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Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 1: General principles

Plastiques — Détermination de la conductivité thermique et de la diffusivité thermique —

Partie 1: Principes généraux

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 22007-1:2009), which has been technically revised.

A list of all parts in the ISO 22007 series can be found on the ISO website.

Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 1: General principles

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory requirements.

1 Scope

This document describes the background to methods for the determination of the thermal conductivity and thermal diffusivity of polymeric materials. Different techniques are available for these measurements and some may be better suited than others for a particular type, state and form of material. This document provides a broad overview of these techniques. Standards specific to these techniques, as referenced in this document, are used to carry out the actual test method.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
heat pulse
heat change in the form of a pulse produced by a heat source

3.2
heat pulse energy
amount of heat produced by a heat source within the heat pulse

Note 1 to entry: It is expressed in joules (J).

3.3
heat source
heater in the form of a wire, strip, plate or foil embedded within or attached to a test specimen or an area irradiated by incident light, e.g. a laser

3.4
heat flux
q
heat source output produced by a planar source per unit time and unit area

Note 1 to entry: It is expressed in watts per square metre (W/m^2).

3.5
linear heat flow
heat source output produced by a linear source per unit time and unit length

Note 1 to entry: It is expressed in watts per metre (W/m).

3.6
penetration depth
characteristic depth used for describing the extent of heat penetration into the specimen during a transient measuring process

Note 1 to entry: It is expressed in metres (m).

3.7
temperature transient
temporary perturbation of temperature in a system initially at a uniform temperature due to a heat pulse for a period during which the system does not attain equilibrium

3.8
volumetric heat capacity
product of the density and the heat capacity

Note 1 to entry: It is expressed in joules per cubic metre kelvin [$J/(m^3 \cdot K)$].

3.9
thermal effusivity
b
heat transport property given by the square root of the product of thermal conductivity and volumetric heat capacity:

$$b = \sqrt{\lambda \cdot \rho \cdot c_p} \tag{1}$$

where

λ is the thermal conductivity in watt per metre kelvin [$W/(m \cdot K)$];

ρ is the density in kilogram per cubic metre [kg/m^3];

c_p is the heat capacity in joule per kelvin kilogram [$J/(K \cdot kg)$]

Note 1 to entry: It is expressed in joules per square metre kelvin square root second [$J/(m^2 \cdot K \cdot s^{1/2})$].

3.10
thermal resistivity
reciprocal of thermal conductivity

Note 1 to entry: It is expressed in metre kelvins per watt [$(m \cdot K)/W$].

4 Principles

Thermal conductivity refers specifically to the mode of heat transfer via conduction. In thermal conductivity measurements, other modes of heat transfer, such as convection, radiation and mass transfer, may occur. Where these modes are significant, the measured property is usually referred

to as apparent or effective thermal conductivity. Thermal conductivity is affected by the conditions under which it is measured, such as temperature and pressure, as well as compositional variation of the material and orientation of the specimen since some materials are not isotropic.

In steady-state methods, an appropriately sized specimen of simple geometry in contact with a heat source, together with one or more temperature sensors, which may be combined with the heat source or separate from it, is allowed to equilibrate at a given temperature. Transient methods may be contact or non-contact. A thermal transient is produced by a heat pulse to generate a dynamic temperature field within the specimen. The temperature change with time (temperature response) is measured by one or more sensors which may be combined with the heat source, placed at a fixed distance from the source or, as in the case of the laser flash method, located on the other side of the specimen. For measuring very thin films (with thicknesses in the nm range), the thermal reflectance method – an ultra-fast variant of the laser flash analysis – is well suited. Two modes are available: rear heating/front detection and front heating/front detection [16]. In any case the response is analysed in accordance with a model, and a set of solutions developed for the representative set-up and designed for the specific geometry and the assumed boundary conditions. Depending upon the geometry of the specimen and source and the means of generating the temperature field, one or more thermo-physical properties can be obtained, either separately or simultaneously. Table 1 contains a summary of the characteristics of different types of transient methods and the properties that may be determined by their use.

NOTE 1 Most unfilled plastics fall into the category of materials of intermediate thermal conductivity (0,1 W/m · K to 1 W/m · K). They are an order of magnitude more conductive than foams and insulation but less conductive than ceramics and glass. Their thermal conductivity can increase dramatically if fillers are added. A variety of test methods may be used, depending on the form and state of the plastic. An overview of these methods is given in Clause 5. Detailed test methods are contained in other parts of ISO 22007 and in other standards referenced.

NOTE 2 Reference materials are necessary to verify the performance of primary methods and to calibrate secondary methods. A number of solid materials have been characterized by national standards laboratories, such as NPL, NIST, LNE, NMIJ and PTB, but currently only poly (methyl methacrylate) and glass fibre board IRMM-440 and glass ceramic BCR-724[1)] have a thermal conductivity which is in the same range as those of most polymer and polymer-filled materials. Polydimethylsiloxane and glycerol are well characterized fluid reference materials with thermal conductivities in the same range as those of plastics.

NOTE 3 The thermal conductivity λ can be obtained by multiplying the thermal diffusivity α with the specific heat capacity at constant pressure c_p and the density ρ, i.e. $\lambda = \alpha \cdot c_p \cdot \rho$.

Table 1 — Basic characteristics of transient methods

Type of method	Heat source/ heat source geometry	Mode of heat generation	Heat source/temperature sensor configuration	Measured and/or derived parameters
Hot wire/line source /hot strip	Contact/Line, strip	Step-wise	Combined[a] or separate[b]	λ, α (c_p and b in some versions of the method)
Pulse transient	Plane	Pulse	Separate	α, c_p, λ
Transient plane source	Contact/Plane	Pulse, step-wise	Combined	α, c_p, λ
Laser or light flash	Laser, Xenon lamp/Plane	Pulse	Separate	α, c_p, λ

λ = thermal conductivity; α = thermal diffusivity; b = thermal effusivity; c_p = specific heat

a One sensor.

b Two sensors.

Annex A provides information on sources of uncertainty on measuring thermal transport properties.

1) Glass fibre board IRMM-440 and glass ceramic BCR-724 are products supplied by the Joint Research Centre (JRC) of the European Commission. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the products named.

5 Test methods

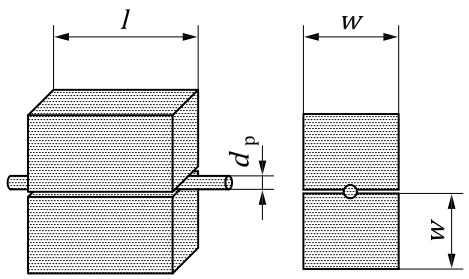
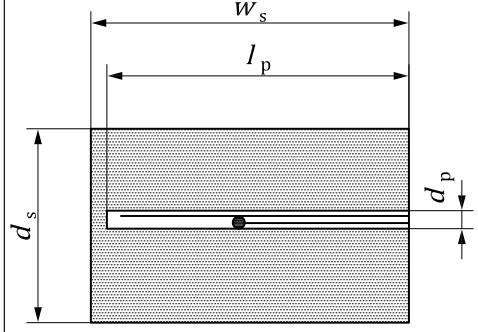
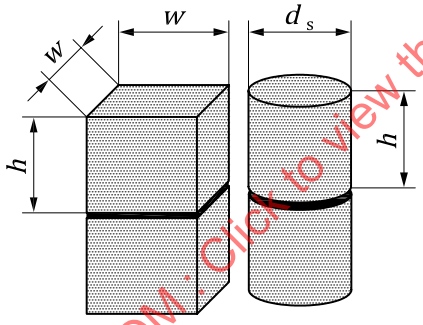
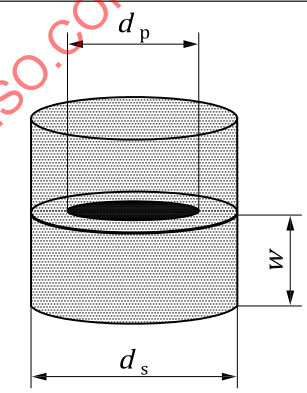
5.1 General

A number of test methods have been developed to provide a means of measuring thermal conductivity and thermal diffusivity based upon the basic principle outlined above. An overview of these methods is given in the following subclauses. Some of the contact methods are summarized in Table 2 and then further explained in more detail. Complete details of the contact and non-contact test methods described in 5.4 to 5.6 can be found in ISO 22007-2, ISO 22007-3, ISO 22007-4 and ISO 22007-6.

In contact methods, the accuracy of the measurement result depends strongly on a good thermal contact between the sensor and the sample. Enough uniaxial pressure should therefore be applied to press the various parts of the specimen and the heat source together.

NOTE In some cases heat sink pastes are used to improve thermal contact, but the user should be aware that it may contribute to the uncertainty of measurement and their effect should be adequately quantified for accurate results. Too much paste and application in wrong places (for example outside the heater area) should be avoided.

Table 2 — Schematic diagrams of various transient experimental methods showing critical dimensions

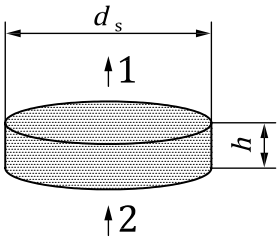
Method	Specimen set-up	Characteristic parameters	Ideal model
Hot wire[a]		l = specimen length w = specimen width, thickness d_p = wire probe diameter	$200d_p < w$ $l > 4w$
Line source[a]		w_s = active zone l_p = probe length d_p = probe diameter d_s = specimen diameter	$w_s > 1{,}5l_p$ $l_p > 33d_p$ $d_s > 6d_p$
Hot plate[b]		w = width, thickness h = height d_s = specimen diameter	$w, h, d_s > 3\sqrt{\alpha t_{max}}$ where t_{max} is the maximum measurement time
Transient plane source[b]		d_p = heat source diameter d_s = specimen diameter w = specimen thickness	$d_s - d_p > 4\sqrt{\alpha t_{max}}$ where t_{max} is the maximum measurement time

a Unless the specimen is a liquid, a suitable groove or hole has to be made for the hot wire or line source.

b Good thermal contact has to be established between the strip or disc and the specimen.

c Round or rectangular sample geometries are possible.

Table 2 *(continued)*

Method	Specimen set-up	Characteristic parameters	Ideal model
Laser or light flash[c]		h = specimen thickness d_s/h = ratio between specimen diameter (d_s) and thickness (h) 1 = IR detector 2 = power source (laser or xenon lamp)	$d_s/h > 5$ The diameter d_s or side length of the sample shall be > 10 mm

[a] Unless the specimen is a liquid, a suitable groove or hole has to be made for the hot wire or line source.

[b] Good thermal contact has to be established between the strip or disc and the specimen.

[c] Round or rectangular sample geometries are possible.

5.2 Hot-wire method

This method can be used to determine the thermal conductivity of polymers as a function of temperature. It is applicable only to isotropic materials, but in any form, e.g. plates, foams, pellets or powders.

NOTE The hot-wire method is mainly used for solid polymers as the temperature-measuring element may be destroyed when working with molten polymers.

The hot-wire method is a transient method. A wire heater is placed in a test specimen or between two test specimens of the same material. The temperature rise is measured either by the wire itself acting as a platinum resistance temperature detector or by a thermocouple placed in close proximity to the wire. The heater current is switched on and the temperature rise is measured by the thermocouple as a function of time.

Starting with the Fourier differential equation, it is possible to describe the transient heat flow for an infinitely long wire as follows:

$$\Delta T(r,t) = -\frac{\phi}{4\pi L\lambda}\mathrm{Ei}\left(-\frac{r^2}{4\alpha t}\right) \tag{2}$$

where

t is the time, in s;

ϕ is the rate of heat flow generated by the wire, in W;

r is the distance between the heater and the thermocouple, in m;

L is the length of the wire, in m;

λ is the thermal conductivity, in W/(m·K);

α is the thermal diffusivity, in m^2/s ($\alpha = \lambda/\rho C_p$);

Ei(x) is the exponential integral, given by:

$$-\mathrm{Ei}(x)=\int_{x}^{\infty}\frac{e^{-u}}{u}du \tag{3}$$

For values of $r^2/4\alpha t$ less than 1, Formula (2) can be simplified to:

$$\Delta T(r,t)=-\frac{\phi}{4\pi L\lambda}\ln\frac{4\alpha t}{r^2 C} \tag{4}$$

where

$C = e^{\gamma}$

where γ is Euler's constant (= 0,577 216).

According to Formula (4), the variation in the temperature, $\Delta T(r,t)$, is a linear function of the natural logarithm of time, and the thermal conductivity of the sample can be determined using Formula (5):

$$\lambda=\frac{\phi}{4\pi LK} \tag{5}$$

where K is the slope of the linear part of the curve of temperature variation plotted against the natural logarithm of time.

With the correct specimen and heater dimensions as indicated in Table 2, Formula (5) can be used for practical applications.

Details of the test method can be found in ISO 8894-1[12] and ISO 8894-2[13] and ASTM C1113[19].

5.3 Line-source method

This technique[2], sometimes called a needle-probe method, is a variant of the hot-wire method. It uses a line-source probe in the form of a needle, which permits repeated measurements of thermal conductivity to be made without destruction of the sensor. This transient method is capable of very fast measurements and is suited to both melt and solid-state thermal-conductivity measurements. It is not suited to the measurement of directional solid-state properties in anisotropic materials.

A line source is located at the centre of the specimen being tested. Both the line source and specimen are kept at a constant initial temperature. During the course of the measurement, a known amount of heat is produced by the line source, resulting in a heat wave propagating radially into the specimen. The governing Formulae are the same as those for the hot-wire method. The line source takes the form of a needle-sensor probe of finite length and diameter. Typical probes are 50 mm to 100 mm long and about 1,5 mm to 2 mm in diameter and contain a heater element that runs the whole length of the needle. A thermocouple sensor located within the needle, with its sensing point half-way down the length of the probe, measures the temperature rise associated with the transient. Deviations from the model, such as the finite probe dimensions, require the probe to be calibrated against a reference material. A probe constant, C, is introduced into Formula (5); it is the ratio of the actual thermal conductivity of the reference material to that measured by the instrument:

$$\lambda=\frac{C\phi}{4\pi LK} \tag{6}$$

NOTE 1 Silicone fluids and glycerol have been used as reference materials [3]. If using glycerol as a reference material, caution is advised since its properties are sensitive to moisture.

Typical transients show an initial non-linearity due to the heat wave propagating through the finite thermal capacity of the probe. This is a region of high conductivity and, hence, low slope. With typical melt state transients, where the specimen has no contact resistance, the transient approaches linearity directly after it overcomes this effect, typically within a few seconds. The slope of interest is the linear

region that follows the initial non-linearity. Acquisition durations typically range from 30 s to 60 s. This is very important in gathering melt state thermal-conductivity data because it dramatically reduces the possibility of thermal degradation.

NOTE 2 Scanning methods have been devised which permit the automated acquisition of data at different temperatures, so that measurements over a wide range of temperatures are possible. With such methods, the same specimen that was used for the melt state measurements can be used for solid-state measurements, thereby permitting measurements across the melt-to-solid transition.

Details of the test method can be found in ASTM D5930[14].

5.4 Transient plane source method

The transient plane source method is capable of measurements of the true bulk properties of materials with a wide range of thermal conductivities.

The technique[4] uses a thin, plane, electrically insulated resistive element as both the heat source and the temperature sensor to measure the thermal conductivity and the thermal diffusivity from one transient recording. This resistive-element sensor is brought into thermal contact with two halves of a specimen of the material under investigation. Each of the specimen halves shall have one flat surface so that the sensor can be fitted snugly between these surfaces.

By supplying constant electrical power to the sensor, which is of known radius, and by recording the increase in resistance as a function of time, it is possible to deduce both the thermal conductivity and the thermal diffusivity from one single transient recording. In order to be able to deduce both these heat transport properties from a single transient recording, it is important that the probing depth, Δp_{prob} – defined as $\Delta p_{prob} = 2(\alpha t)^{½}$, where α is the thermal diffusivity of the sample material and t is the total time of the transient – used for the test be larger than the radius but less than the diameter of the sensor.

The sensor can have different designs and be composed of different materials. A spiral pattern is in common use. Nickel and molybdenum have been used as sensing materials, with the sensing spiral and its connecting leads etched or cut out of a thin foil with a thickness of around 10 µm. Other sensing materials can be used, provided the sensing material has a reasonably large temperature coefficient of resistivity. The reason for this requirement is that the sensor is used not only for increasing its own temperature and that of the specimen near it, but also for recording the temperature changes.

To electrically insulate the sensing material, it is possible to use a variety of materials: so far thin sheets of a polymer (Kapton®[2]), a micaceous material and solid sapphire have been used. When selecting insulating sheets, it is important that these be kept as thin as possible, preferably in the range 25 µm to 100 µm, in order to guarantee good thermal contact between the sensing material and the flat surfaces of the surrounding specimen halves.

For analysing the transient recordings, the heat transfer equations have been solved for a number of concentric, circular line sources embedded in an infinite medium. To fulfil this condition in a test, the size of the specimen shall be such that the distance from any part of the sensor to the nearest outer surface of the specimen is not less than the probing depth. Sensors with diameters from 1 mm to 60 mm have so far been used successfully.

Details of the test method can be found in ISO 22007-2.

5.5 Temperature wave analysis method

The temperature wave analysis method describes a procedure[8],[9] for determining the thermal diffusivity in the thickness direction of a thin polymer film as a function of temperature. It can be used for both solid and molten polymers at a constant temperature or for a temperature scan. Measurements can be performed in ambient air or at reduced pressures.

2) Kapton is a registered trademark of DuPont. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

The principle of the method is to measure the phase shift of a temperature wave propagating in the through-thickness direction of a thin, flat specimen of thickness d, located between backing plates. For this purpose, electrical resistors are sputtered directly onto, or contacted with, each surface of the specimen, one as the heater for generating an oscillating heat wave and the other as the thermometer for measuring the oscillating temperature. If a one-dimensional heat flux is assumed and the specimen can be considered to be thermally thick (i.e. $kd > 1$), then the temperature change is given by:

$$T(d,t) = \frac{\sqrt{2} j_0 \lambda k \exp(-kd)}{(\lambda k + \lambda_b k_b)^2} \exp\left[\mathrm{i}\left(\omega t - kd - \frac{\pi}{4} \right) \right] \tag{7}$$

where

$T(d,t)$ is the temperature oscillation at the rear surface of the specimen;

t is the time;

j_0 is the periodical heat flux generated at the front surface of the specimen;

i is $(-1)^{1/2}$;

ω is the angular frequency;

λ is the thermal conductivity;

k = $(\omega/2\alpha)^{1/2}$, where α is the thermal diffusivity;

b subscript "b" refers to the backing plates.

The phase shift, $\Delta\theta$, between the heater and the sensor is described by

$$\Delta\theta = \sqrt{\frac{\omega}{2\alpha}} d - \frac{\pi}{4} \tag{8}$$

The phase shift, $\Delta\theta$, is a linear function of the square root of the angular frequency, ω, and thus the thermal diffusivity of the specimen can be determined from

$$\alpha = \frac{d^2}{2A^2} \tag{9}$$

where A is the slope of the linear part of the plot of phase shift versus the square root of the angular frequency.

Details of the test method can be found in ISO 22007-3.

5.6 Laser flash method

The laser flash technique is a non-contact, non-destructive method used for measuring the thermal diffusivity of homogeneous, isotropic and anisotropic solids, or liquids. Transparent materials can be measured provided that they are first coated with e.g. graphite (sprayed) to render them opaque. This method is based upon the measurement of the temperature rise at the back face of a thin-disc specimen caused by a short energy pulse on the front face. Due to the well-defined measurement direction, samples can be measured through-plane and in-plane. Alternative energy sources other than lasers can be used.

The specimen, typically 10 mm to 20 mm in diameter and 1 mm to 3 mm thick, is placed in a furnace and heated to a uniform temperature. Then, one face of the specimen is irradiated with a short (< 500 µs) laser or light pulse [10]. The temperature rise at the opposite specimen face is measured by an IR detector. The sensor is the same for all materials or sample dimensions. A high-speed recorder collects data representing the temperature rise. The diffusivity is calculated from the shape of the

temperature-time curve (thermogram) and the thickness of the specimen. The absolute values of the energy absorbed, of the temperature rise and of the emissivity of the specimen surface are not required for measuring the thermal diffusivity.

The thermal diffusivity is calculated by comparison of the experimental thermogram with a theoretical model which takes the heat losses between the specimen and its surroundings into account. Various models such as the partial time moments method [5], the "half-time" method by Parker[10] or the Cape Lehmann Model[23] can be used. The method by Parker was introduced in 1961, initially limited to isotropic materials and adiabatic conditions. Within the recent decades, further correction algorithms such as for heat loss, pulse length effects, etc., were integrated into the mathematical models.

$$\alpha = 0{,}1388 \cdot \frac{h^2}{t_{1/2}} \tag{10}$$

(formula according to the "half-time" method by Parker[10])

The thermal diffusivity increases with the square of the thickness h, whereas $t_{1/2}$ is the time representing half of the maximum temperature increase on the sample's rear side.

More details of the test method can be found in ISO 22007-4.

By measuring a reference material under the same conditions as the sample, the specific heat of the sample material can be calculated (ratio method according to ASTM E1461 — Appendix X2[20]).

5.7 Steady-state methods

5.7.1 Guarded hot-plate method

The guarded hot-plate method described in ISO 8302[11] and ASTM C177[18] is the reference technique for thermal conductivity measurements since it does not require calibration against a material of known thermal conductivity. It is a steady-state method based on achieving steady unidirectional heat flow through the thickness of a large, flat specimen. A temperature gradient across the specimen provides the driving force for heat transfer. According to the Fourier equation at the steady state,

$$Q = \lambda A \Delta T / d \tag{11}$$

where

Q is the heat flow rate, in W;

λ is the thermal conductivity of the specimen, in W/(m · K);

A is the cross-sectional area of the specimen, in m^2;

ΔT is the temperature difference across the specimen, in K;

d is the thickness of the specimen, in m.

Instrument configurations using either two identical specimens placed symmetrically about the principal heater plate or a single specimen on one side of the principal heater plate are possible. The principal heater is used to generate a steady temperature gradient through the specimen, with the heat sink side of the specimen having either a heater or a chiller to control its temperature. Guard heaters are used to achieve unidirectional heat flow through the thickness of the specimen. Measurements can also be performed in different gas environments or under vacuum conditions.

Temperature measurements on each side of the specimen (in the through-thickness direction) are made to determine the temperature difference across the specimen. When measuring plastics, the temperature sensors are mounted on or in the specimen surface, and thermal-contact sheets can be used between the apparatus plates and the specimen to improve heat transfer, which may be poor

due to differential specimen expansion. The heat flow through the specimen is determined from the electrical-power input to the principal heater.

Specimens are typically round or square sheets, 200 mm or more in diameter or length of side. ISO 8302 specifies that specimens should be at least 20 mm thick. Steady temperature and heater-voltage readings indicate thermal equilibrium. The time taken to reach thermal equilibrium is typically about 4 h to 12 h, although this varies with test temperature and specimen thickness. Measurements of thermal conductivity in the range 0,001 W/(m · K) to 2,0 W/(m · K) are typical.

Details of the test method can be found in ISO 8302[11].

5.7.2 Guarded heat flow meter method and heat flow meter method

This quasi-steady-state method is a variant of the guarded hot-plate technique. It uses a heat flux transducer to measure the heat flow rate through the specimen rather than calculating it from the electrical power applied to the principal heater, as is the case in the guarded hot-plate method. Heat flux transducers are, typically, thermopiles that produce an output proportional to the heat flux. They thus provide a means of directly measuring heat flux. However, the heat flux transducer shall be calibrated using materials of known thermal conductivity. Calibration establishes a relationship between the voltage signal of the transducer and the heat flow through it.

NOTE Materials used for calibration include glass fibre board IRMM-440 and glass ceramic BCR-724, available from the Institute for Reference Materials and Measurements (IRMM), https://irmm.jrc.ec.europa.eu/[3)] and NIST 1450d (a fibrous glass fibre board available from the National Institute for Standards and Technology, https://www.nist.gov/).

Specimens are typically in the form of discs 50 mm in diameter and 1 mm to 20 mm thick. During the test, a specimen is held under a compressive load between two polished metal surfaces. The upper surface is the heat source which is maintained at the test temperature. The lower surface constitutes the calibrated heat flux transducer that is attached to a liquid-cooled heat sink maintained at a constant temperature. The temperature drop across the specimen is determined from temperature sensors in the metal surfaces on either side of the specimen. The time required to reach the steady state prior to making measurements is typically about 2 h for tests at near-ambient conditions. The amount of heat that flows from the hot plate to the cold plate is determined by the thermal conductivity and thickness of the specimen as given by the Fourier equation:

$$Q/A=\lambda(T_h - T_c)/d \quad (12)$$

where

Q/A is the heat flux through the specimen, in W/m^2;

λ is the thermal conductivity, in W/(m·K);

T_h and T_c are the hot-plate and cold-plate temperatures, respectively, in °C;

d is the specimen thickness, in m.

The thermal conductivity is calculated from the calibration factor, the specimen thickness and the temperature drop across the specimen. Such instruments can measure thermal conductivities in the range from 0,1 W/(m · K) to 10 W/(m · K) over a temperature range from –173 °C to above 200 °C, depending on the design of the apparatus. Thermal-contact resistance shall be considered with the heat flow meter. Some of its effects can be calibrated out by applying the same load to the test stack as was used during calibration of the instrument. Further, a thermally conductive paste can be applied, if needed.

Details of the test method can be found in ASTM E1530[15].

3) This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product or supplier named.

The heat flow meter method is a variation of the guarded heat flow meter method, but without guard rings. It is again a relative or secondary measurement method as also here specimen of known thermal transmission properties shall be used for calibration. Suitable calibration materials are identical to the ones mentioned beforehand.

Specimens are typically square slabs and shall ideally cover the complete plate assembly surfaces, but at least the metering area (perhaps in combination with a mask of similar thermal conductivity compared to that of the sample).

The thermal resistance (or thermal conductance) of the sample is important for selecting a proper temperature difference between the hot plate and cold plate. The time taken to reach thermal equilibrium within a measurement is typically about 15 min to hours and varies with test temperature and specimen properties. The thermal conductivity range which can be measured with heat flow meter instruments is typically 0,005 W/(m · K) up to 1,0 W/(m · K), and can be expanded to 2,0 W/(m · K) with appropriate auxiliary means.

Details of the test method can be found in ISO 8301[21] and ASTM C518[22].

6 Test report

When using subsequent parts of ISO 22007, the test report shall contain the following information:

a) a reference to the part of ISO 22007 used;

b) the date of the test;

c) identification of the sample tested (type, batch number, etc.);

d) sample details and method of specimen preparation, including the thermal history;

e) the manufacturer of the instrument used, as well as the model and type of instrument;

f) the test temperature(s);

g) the measurement technique used and all relevant details as required by the part of ISO 22007 used;

h) the thermal conductivity and/or thermal diffusivity (to three significant digits) plus other properties, if measured;

i) details of any deviations from the conditions or materials specified in the part of ISO 22007 used.

Annex A
(informative)

Sources of uncertainty on measuring thermal transport properties

A.1 General

A.1.1 Current experience indicates that low measurement uncertainties are readily attained and, for some transient techniques, information on the bulk properties of solid materials can be obtained. Such Information normally requires special care with steady-state methods if possible to retrieve at all.

A.1.2 The major factors affecting the uncertainty of measurement of any transient method are the length of time required for the temperature field to develop inside the specimen and the heat source geometry: both factors affect the development of the temperature field. The optimum experimental set-up requires the specimen size to be such that the temperature field generated by the heat source deviates as little as possible from that of the theoretical model during the measurement period.

A.1.3 An essential criterion for accurate measurements is an undeformed temperature field. Two differences exist between the theoretical model and a real situation, namely the finite size of the specimen and the fact that the construction of the heat source is necessarily different from that of an ideal one. The ideal heat source has negligible thickness, is constructed from a single material and provides perfect thermal contact.

A.1.4 Three thermophysical parameters can be determined when a two-probe system is used. Three parameters can also be determined when a disc heat source, i.e. a single heater/probe system, is used.

A.2 Individual sources of uncertainty

The individual contributions to the measurement uncertainty can be summarized as follows:

a) The degree to which the experimental arrangement does not represent the theoretical model. Of importance to consider are:

   1) the sensitivity to the boundary conditions;

   2) the extent to which the number of parameters required to represent the model can be minimized and their accuracy maximized.

b) External parameters:

   1) the contact resistance [surface thermal resistance (STR)] between heat source and specimen, between specimen and sensor and for the heat source/sensor combination, when these are combined;

   2) contributions due to uncertainties in the measurement parameters, including:

      — the power levels used for different ranges of thermal properties and specimen sizes,

      — the time interval between heat pulses.

c) Material/specimen parameters:

   1) specimen size and configuration;

2) anisotropy of thermal properties;

3) effects in which other heat transfer mechanisms, including radiation, convection and mass transfer, may be present, thus affecting the validity of the basic assumption that all heat is transferred purely by diffusive thermal conduction.

A.3 Uncertainty with steady-state and transient methods

A.3.1 General

In the present section, only methods included in the ISO 22007 series together with the most commonly used and standardized method working with steady state temperature fields will be considered.

The focus will be on how the different methods work and what information it is possible to obtain from the collected data. Special interest will be devoted to the presence of surface layers on solids which do not have the same thermal transport properties as the bulk material. Even a well prepared solid surface – being plane, cylindrical, spherical or of other shape - has a thin layer which forms the transition from the substrate's environment to the first surface displaying the same properties as that of the solid material, here referred to as the bulk properties. There is always some remaining roughness left even after the most ambitious preparation of the substrate (see Figure A.1). In addition to the surface roughness there are, in some cases, probes being employed, which are covered by an electrically insulating material. These films - although thin – and the surface roughness constitute a surface thermal resistance (STR) shall be considered when analysing the data from both transient and steady-state methods (see Figure A.2).

These STR layers can be made rather thin by good substrate preparation and the use of thin insulating layers on the probes. One can ascribe certain thermal properties to these layers and it should be noted that the thermal conductivity of these layers is normally quite low because of the presence of air at ambient or low pressure and therefore the thermal resistance is comparatively high.

A.3.2 Guarded hot plate method (ISO 8302)

When using a guarded hot plate or similar arrangement, the substrate is placed between two thermally high conducting plates and with arrangements for keeping the output of power constant. The procedure is to measure the temperature differences between the plates. With this experimental set-up, it is necessary to take the two STR layers on both sides of the substrate into consideration. There are essentially three ways to compensate for the influence of the STR layers:

a) to measure on a substrate thick enough so that the thermal resistance of the layers can be neglected;

b) to prepare two substrates of different thicknesses (and the same surface roughness) and then mathematically eliminate the influence of the contact resistance;

c) to introduce a fluid with as high thermal conductivity as possible to eliminate the voids, which are normally filled with air in the STR layers.

A.3.3 Hot wire method (ISO 8894-1 and ISO 8894-2)

For these methods, identical arguments apply as for the TPS (hot disc) method. However, it is important to note that the analysis of the transient recordings is performed with the presence of the STR layers.

A.3.4 Transient plane source (hot disc) method (ISO 22007-2)

When using this method an electrically insulated probe, in the shape of a double spiral, is placed between two sample pieces -–each of them with a plane surface facing the probe. Here it is obvious that there is a STR layer, which consists of the roughness of the surface of the substrate and the electrical

insulation of the probe. If a constant output of power is arranged from the probe (see ISO 22007-2) the mean temperature increase of the probe can be given as:

$$T(t) = \Delta T_{STR} + F(P, \Lambda, L) \cdot D(t, a, l) \quad \text{(A.1)}$$

where,

t is the time measured from the start of the experiment;

F is a function of the output of power, the thermal conductivity of the bulk material and a length dimension of the probe;

D is the so-called shape function dependent on;

a is the thermal diffusivity of the bulk material;

l is a length dimension of the probe.

ΔT_{STR} is also a function of time. However, it can be considered a constant throughout the transient recording if the relaxation time d_i^2 / a_i is less than the reciprocal sampling rate and the total number of samples of the transient is not less than 100. d_i is the thickness and a_i is the thermal diffusivity of the STR layer. The constancy of ΔT_{STR} under these conditions has been proven both experimentally and in computer simulations.

If the output of power is constant and selected at such a level that the thermal transport properties can be considered constant, a plot of the mean temperature increase versus the shape function would constitute a straight line. The slope of this line is identical with the F – function, from which normally the thermal conductivity can be retrieved.

Consequently, following the procedures indicated above, we are then actually able to obtain the bulk properties of the solid material, and this is achieved by removing the influence from the STR layers. This convenient possibility rests with some of the transient methods, but is difficult to be achieved with steady state methods.

There are four main configurations when using the TPS (hot disc) method.

These are:

— bulk,

— anisotropic,

— slab and thin film substrates.

It can be seen from Formulae (3), (4), (6), (8) and (11) of ISO 22007-2:2015 that the condition outlined by Formula (A.1) above is applicable and it is possible to avoid the influence of the STR layer. However, when using the thin film arrangement, the STR layer influences the thermal properties measured, which is obvious from Formula (13) of ISO 22007-2:2015. This means that the thin film method operates essentially like the hot plate steady-state methods, and similar precautions shall be considered if the influence of the STR layer is to be eliminated.

A.3.5 Temperature wave analysis method (ISO 22007-3)

The recommendation on substrate and probe preparation for this method is to use a plane slab substrate with heater and probe attached on opposite sides. It is also suggested that the heater and probe be deposited on the slab with some physical vapour deposition (PVD) method. Experimental evidence from numerous methods indicate, that the PVD produced heater/probe is filling out the surface voids and consequently covering the surface in such a way that the STR layers do not have any influence.