a metal of high thermal conductivity and shall be smoothly finished to conform to a true plane within 0,025 %.

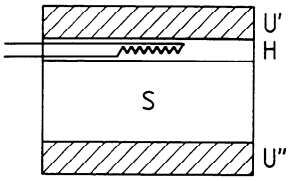
The cooling unit shall be so constructed that an isothermal working surface is obtained with a surface dimension at least as large as that of the working surface of the heating unit.

The heating and cooling units may be identical.

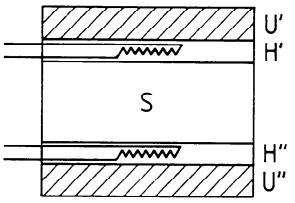
2.2.1.2 Temperature requirements

The temperature uniformity on each working surface will be better than 1 % of the temperature difference across the specimen(s).

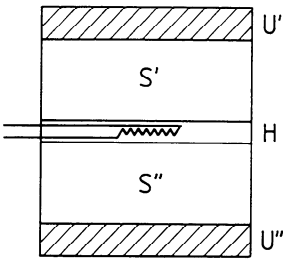
In addition, if a heat flow meter is placed in contact with the working surface of a heating or cooling unit and is sensitive to the temperature differences along this surface, this difference shall be as small as necessary to maintain an error in measured heat flow-rate below 0,5 %.



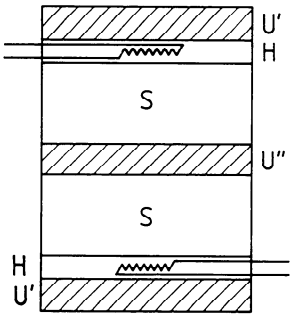
a) Single-specimen asymmetrical configuration



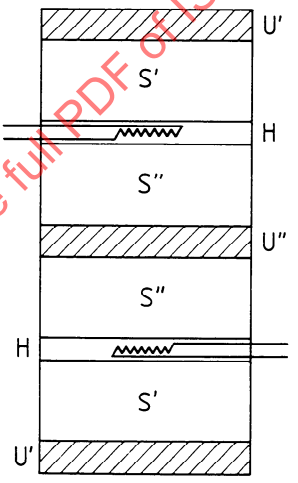
b) Single-specimen symmetrical configuration



c) Two-specimen symmetrical configuration



d) Double apparatus



e) Double apparatus

U', U'' cooling and heating units

H, H', H'' heat flow meters

Figure 1 — Typical layouts of heat flow meter apparatus configurations

The working surface temperatures during the test period shall not fluctuate or change by more than 0,5 % of the temperature difference across the specimen. In addition, the surface temperature fluctuations on the face of the heat flow meter in contact with the specimen(s) shall be less than 0,5 % of the temperature difference across the specimen; see also annex B. The temperature fluctuations (as a function of time) at the surface of the heat flow meter shall not cause fluctuations in the electrical output greater than 2 % during the test period. These fluctuations are due to poor quality of automatic controllers combined with the thermal capacity of the heat flow meter. The insertion of a thin layer of insulating material between the heat flow meter(s) and the working surface(s) can reduce this trouble.

2.2.2 Heat flow meter

2.2.2.1 Purpose

The heat flow meter is an assembly that measures the density of heat flow-rate through the specimen(s) by a temperature difference generated by this density of the heat flow-rate crossing the specimen(s) and the heat flow meter itself. Several types of heat flow meter are described in 2.2.2.6.

Most commonly, it consists of a homogeneous core, a surface temperature difference detector and a surface temperature detector(s). The heat flow meter region occupied by the core, where temperature

difference detectors are placed, is called the metering area. It can also have cover sheets to provide protection and thermal damping. Metal temperature-levelling plates or foils are sometimes used to improve or simplify the measurements, but these shall be situated so as to prevent the temperature difference from being dependent on specimen thermal properties.

2.2.2.2 Core

The core shall be constructed from a suitable non-hygroscopic material which shall be sufficiently uniform and isotropic, and shall have sufficiently parallel faces, to ensure a uniform heat flow normal to the faces. The core material shall not effectively change under the temperature and humidity conditions of use and storage nor from typical handling. It shall be thermally uniform and shall remain stable over a long period of time. It shall be hard, with low compressibility. The following are some of the materials that can be used for heat flow meters: cork composition, hard rubber-plastics, ceramics and phenolic laminates, epoxy, or silicone-filled glass-fibre cloth.

2.2.2.3 Thermopile

The temperature difference across the core material shall be measured with a sensitive stable temperature detector (see annex B, B.1 and B.2). Multi-junction thermopiles have been used successfully. Some types of these are represented in figure 3. The junctions are placed on the surfaces of the core material of the heat flow meter to measure the temperature difference through the core.

The resulting effect of the presence of the thermopile in the core is a thermopile output e that is related to the density of heat flow-rate q, through a parameter f called the calibration factor, as follows:

$$q = f e$$

The parameter f is not strictly a constant but depends upon temperature and to a more limited extent upon the density of heat flow-rate itself.

To avoid the effects of heat conduction along the element passing from one face to the other, it is recommended that the cross-sectional area of the conductors in the thermopile be smaller than that of an 0,2 mm diameter wire.

Thermo-elements that produce a high emf output and have a low thermal conductivity are recommended. Both ribbon and plated thermocouples have proved advantageous in certain designs. Metallization and photoetching techniques have been also used successfully; see [16], [24] and [25]. The main meter surfaces are assumed isothermal, so that density of heat flow-rate will be normal to the main meter surfaces. If the hypothesis is not true, there will be a component of the density of heat flow-rate that will be parallel to the main heat flow meter surfaces. The sensitivity to this component depends on the layout of the thermopile junctions: the junctions of figure 3b) and figure 3c) are not sensitive to temperature differences along the main surfaces, while the junctions of figure 3a) are sensitive to temperature differences along directions both normal and parallel to the main meter surfaces; such designs should therefore be avoided whenever possible. Precautions must be taken to limit the effect of heat flow through the leads on the output of the temperature difference detector.

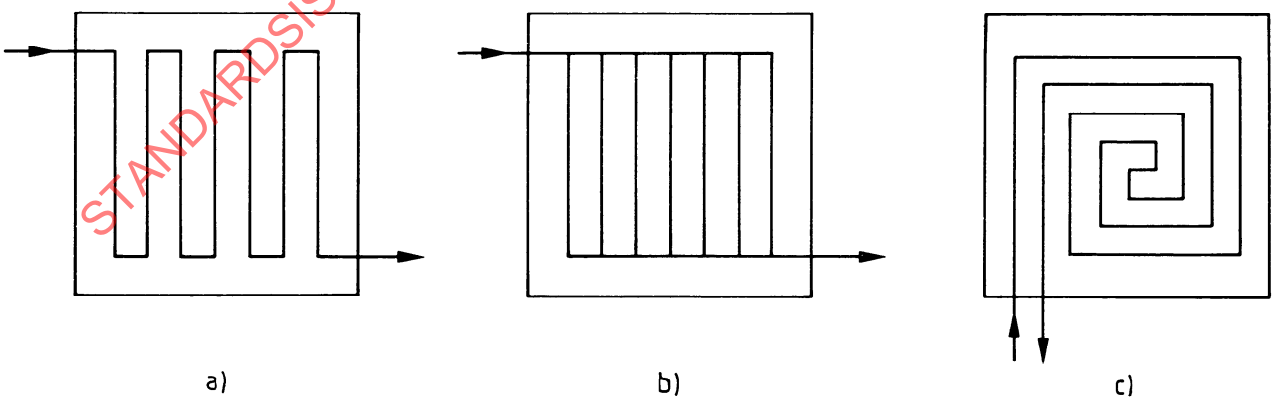


Figure 2 — Examples of schematic designs of heating and cooling units in the case of external liquid supply

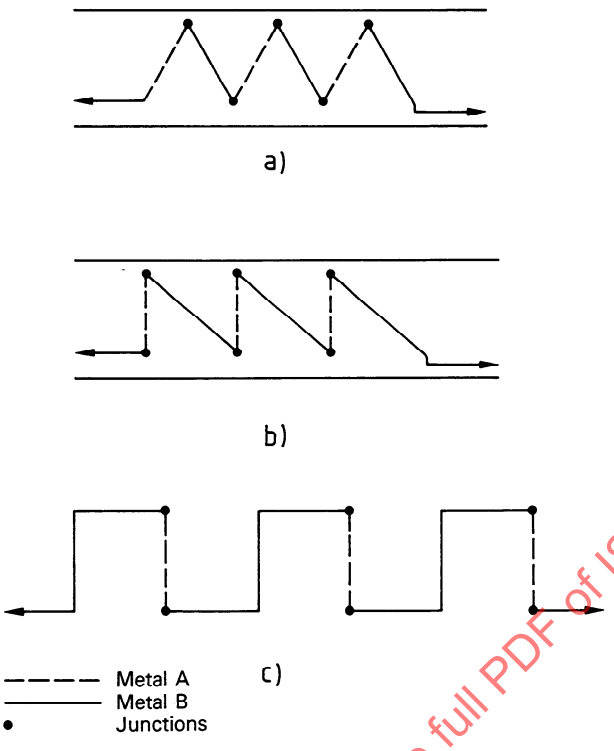


Figure 3 — Examples of schemetic designs of thermopiles

When the heat flow meter output is less than 0,000 2 V, special techniques must be used to prevent extraneous thermal emf's in leads, the measuring circuits, and the heat flow meter itself. The latter can only be found by testing the heat flow meter at several heat flow-rates, half in one direction, half in the other, and examining the zero intercept of the line joining the points.

To ensure uniform thermal resistance in the heat flow meter, the temperature difference detectors shall either

a) be uniformly distributed within the metering area of the heat flow meter — an area not larger 40 % nor less than 10 % of the entire surface area; or

b) be concentrated in areas of not less than 10 % of the entire surface area; these areas shall be within the most central 40 % of the meter.

2.2.2.4 Surface sheets

To prevent damage to the temperature-difference detector that will affect its calibration, both surfaces shall be covered with a layer of material as thin as is compatible with protection from thermal shunting of the temperature-difference detector wires. A properly designed heat flow meter should have a sensitivity as independent as possible of the thermal conductance of the specimens for a large range of thermal conductances. The surface sheet may also be chosen to aid in damping any temperature fluctuation. The surface sheet should be similar to the core material and should be tightly bonded to the core by chemical means such as adhesive films or fusible materials or other adequate technology. The metering area of the heat flow meter shall be smoothly finished to conform to a plane to within 0,025 %.

2.2.2.5 Surface temperature sensors

A suitable device shall be employed for measuring the average temperature of the specimen side(s) of the heat flow meter(s).

It has been found that an 80 µm thick piece of copper foil, adhering to the surface sheet, can be used to average the surface temperature of the heat flow meter over the area where the junctions of the thermopile are placed. The foil should extend beyond this area by a distance approximately equal to the thickness of the heat flow meter. The foil can be used as a part of a copper-constantan thermocouple circuit, or can be equipped with a platinum resistance sensor. When thermocouples are used for this purpose, copper and constantan wires 0,2 mm in diameter or smaller, are strung through the surface sheet before it is attached to the core. The constantan wire is soldered to the centre of the foil

while the positive lead is soldered near one of the edges. All the excess solder should be removed. The surface sheet can be sanded away to eliminate any lumps. The surface of the heat flow meter not covered by the metal foil is masked by an applied sheet of non-metal, 80 μ in thickness, to ensure a smooth surface. The thermocouple wires shall comply with 2.2.3.1.3.

2.2.2.6 Types of heat flow meters

There are several types of heat flow meters. The type of heat flow meter described in this test method is called a gradient-type. It consists of a slab of material across which the temperature gradient is measured, normally with a thermopile.

They are generally used for steady-state or quasi-steady-state measurements, although they have been adapted to transient work, for which approximate equations have been derived (see [13] and [20]).

Two general constructions of gradient heat flow meters are used in practice. They are of respectively high-thermal resistance and low-thermal resistance.

The high-thermal resistance type consists of a high-thermal resistance slab of material, such as cork, with a small thermopile wound around it to measure the temperature difference. A facing material is placed on each surface and a set of temperature sensors, or a single sensor, and a temperature-levelling plate are placed on each surface, and the two surfaces of the assembly covered with a low-permeability membrane. The characteristics of this heat flow meter are a large temperature drop, adequate sensitivity with a small thermopile, ease of manufacture, and low dependence for the sensitivity value on the properties of the test specimen.

The low-resistance type consists of a thin slab of relatively low-resistance material, such as epoxy or silicone-filled glass-fibre cloth, with a very sensitive thermopile wound around it to measure the small temperature difference. Again, a facing material is placed against each surface. In many cases this material is simply an insulating film and a metal plate. The characteristics of this heat flow meter are a small temperature drop across it, adequate or high sensitivity; it requires special techniques of design and manufacture (see [24] and [25]).

2.2.3 Other measuring devices

2.2.3.1 Temperatures

2.2.3.1.1 Apparatus temperatures

Any proven method allowing temperature difference measurements between the working surfaces of the heating/cooling units (and heat flow meter(s) if needed) in contact with specimen(s) to an accuracy of 1 % may be used for measurements of temperatures in the apparatus. For the heat flow meter surface temperature determination, see 2.2.2.5.

The temperature of the working surfaces of the heating and cooling units are often measured by permanently mounted temperature sensors such as thermocouples which are set in grooves or just under the working surfaces. When configuration c) in figure 1 is chosen (see 2.1), the sensors placed on the working surfaces of the cooling and heating units are sometimes connected differentially. For thermocouples, this is usual. In such cases, they must be electrically insulated from the plates, the metal plates shall be grounded and a resistance greater than 1 MΩ is recommended. The number of such thermocouples on each side should be not less than $10\sqrt{A}$, or 2, whichever is greater, where A is the metering area section in square metres. One thermocouple per surface has been found adequate on existing plates having surface areas of less than 0,04 m^2, provided that either the thermocouples are changed frequently or the thermocouple calibration is checked regularly. A minimum of two thermocouples is required for all new apparatus.

For certain types of materials, this procedure can lead to errors in the measurements due to the thermal resistance between the sensors and the surfaces of the specimens (see [10]).

In this case, see 2.2.3.1.2.

2.2.3.1.2 Temperature differences through the specimen(s)

2.2.3.1.2.1 For non-rigid specimens (see 1.7.3) with surfaces that conform well to the flat surfaces of the plates and of thermal resistance more than 0,5 $m^2 \cdot K/W$, the temperature difference through them is normally measured from the indications provided by the sensors permanently mounted in the working surfaces of the unit(s) and/or heat flow meter(s) in contact with specimen(s).

2.2.3.1.2.2 In some cases there exists the possibility of an influence of contact resistances between the specimen(s) and the apparatus working surfaces; some special methods may be required to determine the temperature difference through the specimen(s). Errors in some methods are described in [10], yet in some instances the choice of method itself is left to the judgement of the operator. A proven technique for rigid specimens (see 1.7.3) consists of interposing thin sheets of suitable homogeneous material between the specimen(s) and each apparatus working surface involved.

The temperature difference through the specimen(s) is then determined by means of separate thermocouples mounted flush with or interior to the surface of the specimen(s). This technique can also

be used in conjunction with thin layers of low-resistance material interposed between the specimen(s) and the working surfaces.

2.2.3.1.3 Temperature sensors

When thermocouples are used, those mounted in the surface of the units shall be made of wire not larger than 0,6 mm in diameter — and preferably not larger than 0,2 mm in diameter for small size apparatus.

Other solutions, such as thermocouples embedded in thin sheets, require particular care to reduce errors in the detection of surface temperatures, mainly with low-resistance specimens.

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 the thermocouples be placed within the specimen surfaces whenever possible. Otherwise thinner diameter thermocouples must be used. The thermocouples that are used to measure the temperature of the faces of the specimen(s) as indicated above should be manufactured from either calibrated thermocouple wire or from wire that has been certified by the supplier to be within the special limits of errors given in table D.1 in annex D. Other temperature sensors or detectors such as for example platinum resistance sensors must have equivalent or better accuracy, sensitivity and stability. The resulting error in temperature differences due to distortion of the heat flow around the sensor, to sensor drift, and other sensor characteristics shall be less than 1 %.

2.2.3.2 Electrical measuring systems

2.2.3.2.1 General features

The design of the measuring system will depend on the type of temperature sensors used and the sensitivity of the thermopile or 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, multi-range, measuring instruments. The choice will be governed by the general requirements of the user. The capability of the heat flow meter apparatus to make rapid measurements is best utilized where the measuring system computes and displays either thermal resistance, conductance, or thermal conductivity. Nevertheless, the measuring system shall provide means for monitoring each individual temperature sensor. The computing circuitry may be digital or analog. Where digital, it need not add any significant error or limitations. Where analog, it can degrade the short and long-term stability, linearity, and accuracy, and degrade the sensitivity. The computing circuitry must be considered part of the measuring system in certification testing, and the requirements in 2.2.3.2.2 met for the overall system. Long-term stability remains a problem with analog computing circuitry. A special check must be made at each reporting. All factors cited in 2.2.2 should be considered carefully if reliable results are to be obtained.

2.2.3.2.2 Measuring system requirements for high-level performance and reporting testing

When the apparatus is used for high-level performance in measurements or reporting testing, the measuring system shall have the following capabilities, regardless of whether a wide-range or a multi-range measuring system is used:

a) a sensitivity, linearity, accuracy and input impedance adequate to measure the temperature differences across the specimens to within ± 0,5 % and the output from the thermopile to within ± 0,6 %;

b) a sensitivity of better than 0,15 % at minimum output from the temperature-difference detector;

c) sufficient linearity so that it contributes less than 0,1 % to the error at all expected outputs of the temperature-difference detector;

d) sufficient input impedance so that it does not cause an error of more than 0,1 % to the reading under any possible condition (1 MΩ has been found adequate for many apparatus);

e) stability so that it contributes less than 0,2 % error to any reading during a normal period between calibration, or 30 d, whichever is greater;

f) adequate noise immunity so that with the type of leads, grounding and shielding used in apparatus, less than 0,1 % rms noise occurs in the values of temperature difference and thermopile output.

2.2.3.3 Thickness measurement

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 temperature, 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 plates or along the axis perpendicular to the plates at their centres, will serve for these measurements. The effective combined specimen thickness is determined by the average difference

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 plates towards each other. In the case of use of an electrical transducer, provision must be made for checking its linearity and its electronic circuitry. Check of this linearity shall be made at intervals of less than one year.

2.2.4 Mechanical devices

2.2.4.1 Framework

A framework should be provided to hold the apparatus in one or more orientations.

2.2.4.2 Clamping force

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, regardless of orientation. A steady force, which will thrust the 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 on the specimens will be required. When compressible 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 and heating units to limit the compression of the specimen(s). Other means may be used to impose the distance between working surfaces; a constant pressure arrangement is not needed for such tests.

2.2.5 Edge insulation and edge heat losses

2.2.5.1 General

Heat losses from the outer edges of the heat flow meter apparatus shall be restricted by edge insulation or by controlling the surrounding air temperature or by both.

A cabinet or enclosure surrounding the heat flow meter assembly to maintain the ambient temperature at the mean temperature of the specimen(s) shall be provided, especially when conducting tests at mean temperatures differing substantially from the laboratory air temperature.

A cold radiator existing generally as a part of the temperature control system in the enclosure will provide a dew point temperature at least 5 K lower than the temperature of the cooling unit and consequently will prevent condensation and moisture pick-up by the specimen(s).

2.2.5.2 Influence on test configuration type

The three different configurations (see 2.1) differ in their behaviour regarding edge heat losses and consequently require different solutions to minimize them.

2.2.5.2.1 The single-specimen asymmetrical configuration is similar to the guarded hot plate apparatus as regards edge heat losses through the specimen. The edge heat losses in the heat flow meter are more important than in the guarded hot plate apparatus because they may introduce additional errors due to temperature non-uniformity on the side in contact with the specimen.

2.2.5.2.2 The two-specimen symmetrical configuration is sensitive to edge heat losses on the heat flow meter since the heat that flows through the edges is supplied via the specimens rather than directly from the heavy metal units. Since the working surface of these units is nearly isothermal, the surface temperature may no longer be uniform. If the heat flow meter is sensitive to temperature differences along its main surfaces, edge heat losses may now create serious errors. To prevent them, the enclosure described in 2.2.5.1 maintained at the mean test temperature is mandatory. As regards the edge heat losses within the specimens, they are similar to those in the guarded hot plate when the surrounding temperature is that of heating and cooling units.

2.2.5.2.3 The single-specimen symmetrical is the least sensitive to edge conditions if the average of readings of the two heat flow meters is assumed as the measured heat flow-rate through the specimen. In addition, this configuration will make the evaluation of edge heat losses easier if the two heat flow meters are identical and if uniformity of temperature of the working surfaces is obtained.

2.2.5.3 Edge heat losses evaluation

For all the configurations given in 2.2.5.2.1 to 2.2.5.2.3, the sensitivity to edge heat losses is closely connected to the sensitivity of the heat flow meter and to the temperature differences along its main surfaces; therefore, only experimental checks while changing environmental conditions can confirm for each operating condition the magnitude of the effect of edge heat losses on the measured heat flow-rate. In all cases the limitation requirements on specimen thickness specified in 1.7.2 shall be satisfied.

In such conditions, the edge heat loss errors should be smaller than 0,5 %. A very rough guideline to obtain small edge heat loss errors is that of maintaining the heat flow rate lost through the edges below 20 % of the heat flow rate through the specimen(s).

A complete analysis of the edge heat losses is given in [19] and in ISO 8302.

2.3 Guidelines for apparatus design

2.3.1 Required performance

The design of a heat flow meter apparatus will depend on preliminary awareness of the following parameters:

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

— minimum and maximum specimen thermal resistance;

— minimum and maximum temperature difference across the specimen;

— minimum cooling unit temperature;

— maximum heating unit temperature;

— overall apparatus accuracy and reproducibility as maximum acceptable error in measured property in a defined worst case condition;

— surrounding environment;

— type of heat flow meter apparatus;

— sensitivity coefficient of the heat flow meter, $\frac{\Delta e}{\Delta q}$, in millivolts per watt square metre.

2.3.2 Tentative selection of apparatus dimensions

As a first trial, assume the side, L_m, of the metering area of the heat flow meter to be four times the maximum specimen thickness and the external side, L, eight times the maximum specimen thickness.

2.3.3 Heating and cooling units

Compute the maximum heat flow-rate crossing the specimen when its thermal resistance is a minimum and the temperature difference across the specimen is a maximum. Add the heat flow-rate due to edge heat losses and the heat flow-rate exchanged from the units towards the environment. Define heating and cooling systems, working surface metal plate thicknesses and mass flow rate of the cooling fluid (where pertinent) to reach the temperature uniformity stated in 2.2.1.2.

2.3.4 Heat flow meter

The design must ensure a uniform temperature at the HFM surface adjacent to the specimen so that the higher edge heat loss associated with thick specimens will not produce lower edge surface temperatures on the HFM and thus avoid an additional distortion of the heat flow lines. It is at least equally important that it should not produce a lateral heat flow-rate in the core of the HFM. If lateral heat flow-rate does occur in the core, the thermopile output will not be proportional to the heat flow-rate into the specimen (see [21]).

2.3.5 Detailed design

Satisfactory values for the size and dimensions of the apparatus shall first be determined from the following parameters:

— define surface tolerances according to minimum specimen thickness;

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

— define all apparatus details such as temperature-sensing element positions and mounting, heater layout, wiring, mechanical connections, thickness measuring device, etc.;

— select cooling and heating systems according to minimum cooling unit and maximum heating unit temperatures;

— select temperature automatic control systems according to minimum temperature drifts and fluctuations acceptable for the apparatus;

— select a conditioning system according to the required surrounding environment and according to the needs on its stability and drifts to keep edge heat losses errors within the stated values;

— select the heat flow meter type and its characteristics (see annex B).

2.4 Calibration

Heat flow meter apparatus calibration is a very critical operation. Since lateral heat losses or gains of heat are not automatically controlled, but only "ensured" by the size of the guard area and edge insulation, there is no guarantee that the heat losses or gains are minimized under all test conditions. To ensure that the equipment performs properly with specimens of different thermal resistances, the apparatus must be calibrated with materials having similar heat-transfer characteristics as the materials

to be evaluated; extrapolations should be avoided whenever calibration specimens exist covering the intended test range.

These reference materials must be evaluated through an absolute test method such as the guarded hot plate.

2.4.1 General

The calibration of the heat flow meter apparatus should be made using a pair of specimens as similar as possible tested with the guarded hot plate apparatus. To understand the calibration procedure correctly, it is necessary to develop a minimum of theoretical background (see [20]) and to analyse first the measurements on the two specimens in the guarded hot plate apparatus and then to analyse the measurements on the same specimens in the heat flow meter apparatus. Similar computations can be made for the one-specimen guarded hot plate apparatus. First of all it is assumed as in 1.8.2 that the reference specimens will be homogeneous (as defined in ISO 9251 and 1.3.1) and that steady-state heat transfer properties will be independent on thickness and temperature gradients, so that reference to thermal conductivity or thermal transmissivity will be correct.

The index (′) is used to refer to properties of the first specimen, while the index (″) refers to properties of the second specimen. For each specimen tested in the guarded hot plate apparatus, the thickness d' or d'', the thermal conductivity λ' or λ'', the hot side temperature T'_1 or T''_1, the cold side temperature T'_2 or T''_2 are considered. The mean thickness $d_m = 0{,}5(d' + d'')$ and the thickness difference $\delta d = 0{,}5(d' - d'')$ are defined so that $d' = d_m + \delta d$ and $d'' = d_m - \delta d$.

In a similar way the following mean temperatures and temperature differences are defined.

$$T'_m = \frac{T'_1 + T'_2}{2} \qquad \Delta T' = T'_1 - T'_2$$

$$T''_m = \frac{T''_1 + T''_2}{2} \qquad \Delta T'' = T''_1 - T''_2$$

$$T_m = \frac{T'_m + T''_m}{2} \qquad \delta T_m = \frac{T'_m - T''_m}{2}$$

$$\Delta T = \frac{\Delta T' + \Delta T''}{2} \qquad \delta T = \frac{\Delta T' - \Delta T''}{2}$$

$$\delta_+ = \delta T_m + \frac{\delta T}{2} \qquad \delta_- = \delta T_m - \frac{\delta T}{2}$$

so that

$$T'_1 = T_m + \frac{\Delta T}{2} + \delta_+ \qquad T'_2 = T_m - \frac{\Delta T}{2} + \delta_-$$

$$T''_1 = T_m + \frac{\Delta T}{2} - \delta_+ \qquad T''_2 = T_m - \frac{\Delta T}{2} - \delta_-$$

As defined in 1.4,

T_m is the mean temperature;

ΔT is the mean temperature difference.

and δ_+ and δ_- take into account the deviations in each specimen of the mean temperature from T_m and of temperature difference from ΔT. In an ideal guarded hot plate apparatus it should be $\delta_+ = \delta_- = 0$.

Assuming that at the mean temperature T_m, both specimens have thermal conductivities of respectively $\lambda'_m = \lambda_m + \delta\lambda$ and $\lambda''_m = \lambda_m - \delta\lambda$ and that the temperature derivatives $\lambda(T)$ and $\lambda(T)$ at T_m are the same for the two specimens [$\lambda(T)$ and higher order derivatives being neglected] and are known at the same mean temperature T_m, and omitting the products of δT and δT_m and their powers, a series expansion is used to calculate the densities of heat flow-rate q' and q'' through each of the two specimens.

From the mean value of $q = 0{,}5(q' + q'')$ the mean thermal conductivity λ_M in the guarded hot plate apparatus is

$$\lambda_M = q \frac{d_m}{\Delta T}$$

and may be expressed as follows when the ratio $\lambda_m/\lambda(T)$ is far larger than unit temperature difference and when ΔT is limited to 20 K to 40 K:

$$\lambda_M = \frac{\lambda_m}{1 - \left(\frac{\delta d}{d_m}\right)^2}\left[1 - \frac{\delta\lambda}{\lambda_m}\left(\frac{\delta d}{d_m} - \frac{2\delta T}{\Delta T}\right) - \right.$$

$$\left. - \frac{\delta d}{d_m} \times \frac{2\delta T}{\Delta T} + \frac{\lambda(T)}{3!\lambda_m}\left(\frac{\Delta T}{2}\right)^2\right] \qquad \ldots (2.1)$$

If $\delta d = 0$

$$\lambda_M = \lambda_m\left[1 + \frac{2\delta T}{\Delta T} \times \frac{\delta\lambda}{\lambda_m} + \right.$$

$$\left. + \frac{\lambda(T)}{3!\lambda_m}\left(\frac{\Delta T}{2}\right)^2\right] \qquad \ldots (2.2)$$

For calibration purposes, $\delta\lambda/\lambda_m$ is assumed 0,02 if it is unknown and λ_M should differ by less than 0,2 % from λ_m.

2.4.2 Single-specimen asymmetrical configuration

The first specimen is first mounted in the HFM apparatus and e', T'_1 and T'_2 are measured, then the second specimen is mounted and e'', T''_1 and T''_2 are measured, e' and e'' being the HFM output. Also d' and d'' should be known. The formal definitions of T'_m, T''_m, T_m, $\Delta T'$, $\Delta T''$ and δT_m are as above, so that

$$T'_1 = T_m + \delta T_m + \frac{\Delta T'}{2} \qquad T'_2 = T_m + \delta T_m - \frac{\Delta T'}{2}$$

$$T''_1 = T_m - \delta T_m + \frac{\Delta T''}{2} \qquad T''_2 = T_m - \delta T_m - \frac{\Delta T''}{2}$$

where

T_m is the mean temperature of both tests;

$\Delta T'$ and $\Delta T''$ are the temperature differences in the two tests;

δT_m is the difference between the mean temperature in each test and T_m.

If both tests were conducted in ideal conditions, it would be $\delta T_m = 0$ and $\Delta T' = \Delta T''$.

If

$$\Lambda' = \frac{\lambda_m}{d'}$$

$$\Lambda'' = \frac{\lambda_m}{d''}$$

$$\lambda'_m = \lambda_m + \delta\lambda$$

$$\lambda''_m = \lambda_m - \delta\lambda$$

using again the first terms of a power-series expansion for the density of heat flow-rate $q' = e'f$ in the first test and $q'' = e''f$ in the second test, we can deduce the calibrating factor f by computing $q = 0{,}5(q' + q'')$ so that we will obtain the following expression:

$$f = \frac{2}{\dfrac{e'}{\Lambda'\Delta T'} + \dfrac{e''}{\Lambda''\Delta T''}} \times \left[1 + \frac{2\dot{\lambda}(T)}{3!\lambda_m}\left(\Delta T'^2 + \Delta T''^2\right)\right] \quad \ldots (2.3)$$

It is important to observe that Λ' and Λ'' are not the actual thermal conductances of the two specimens, that are usually unknown, but are defined through d', d'' and λ_m.

The calibration should be conducted so that the term within brackets will differ from 1 by less than 0,2 %.

2.4.3 Single-specimen symmetrical configuration

The calibration of each HFM is exactly as the calibration of the HFM in the single-specimen asymmetrical configuration, with two equations similar to equation (2.3), where in each case f, e', e'' correspond to the heat flow meter considered as its own mean temperature.

2.4.4 Two-specimen symmetrical configuration

Equation (2.3) for the single-specimen asymmetrical configuration applies again, provided that e' and e'' are both replaced with the output e of the heat flow meter to be calibrated.

We have now $q' = q'' = ef$ and δT_m is nearly equal to $\Delta T'/2$ and $\Delta T''/2$.

NOTE 1 This assumes the possibility of operating in the case of 2.4.2 and 2.4.3 with a specimen consisting of a pair of specimens measured in the guarded hot plate apparatus by using equation (2.3) as here.

2.4.5 Calibration curve

Most heat flow meters are mean temperature-sensitive and the calibration factor will change with the mean temperature T_m. So the above measurements shall be repeated at different mean temperatures to cover the temperature range of the heat flow meter apparatus. A calibration curve or equation shall be prepared (calibration factor versus mean temperature of the heat flow meter apparatus assembly).

The specimen may be placed between the warm plate and the heat flow meter if determination of heat transfer properties at higher temperatures than the safe temperature limit of the heat flow meter is intended.

The calibration must be carried out with at least two specimens and preferably three, of widely differing thermal resistances in order to check linearity of the heat flow meter(s) e.m.f. response versus q (see [16]). If the curve $q = F(e)$ is not linear (slope equal to f), f will vary with q and this fact shall be taken into account after checking possible reasons for such non-linearity (excess of edge heat losses for example).

A preliminary investigation of the sensitivity and the linearity of the HFM to be mounted in an HFM apparatus can be attempted in a guarded hot plate apparatus (preferably a single specimen apparatus), sandwiching the HFM with reference specimens.

2.5 Performance check

2.5.1 Geometry

The planeness of the working surfaces can be checked with a steel straightedge, of a length greater than the width or diameter of them, held against each surface and viewed with a light behind the straightedge. Departures as small as 25 µm are readily visible, and larger departures can be measured using shimstock or thin paper.

2.5.2 Computing circuity

Where direct readout equipment is provided, adequate provision shall be made for calibration of the analog electronic circuitry, independent of the rest of the apparatus. Isolated voltage sources with calibrated output, or equivalent, shall be substituted for thermopile, temperature sensors, and thickness transducer — the latter only where it has an active output. High-quality switches, or equivalent, shall be used to switch these into the circuit. Two test circuits shall be provided for checking the calibration. One circuit shall check the calibration at between 0 % and 10 % of the range, and one at between 90 % and 100 % of the range. The direct-reading equipment shall be checked immediately prior to and following a reporting test. Results shall be reported as outlined in section 3.

2.5.3 Heat flow meter

2.5.3.1 Preliminary test

Any heat flow meter that is new or has been modified must be tested for the following characteristics:

2.5.3.1.1 Zero offset

If there is a non-zero output from the thermopile for zero heat flow, this may be due to

a) bad electrical connections of a sensing thermopile with low output: improve connections to eliminate the problem as this type of drift is temperature-sensitive;

b) sensitivity to warm or cold plate temperature non-uniformity: check the temperature non-uniformity; during all operating conditions it shall be within the limits given in this International Standard.

In both cases, no corrections are allowed.

2.5.3.1.2 Drift in the heat flow meter

This could be due to material ageing or delamination.

2.5.3.1.3 Temperature coefficient of the calibration factor

This depends on the type of temperature detectors used in the temperature-difference detector (thermocouple materials used in the thermopile) and the type of material used for the metering slab.

2.5.3.1.4 Heat flow down the leads

This may occur.

2.5.3.1.5 Effect of the thermal conductivity of the specimen on the calibration factor (linearity range of the heat flow meter)

A "thermal shorting" effect between elements, caused by low thermal resistance between the metal windings of the thermopile and a funnelling of heat through the windings, can change the calibration factor of the heat flow meter.

2.5.3.1.6 Effect of loading pressure on the calibration factor

See annex B, B.1.

2.5.3.1.7 Non-linear output from the temperature-difference detector

This may occur.

2.5.3.2 Corrections

In all cases, corrections shall be made where a change of greater than 1 % occurs over the range of operation; corrections are recommended for changes of 0,3 % over the operation range.

2.5.4 Calibration drift

When a new heat flow meter apparatus is built, its calibration shall be checked at least once every week. Less frequent calibrations may be performed when many subsequent checks are satisfactory.

Maintain a running record of calibration from standard specimen calibration tests. This record will also show the reproducibility of the heat flow meter apparatus as a function of time. If the results indicate a difference in the measurement of the thermal resistance of the standard specimens of more than ± 1 %, check the controllers of the apparatus (temperature of the units, etc.). If necessary, determine a new calibration factor curve or equation. It is unlikely that the shape of the curve will change. The curve can be shifted into a new position.

When this apparatus is used for daily quality control of a certain type of insulating product at a constant mean temperature, the best reliability in data is obtained by adding a daily calibration check with sec-

ondary standards specimens taken from the same product to the above procedure.

In all cases, any progressive drift of the calibration must be carefully investigated and action taken in order to detect the origin of this drift and to solve this problem.

2.5.5 Overall performance checking

The test results obtained by this method can only be guaranteed if the limitations of the apparatus are known. To establish the limitations, the performance should be confirmed by comparing the results with materials of similar heat transfer properties to those to be evaluated.

2.5.5.1 A single reference point may lead to serious errors. It is best to select a range of materials having heat transfer properties which cover a range of values to be tested (see 2.4).

2.5.5.2 When the apparatus is to be used at thicknesses greater than those of the reference material, a series of measurements must be performed to ensure that the equipment does not cause additional errors which may be due to lateral losses or gains brought about by insufficient guarding. A simple technique is to insert radiantly opaque septa between layers of insulation, each tested by the primary reference technique. If no errors are discovered and no infrared radiation re-emitted, the measured average thermal resistance value should be equal to the average thermal resistance value obtained for the individual insulation specimen (see [18]).

Section 3: Test procedures

3.1 General

The measurement of the heat transfer properties of a low thermal conductance sample of a thermal insulating material can be determined in accordance with a set of specifications based upon sections 1 and 2. It is assumed that the operator is fully conversant with all of the foregoing basic principles of heat transfer and those of the design and operation of the heat flow meter 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 a material, product or system.

Thus, before any measurements are undertaken, 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 the following.

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, number and form of specimens either provided or needed. This will depend on the ultimate requirements of a particular sample or material. If the material, product or system is highly anisotropic in nature, whether measurements are possible with the heat flow meter 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 on instrumenting the specimen with temperature measurement sensors or thermocouples (see 2.2.3.1.2.2). These techniques are intended to make correct measurements of the temperature difference across the specimen of low thermal resistance and/or of rigid material by eliminating the possible influence of contact resistances.

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

e) The need of imposing either specimen thickness or pressure on the specimen.

The operator must also be conscious of the difference between a measurement the goal of which is to define one of the steady-state heat transfer properties given in section 1, and a measurement required by a material specification. The latter one may also 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 , or tested at a thickness far from the end use. The numerical results of these tests must therefore be regarded as conventional tools to accept or reject lots of a particular material, but not necessarily as a meaningful heat transfer property of the material.

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 1.6.1 and 2.1). When two specimens are required, they shall be as nearly identical as possible, with thicknesses differing by less than 2 %.

The specimen(s) shall be of such a size as to cover completely the whole area of working surfaces of heating and cooling units and heat flow meter(s), and 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 3.4.2 also). They shall also meet the general requirements outlined in 1.7 and 1.8. The thickness of the test specimen(s) used shall be restricted according to 1.7.2.

3.2.2 Preparation and conditioning

3.2.2.1 General

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 materials (except loose-fill materials)

3.2.2.2.1 Preparation

The surfaces 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 working surfaces can be obtained. For rigid materials, the faces of the specimens shall be made as flat as the working surfaces in contact with them (see 2.2.1.1 and 2.2.2.4) and shall be parallel over the total surface area within 2 % of the sample thickness.

When the specimen is of rigid material and/or has thermal resistance smaller than 0,1 $m^2 \cdot K/W$, either thin sheets or temperature sensors mounted on the specimen shall be used to determine the temperature-difference across the specimen as indicated in 2.2.3.1.2.

The thermal resistance of the inserted thin sheets, when used, shall be not larger than one-tenth of the thermal resistance of the specimen. The resistance of the composite sandwich (sheet/rigid specimen/sheet) will then be determined using the temperature drop indicated by the permanent thermocouples in the hot and cold working surfaces. The number of uniformly distributed thermocouples used on each side of the specimen in the area contiguous to the metering area (see 2.2.3.1.1) should be not less than $10\sqrt{A}$, or 2, whichever is the greater, where A is the area in square metres of one side of the metering area. If separate thermocouples are used, the effective thickness of the specimen shall be taken as the average distance, perpendicular to the face of the specimen between the centres of the thermocouples on the two sides. For the type and placement of the thermocouples, see 2.2.3.1.

3.2.2.2.2 Conditioning

After determination of the mass of the specimen(s), they must be conditioned to constant mass in a desiccator or a ventilated oven at an appropriate temperature for the material or at the temperature specified in the material specification. Thermally sensitive materials should not be exposed to temperature that will change the specimens untypically. 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 absorbent or adsorbent is used. One example is a sealed desiccator at 330 K to 335 K with moving air for conditioning of certain foam plastics.

The 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 to 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 put quickly into the apparatus at the end of drying to prevent moisture pick-up. In the opposite situation (for example when testing specimens of low-density fibrous materials or of plastic foams) it is suggested that the conditioning be continued, leaving the specimen in a room at the standard laboratory atmosphere [temperature (296 ± 1) K; relative humidity (50 ± 10) %] to reach the equilibrium with room air (constant mass). In intermediate situations (for example with some high-density fibrous materials), judgement of the conditioning procedure is left to the experience of the operator.

After conditioning to a constant mass, the specimen(s) shall be both cooled and stored in a sealed desiccator or in a sealed, partially evacuated, polyethylene bag. The specimens shall be removed, weighed, and installed in the apparatus immediately before testing. To reduce test time, the specimen(s) may be conditioned to an appropriate mean 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 causes significant specific thermal resistance between the specimen and the apparatus, the envelope shall be treated similarly to the thin sheets used to test rigid specimens, as described in 2.2.3.1.2.2.

3.2.2.3 Guidelines for loose-fill materials

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, grain, etc. are rigid. When the requirement cannot be fulfilled alternate test methods 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 to constant mass 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 either 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 surfaces of the hot and cold plates and heat flow meter(s) in contact with specimen(s).

3.2.2.3.1 Method A

This method is suggested when operating the apparatus vertically. Set up the heat flow meter apparatus with the required spacings between the two (or four) measurement surfaces. These spacings of small cross-sectional area should be made of low-thermal-conductivity materials.

Place a thin layer of low-conductivity material that is suitable to confine the sample around the outer edges of the heat flow meter(s) and the plates such that it forms one (two) container(s) (each) open at the top. 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 into position until it occupies its appropriate one-quarter volume of the space, and taking care to produce specimen(s) of uniform density.

3.2.2.3.2 Method B

This method is suggested when operating the apparatus horizontally. Use a (two) shallow container(s) of thin-walled low-conductivity material having outside dimensions the same as those of the heating unit. The container edges shall be of such width as to make the depth of the container equal to the thickness of the specimen to be tested. Place spacers of small cross-sectional area, made of low-thermal-conductivity materials, with a thickness equal to the test thickness in the corners of the container or containers to ensure that the spacing between the covers for the frame is equal to the test thickness.

Make covers for the open faces of the container(s), 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). In either case, these shall be glued or otherwise fastened to the edges of the container(s). The total hemispherical emittance of the surfaces seen from the specimen shall be 0,8 or greater at operating temperatures. If the covers have significant thermal resistance, the method of determining the net specimen thermal conductance 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 container(s) lying horizontally on a flat surface, place a portion in the (each) container, 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 heat flow meter.

Fluff compressible materials during placement, so that the covers bulge slightly and will make good contact with the working surfaces of the apparatus at the desired density.

For some materials, material loss during preparation of the specimens may necessitate reweighing before test; in this case, determine the mass of the conditioned container and covers after the test to compute the as-tested density of the material.

3.3 Test method

3.3.1 Mass

Just before placing 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 (see note 2 below) is either the thickness imposed by positioning the units and heat flow meter(s) or the thickness of the specimen(s) as measured at the beginning of the test. Specimen(s) thickness can be measured as indicated in 2.2.3.3 or outside the apparatus with instrumentation that will reproduce the pressure on the specimen(s) 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.

Blanket or batt-type materials are usually tested at imposed thickness; material specifications define this thickness for many materials but sometimes the result of the measurements is merely a conventional one, 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 is 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 2.2.3.1.2.2 is used, the thickness to be used to evaluate heat transfer properties shall be adjusted making appropriate allowance for the position of the thermocouples.

NOTE 2 For calculation of the as-tested volume, use the figures obtained for lateral dimensions of the specimen by the lateral dimension test method described in the material specification or other appropriate method giving accuracy of the same order as that for thickness measurement.

3.3.3 Selection of temperature difference

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

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

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

c) As low as possible, for example from a minimum of 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) shall be reduced to a minimum; this may imply non-compliance to this International Standard and shall be noted in the report (see 3.6.19).

3.3.4 Ambient conditions

According to the type of apparatus and the test temperature, apply edge insulation and/or ambient specified conditions as required in 2.2.5.

3.3.5 Heat flow-rate and temperature measurements (settling time and measurements)

3.3.5.1 Observe the mean temperature and the emf output of the heat flow meter, the mean temperature and the temperature drop across the specimen(s) to check when they are stabilized.

3.3.5.2 The factor $\rho c_s dR$ of the specimen plays a major part in determining the time required for the meter output to reach equilibrium, as does the construction of the apparatus (see [8]). Intervals between readings may need only be one-tenth of that specified above for many tests. Experimental correlations are recommended (see annex B). In the absence of a better approximation or experience with similar specimens in the same apparatus, the above rule shall be used. Make observations at intervals equal to the value of $\rho c_s dR$ of the specimen or 300 s, whichever is greater, until five successive observations yield a thermal resistance value agreeing to within 1 % without changing monotonically in one direction. d is the as-tested specimen thickness in metres and ρc_s is the product of as-tested density and specific heat of the specimen, in joules cubic metre Kelvins. This product is to be estimated through knowledge of the material.

3.3.5.3 Monitoring of the evolution of the heat flow meter output function of time can be helpful to check the stability of the equilibrium, particularly with an unknown type of material or when there is some doubt about risks of sensitivity to environmental humidity of the material tested.

If this output varies by more 1,5 % in respect of its mean value, the operator shall make investigations to discover the reasons.

3.3.5.4 After reading of the equlibrium and when the procedure in 2.2.3.1.2.2 is used, determine the temperatures indicated by the thermocouples installed in the faces of the specimen(s).

3.3.6 Final mass and thickness measurements

Upon completion of the observations in 3.3.5, measure the mass of the specimen(s) immediately. In a case where the specimen thickness is not imposed, it is strongly recommended to measure it as at the beginning of the test. Report also any specimen(s) 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 selected from the same sample so that they have as widely different structures as possible 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 variation in structure occurs over small distances, it may be possible to use single specimens cut larger than the lateral dimensions of the plates and heat flow meter(s).

The specimen is tested twice, in each case carefully positioned so that the edges of the test area are exposed to the two extremes in structure. The two results are 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. This will 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 test area, the smaller the effect on the result. The effects 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 connecting 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 shall be ground as flat as the plate surfaces (see 2.2.1.1).

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.

It is also possible to evaluate the effect of thermal field distortions through the use of analyses 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 thermal 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 thickness 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:

$$\rho_d = M_2/V$$

$$\rho_s = M_3/V$$

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 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 drying, m_r, or due to a more complex conditioning procedure, m_c,

$$m_r = (M_1 - M_2)/M_2$$

$$m_c = (M_1 - M_3)/M_3$$

where

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

M_2 and M_3 are defined in 3.5.1.1.

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

$$m_d = (M_3 - M_2)/M_2$$

Calculate the relative mass gain, m_w, of the specimen during the test as follows:

$$m_w = (M_4 - M_5)/M_5$$

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, in kilograms.

3.5.2 Heat transfer properties

To make all the computations, use average values of the observed steady-state data. The five measurements described in 3.3.5 shall be used as data in these calculations. Additional measurements may be used so long as they do not differ from any of the other five by more than 1 %.

3.5.2.1 Single-specimen configuration

3.5.2.1.1 Single heat flow meter configuration

Calculate the thermal resistance, R, of the specimens as follows:

$$R = \frac{\Delta T}{f e}$$

where

f is the calibration factor of the heat flow meter, in watts per millivolt per square metre;

e is the heat flow meter output, in millivolts.

If applicable (see 1.8.2 and 1.8.3), compute thermal conductivity λ or thermal resistivity r from the following equation:

$$\lambda = \frac{1}{r} = f e \frac{d}{\Delta T}$$

where d is the average specimen(s) thickness.

3.5.2.1.2 Two heat flow meter configuration

All the requirements in 3.5.2.1.1 are applicable to this configuration, with fe replaced by $0{,}5\ (f_1e_1 + f_1e_2)$ where the indices 1 and 2 refer to the first and second heat flow meters respectively (of which the surface temperatures are respectively T_1 and T_2).

3.5.2.2 Two-specimen configuration

Calculate the total thermal resistance, R_t, as follows:

$$R_t = \frac{1}{fe}(\Delta T' + \Delta T'')$$

and, if applicable as in 3.5.2.1, the average thermal conductivity λ_{avg} or thermal resistivity r_{avg} as follows:

$$\lambda_{avg} = \frac{1}{r_{avg}} = \frac{fe}{2}\left(\frac{d'}{\Delta T'} + \frac{d''}{\Delta T''}\right)$$

where the symbols are the same as above and the subscripts refer to the two specimens (′ for the first specimen, ″ for the second specimen).

3.6 Test report

If results are to be reported as having been obtained by this method, then all pertinent requirements 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. Conformity to a material specification where applicable. Method of specimen preparation for loose-fill materials with indication of the measured resistance of cover materials used for the containers.

3.6.3 Thickness of the specimen(s) as tested expressed in metres. In a two-specimen configuration, this is the total thickness of the two specimens. Specify whether thickness is imposed or measured. Criteria to define the imposed thickness.

3.6.4 Method and temperature of conditioning.

3.6.5 Density of the conditioned specimen(s) as-tested.

3.6.6 Relative mass changes during drying and/or conditioning (see 3.5.1.2).

3.6.7 Relative mass change during test (see 3.5.1.2). Observed thickness and volume changes during test (see 3.3.6).

3.6.8 Average temperature difference across the specimen(s) during the test as computed from the temperatures of the hot and cold working surfaces and procedures for its determination (see 3.3.5).

3.6.9 Mean temperature of test, kelvins or degrees celsius.

3.6.10 Density of heat flow rate through specimen at the equilibrium, watts per square metre (see 3.3.5).

3.6.11 Thermal resistance, square metre kelvin per watt, of specimen(s). Where applicable, the thermal resistivity, metre kelvins per watt, thermal conductivity, watts per metre kelvin, and range of thickness for which these values have been measured or are known to apply.

3.6.12 Type of heat flow meter apparatus used, with one or two specimens. Method to reduce edge heat losses and the environment temperature surrounding the plates during test. Number and position of heat flow meter(s).

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

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. If temperature sensors were used to determine the temperature difference in the specimen, information shall be given on the solution adopted.

3.6.15 Date of the test, the date of the last heat meter calibration, and the type, or types, of materials used.

3.6.16 Duration of the full test and of the steady state-part of the test if this information can help in interpreting results.

3.6.17 The specimens used in calibration must be identified as to type, thermal resistance, date of specimen certification, source of certification, expiration date of calibration, and the certification test number.

3.6.18 Estimation of errors: a statement on the maximum expected error in measured property is strongly recommended within the report; when one or more requirements stated in this International Standard are not fulfilled (see also 3.6.19), it is recommended to include a complete report on the estimation of error or errors on the measured property.

3.6.19 Statement of compliance: 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 reports. A suggested wording is: "This test met all requirements of ISO 8301 Standard Test Method with the exception of ... (a complete list of exceptions)."

For direct-reading apparatus, the results of the calibration of electronic circuitry and equipment, or a statement of compliance including date, and a statement of compliance on linearity requirements shall be included.

Annex A
(normative)

Limit values for apparatus performance and testing conditions

Clause	Description	Value
1.1.1	Minimum measurable thermal resistance in a heat flow meter apparatus (HFM)	0,1 $m^2 \cdot K/W$
1.5.3.1	Expected reproducibility with specimen maintained within the apparatus	better than 1 %
1.5.3.1	Expected reproducibility with specimen removed and mounted again after long time intervals	better than $\pm$ 1 %
1.5.3.2	Expected accuracy of the calibration of HFM method (when mean temperature of the test is near the room temperature)	$\pm$ 2 %
1.5.3.3	Expected HFM method accuracy (when mean temperature of the test is near the room temperature)	$\pm$ 3 %
1.5.4.1	Suggested time limit to check the stability of calibration standards	5 years
1.5.4.2	Suggested calibration time intervals for the HFM method	24 h before or after the test
1.5.4.2	Calibration intervals if short and long-term stabilities of the HFM have been proved to be better than $\pm$ 1 %	15 d to 30 d
1.5.4.2	Upper acceptable limit in calibration stability	$\pm$ 1 %
1.8.2	Upper acceptable dimension limit of any non-homogeneity of the specimen	$\frac{1}{10} d$
1.8.3.1	Upper acceptable limit for transfer factor changes with thickness to assign thermal conductivity or thermal transmissivity to the material	2 %
2.2 ; 2.3.5	Minimum thermal hemispherical emittance for any surface in contact with the specimen	0,8
2.2.1.1	Maximum departure from a plane of working surfaces of heating and cooling units	0,025 %
2.2.1.2	Required heating unit temperature uniformity related to temperature difference across the specimen	1 %
2.2.1.2	Maximum error in measured heat flow-rate when HFM is placed in contact with the working surface of a heating or cooling unit	0,5 %
2.2.1.2	Required stability of the working surface temperature of the cooling and heating units during the test period, related to the temperature difference across the specimen	0,5 %
2.2.1.2	Required stability of the face of the HFM in contact with the specimen related to the temperature difference across the specimen	less than 0,5 %
2.2.1.2	Maximum allowed fluctuations in the electrical output of the HFM related to the temperature fluctuations at the surface of the HFM	2 %
2.2.2.3	Suggested maximum diameter for the cross-sectional area of the conductors in the thermopile	0.2 mm
2.2.2.3	Minimum HFM output without use of special techniques to prevent extraneous thermal emf's in leads, the measuring circuits and the HFM itself	0,000 2 V
2.2.2.3	Required ratio between metering area and the total surface of the HFM	10 % $\leqslant A \leqslant$ 40 %
2.2.2.4	Maximum departure from a plane of the metering area of the HFM	0,025 %
2.2.2.5	Suggested thickness of a metal or non-metal foil to be used to cover the metering area	80 µm
2.2.2.5	Suggested diameter for thermocouples used as surface temperature sensors of the HFM	0.2 mm
2.2.3.1.1	Required accuracy in the measurement of temperature difference between heating and cooling units in contact with specimen	1 %

Clause	Description	Value
2.2.3.1.1	Minimum number of temperature sensors on each side of the working surfaces of heating and cooling units	$10\sqrt{A}$ or 2
2.2.3.1.1	Minimum electrical resistance of the insulation between thermocouples and apparatus metal plates	1 MΩ
2.2.3.1.2.1	Minimum thermal resistance for non-rigid specimens to use permanently mounted temperature sensors in the working surfaces of cooling and heating units	0,5 $m^2 \cdot K/W$
2.2.3.1.3	Maximum thermocouple diameter when mounted in the surface of the plates to measure temperature differences between heating and cooling units	0,6 mm
2.2.3.1.3	Suggested maximum thermocouple diameter when mounted as above in the surface of small size apparatus	0,2 mm
2.2.3.1.3	Suggested maximum thermocouple diameter when placed against or set into the surfaces of the specimens	0,2 mm
2.2.3.1.3	Maximum resulting error in measurements of temperature differences due to distortion of the heat flow rate around the sensor, to sensor drift, etc.	less than 1 %
2.2.3.2.2	Required accuracy of electrical measurements of temperature differences across the specimen(s)	± 0,5 %
2.2.3.2.2	Required accuracy of electrical measurements of the output from the HFM	± 0,6 %
2.2.3.3	Required accuracy in the measurement of specimen thickness	0,5 %
2.2.4.2	Maximum suggested apparatus pressure on the specimen for most insulating materials	2,5 kPa
2.2.5.1	Minimum required difference between air dew point and cooling unit temperature	5 K
2.2.5.3	Maximum value for the edge heat loss error	0,5 %
2.3.2	Suggested ratio between the side of the HFM metering area and the maximum specimen thickness	4
2.3.2	Suggested ratio between the external side of the HFM and the maximum specimen thickness	8
3.2.1	Maximum thickness difference for two specimens to be mounted in a two-specimen apparatus	2 %
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 for rigid specimens to measure temperature difference across the specimen either with thin sheets or temperature sensors mounted on the specimen	0,1 $m^2 \cdot K/W$
3.2.2.2.1	Maximum resistance of interposed sheets with respect to the specimen resistance	0,1
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	Minimum suggested ratio between specimen thickness and mean dimension of beads, grains, flakes, etc.	10, better 20
3.2.2.3.2	Maximum thickness for plastic sheets in method B for loose-fill materials	50 μm
3.2.2.3.2	Total hemispherical emittance of the surfaces seen from the specimen at operating temperature	0,8 or greater
3.3.1	Required accuracy in the determination of specimen mass	0,5 %
3.3.3	Lower limit for temperature differences across the specimen when determining an unknown relationship between temperature and heat transfer properties	5 K
3.3.3	Upper recommended limit for temperature differences across the specimen as above	10 K
3.3.5.2	Maximum thermal resistance change in five successive sets of observations to assess steady-state attainment	1 %
3.3.5.3	Upper acceptable variation function of time of the HFM output in respect of its mean value	1,5 %