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Plastics — Determination of thermal conductivity and thermal diffusivity

Part 1: General principles (ISO 22007-1:2009)

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee PRI/21, Testing of plastics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Foreword

The text of ISO 22007-1:2009 has been prepared by Technical Committee ISO/TC 61 “Plastics” of the International Organization for Standardization (ISO) and has been taken over as EN ISO 22007-1:2012 by Technical Committee CEN/TC 249 “Plastics” the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2012, and conflicting national standards shall be withdrawn at the latest by July 2012.

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Endorsement notice

The text of ISO 22007-1:2009 has been approved by CEN as a EN ISO 22007-1:2012 without any modification.

Contents

Page

Foreword ... **iv**

1 Scope ... **1**

2 Normative references ... **1**

3 Terms and definitions ... **1**

4 Principles ... **2**

5 Test methods ... **3**
5.1 General ... **3**
5.2 Hot-wire method ... **5**
5.3 Line-source method ... **6**
5.4 Transient plane source method ... **6**
5.5 Temperature wave analysis method ... **7**
5.6 Laser flash method ... **8**
5.7 Guarded methods ... **8**
5.7.1 Guarded hot-plate method ... **8**
5.7.2 Guarded heat flow meter method ... **9**

6 Test report ... **10**

Annex A (informative) **Sources of uncertainty in transient methods** ... **11**

Bibliography ... **17**

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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ISO 22007-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

ISO 22007 consists of the following parts, under the general title *Plastics — Determination of thermal conductivity and thermal diffusivity*:

— *Part 1: General principles*

— *Part 2: Transient plane heat source (hot disc) method*

— *Part 3: Temperature wave analysis method*

— *Part 4: Laser flash method*

— *Part 5: Determination of thermal conductivity and thermal diffusivity of poly(methyl methacrylate)* [Technical Report] (in preparation)

INTERNATIONAL STANDARD **ISO 22007-1:2009(E)**

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 part of ISO 22007 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 part of ISO 22007 provides a broad overview of these techniques. Standards specific to these techniques, as referenced in this part of ISO 22007, are used to carry out the actual test method.

2 Normative references

The following referenced documents are indispensable for the application 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.

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 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 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 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 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 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:

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

where

λ is the thermal conductivity;

ρ is the density;

c_p is the heat capacity

NOTE 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 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. The response is then 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 contact transient method 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 about five times 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 Pyrex® 7740[1)] glass 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.

Table 1 — Basic characteristics of contact transient methods

Type of method	Heat source geometry	Mode of heat generation	Heat source/temperature sensor configuration	Measured and/or derived parameters
Hot wire/line source/hot strip	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
Plane source transient	Disc	Pulse	Combined	α, C_p, λ
λ = thermal conductivity; α = thermal diffusivity; b = thermal effusivity; C_p = specific heat				
[a] One sensor. [b] Two sensors.				

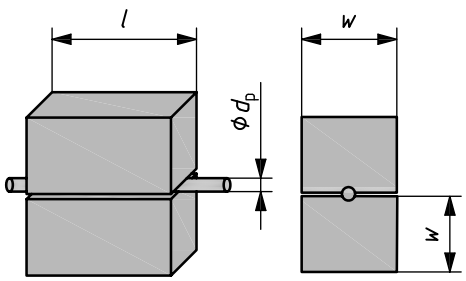
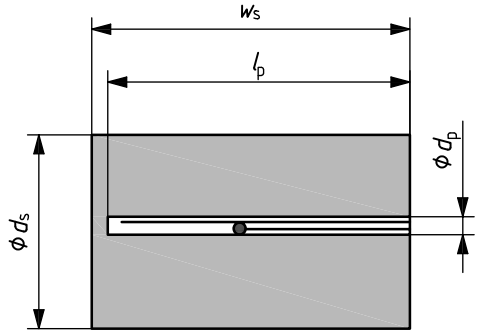
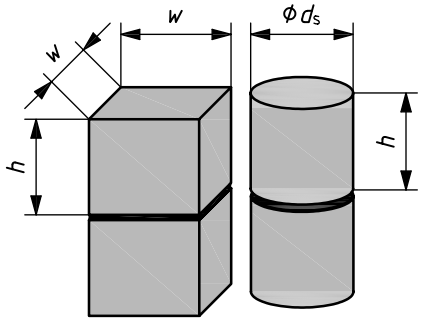
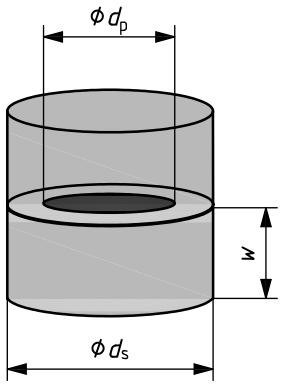
5 Test methods

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 these methods are summarized in Table 2 and then further explained in more detail. Complete details of the test methods described in 5.4 to 5.6 can be found in ISO 22007-2 [14], ISO 22007-3 [15] and ISO 22007-4 [16].

1) Pyrex is a registered trademark of Corning Incorporated. This information is given for the convenience of users of this part of ISO 22007 and does not constitute an endorsement by ISO of this product.

Table 2 — Schematic diagrams of various contact 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	$200 d_p < w$ $l > 4w$
Line source [a]		w_s = active zone l_p = probe length d_p = probe diameter d_s = specimen diameter	$w_s > 1{,}5 l_p$ $l_p > 33 d_p$ $d_s > 6 d_p$
Hot plate [b]		w = width, thickness h = height d_s = specimen diameter	$w, h, d_s > 3\sqrt{\alpha t_{max}}$ where t_{max} = maximum measurement time
Plane source transient [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} = 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.

In contact methods, enough uniaxial pressure should be applied to press the various parts of the specimen and the heat source together in order to obtain good thermal contact. Heat sink paste can be used to improve contact, but there should be no heat sink paste outside the heater, or the temperature field can be disturbed. Furthermore, the use of heat sink pastes can contribute to the uncertainty of the measurement and their effect must be adequately quantified for accurate results.

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 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 in the thermocouple is measured 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:

$$\Delta T\left(r,t\right) = -\frac{\Phi}{4\pi L\lambda}\,\mathrm{Ei}\left(-\frac{r^2}{4\alpha t}\right) \tag{1}$$

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 ($\alpha = \lambda/\rho C_p$);

ρ is the density, in kg/m^3;

C_p is the isobaric specific heat, in J/(kg·K);

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

$$-\mathrm{Ei}(x) = \int_x^{\infty} \frac{\mathrm{e}^{-u}}{u}\,\mathrm{d}u \tag{2}$$

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

$$\Delta T(r,t) = -\frac{\Phi}{4\pi L\lambda}\ln\frac{4\alpha t}{r^2 C} \tag{3}$$

where

$C = \mathrm{e}^{\gamma}$ where γ is Euler's constant (= 0,577 216).

According to Equation (3), 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 the equation:

$$\lambda = \frac{\Phi}{4\pi LK} \tag{4}$$

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, Equation (4) can be used for practical applications.

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

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 equations 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 Equation (4); it is the ratio of the actual thermal conductivity of the reference material to that measured by the instrument:

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

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 D 5930 [17].

5.4 Transient plane source method

The transient plane source method is capable of solid-state measurements on sheets of materials. It can be applied to cases where orientation effects exist and can also be extended to thin films.

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 must 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

$\Delta p_{prob} = 2(\alpha t)^{1/2}$, 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 must 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 [14].

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.

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:

$$T(d,t) = \frac{\sqrt{2} j_0 \lambda k \exp(-kd)}{\left(\lambda k + \lambda_b k_b\right)^2} \exp\left[\mathrm{i}\left(\omega t - kd - \frac{\pi}{4}\right)\right] \tag{6}$$

where

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

t is 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;

2) Kapton is a registered trademark of DuPont. This information is given for the convenience of users of this part of ISO 22007 and does not constitute an endorsement by ISO of this product.

λ is the thermal conductivity;

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

subscript "b" refers to the backing plates.

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

$$\Delta\theta = -\sqrt{\frac{\omega}{2\alpha}}d - \frac{\pi}{4} \tag{7}$$

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

$$\alpha = \frac{d^2}{2A^2} \tag{8}$$

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 [15].

5.6 Laser flash method

The laser flash technique is a non-contact method used for measuring the thermal diffusivity of homogeneous, isotropic and opaque materials. Transparent materials can be measured provided that they are first coated 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. 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 pulse [10]. The temperature rise at the opposite specimen face is measured by an IR detector. 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 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. The partial time moments method [5] is recommended in ISO 22007-4, although other models, such as that based upon the so-called "half-time" method proposed by Parker, can be used.

Details of the test method can be found in ISO 22007-4 [16].

5.7 Guarded methods

5.7.1 Guarded hot-plate method

The guarded hot-plate method described in ISO 8302 [11] 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,

$$Q = \lambda A \Delta T / d \tag{9}$$

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 12 h, although this varies with test temperature and specimen thickness. Measurements of thermal conductivity in the range 0,014 W/(m·K) to 2,0 W/(m·K) are typical.

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

5.7.2 Guarded 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 must 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 Pyrex® 7740 glass, available from the Institute for Reference Materials and Measurements (IRMM), http://www.irmm.jrc.be/.[3)]

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:

$$Q/A = \lambda(T_h - T_c)/d \quad (10)$$

3) This information is given for the convenience of users of this part of ISO 22007 and does not constitute an endorsement by ISO of the product or supplier named.

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 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 must 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 E 1530 [18].

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;

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 in transient methods

A.1 General

A.1.1 Current experience indicates that low measurement uncertainties are not readily attained and, for some techniques involving multi-property measurements, the results are often internally inconsistent.

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 ideal model during the measurement period.

A.1.3 The essential criterion for accurate measurements is an undeformed temperature field. Two differences exist between the ideal model and a real situation, namely the finite size of the specimen and the fact that the construction of the heat source is 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. The ideal model gives the optimum measurement time and the maximum time window for evaluation of the temperature response. However, significant deviations may occur between the ideal model and a real experimental set-up which may result in correspondingly significant measurement uncertainties.

A.1.4 The basic geometries of the experimental set-ups used in the various methods are given in Table 2. Three thermophysical parameters can be determined when a two-probe system (a plane heat source and a line temperature sensor) is used as shown in Figure A.1. Three parameters can also be determined when a disc heat source, i.e. a single-probe system, is used.

A.1.5 Heat source and surface effects can also deform the temperature field generated by the heat source, causing a deviation from the ideal model. For example, Figure A.2 shows the deformation of the temperature isotherm when the specimen has a cylindrical shape, a plane heat source is used and heat loss occurs into the surroundings from the specimen surface. Figure A.3 shows the deformation of the temperature field due to thermal-constriction resistance between two bodies in contact, while Figure A.4 shows the induced thermal-constriction resistance due to the construction of a non-idealized heat source with which the heating is not uniform across the heater area. Figure A.5 shows the region in the specimen in which the temperature field is deformed due to both thermal-constriction resistance and induced thermal-constriction resistance. The experimental set-up has to be designed in such a way that the size of the region in which the temperature field is deformed is negligible compared to that in which it is undeformed. Usually, the region in which the temperature field is deformed by induced thermal-constriction resistance is significantly larger than that in which the temperature field is deformed by thermal-constriction resistance.

A.1.6 The difference between the ideal time window identified by the model and the real one in an actual test is illustrated by an example shown in Figure A.6. This uses difference analysis to obtain the time window representing a period of data stability. The measurements were performed on a 4,9-mm-thick specimen of poly(methyl methacrylate) having known properties [$\lambda = 0{,}192$ W/(m·K), $\alpha = 0{,}11 \times 10^{-6}$ m^2/s, $C = 1\,460$ J/(kg·K) and $\rho = 1\,184$ kg/m^3]. The time window for the ideal model is limited by the sensitivity coefficient and the corresponding experimental time window is limited, in addition, by heat source effects at the beginning and by surface effects at the end of the time window.

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 ideal model, as the temperature functions used for the calculation of the thermophysical properties are those for the ideal model. Of importance 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 between heat source and specimen, between specimen and sensor and within 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,

      — determination of heat pulse size by analysis of temperature response.

c) Material/specimen parameters:

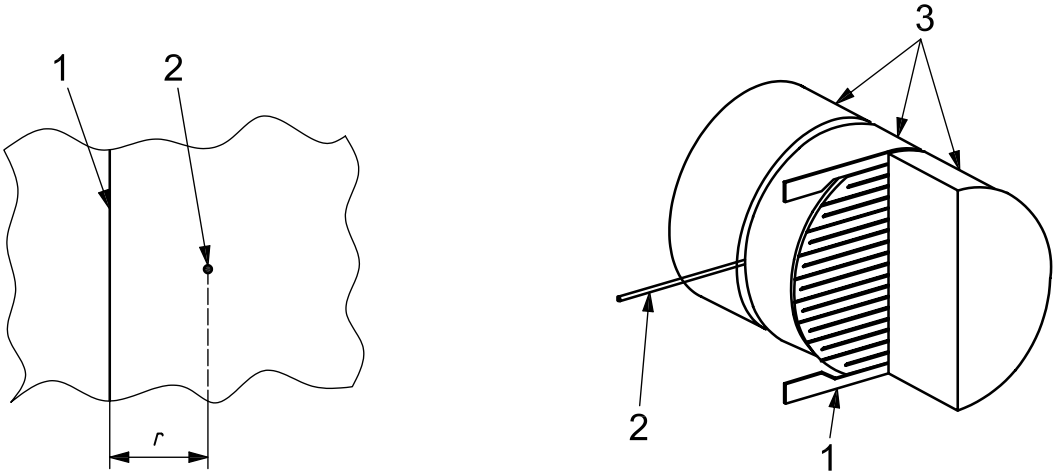
   1) specimen size and configuration;

   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 conduction.

A.3 Use of automated instruments

These methods are often used in the form of fully automated systems in which the results are provided using a specific analysis of the model by specialized or proprietary software. Users often lack first-hand knowledge of the materials and/or method involved and, as a result, assume, not necessarily correctly, that the method will produce a solution which corresponds exactly to the model chosen.



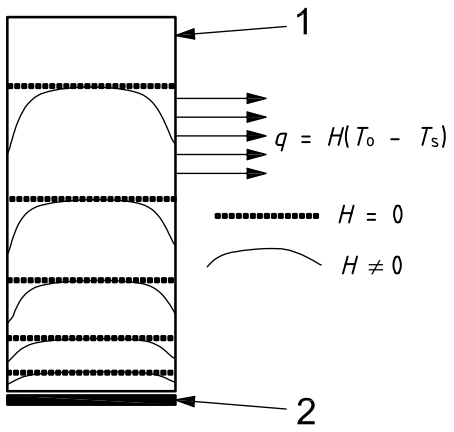
a) Ideal model **b) Experimental set-up**

Key

1 heat source
2 sensor
3 specimen
r distance between heat source and sensor

NOTE Part of the specimen has been cut away to show the construction of the heat source.

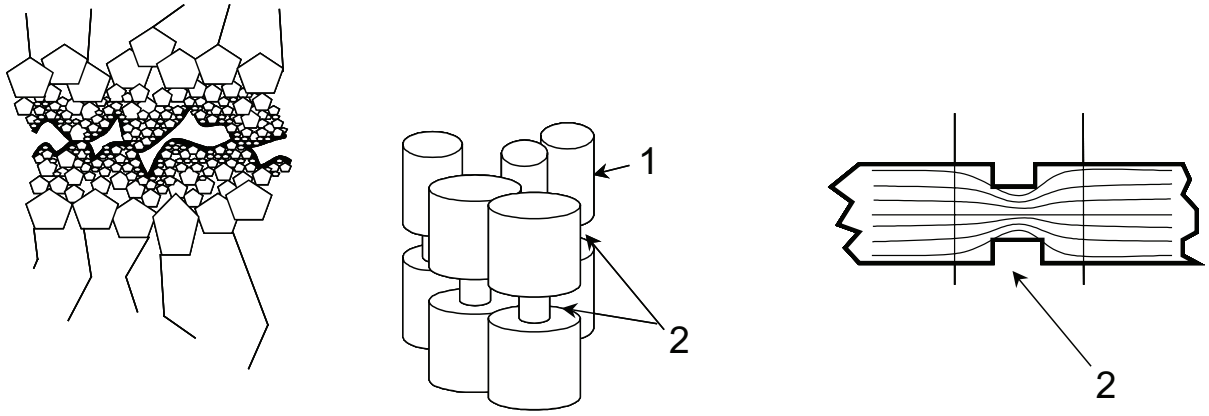
Figure A.1 — Difference between the ideal model and a real one for pulse transient and step-wise transient methods



Key

1 specimen
2 heat source
q heat lost to surroundings
H heat transfer coefficient
T_s specimen surface temperature
T_0 temperature of surroundings

Figure A.2 — Deformation of the temperature field for a plane heat source and cylindrical specimen



a) Real contact [a] **b) Idealized-contact model** [b] **c) Contact region** [c]

Key

1 flux channel

2 contact region

[a] Real contact between two bodies.

[b] Ideal model showing a set of flux channels connected by contact spots. The cross-sectional areas of the channels are the same as the cross-sectional areas of the relevant parts of the two contacting bodies and the cross-sectional areas of the contact spots are the same as the areas of surface in actual contact with each other.

[c] The contact region is the region in the channel where the flux lines are deformed (i.e. the deformed temperature field).

Figure A.3 — Thermal-constriction resistance

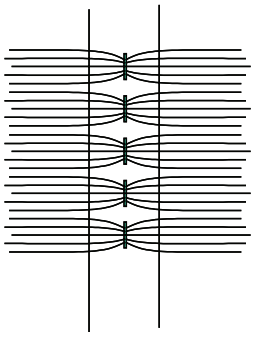
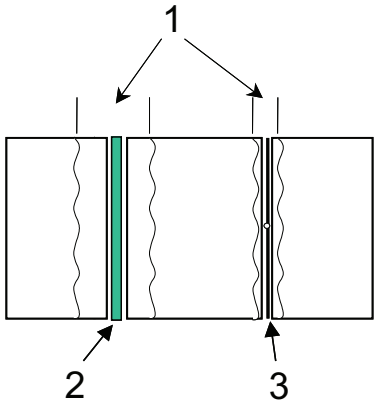


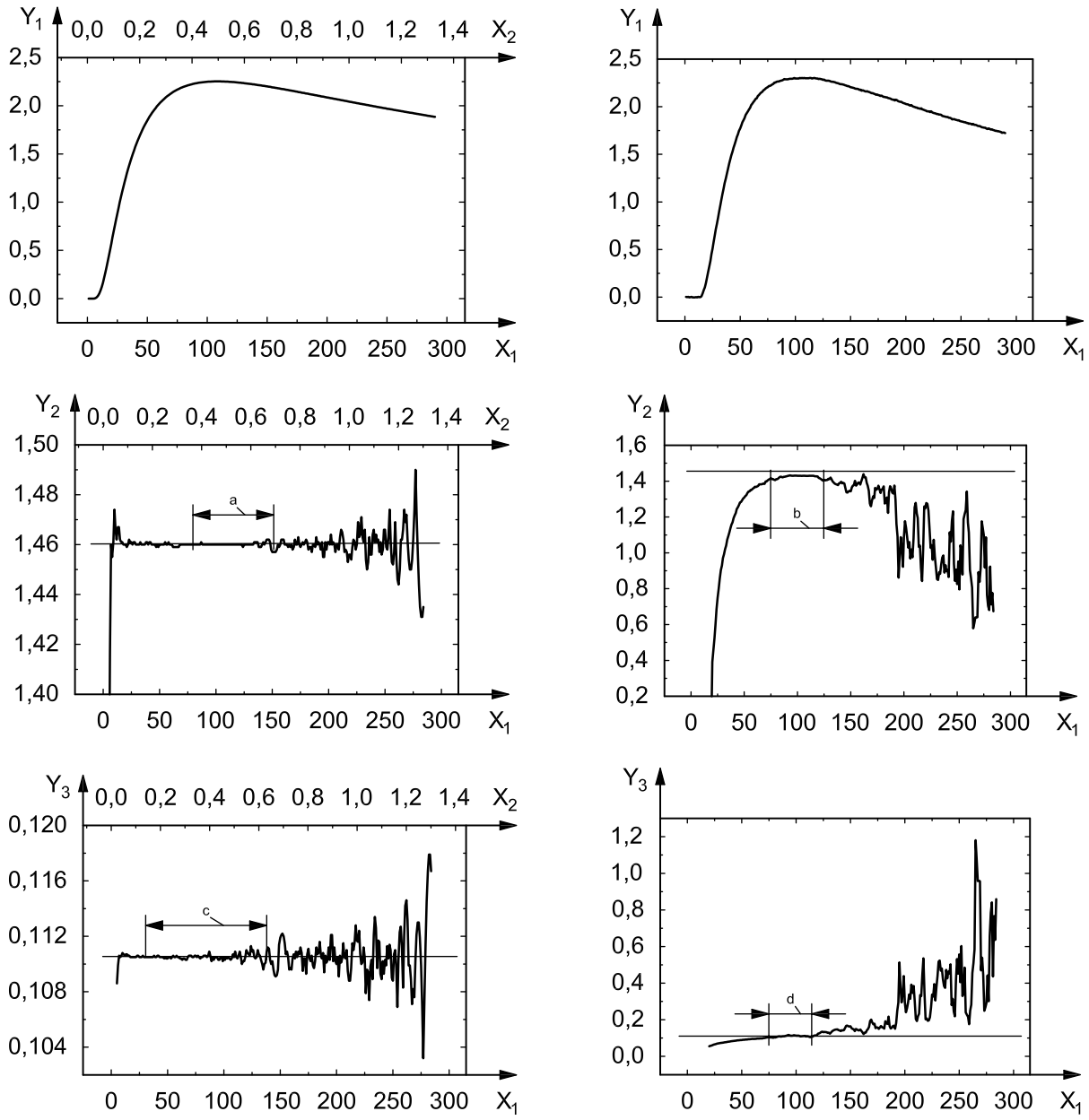
Figure A.4 — Induced thermal-constriction resistance caused by the construction of the heat source shown in Figure A.1



Key

1 non-homogeneous (deformed) temperature field
2 heat source
3 sensor

Figure A.5 — Deformation of the temperature field in the heat source and sensor regions



a) Ideal model **b) Experiment**

Key

X_1 time (s)
X_2 $F = \alpha t / h^2$
Y_1 temperature (°C)
Y_2 $c_p \times 10^3$ [J/(kg·K)]
Y_3 $\alpha \times 10^{-6}$ (m^2/s)

a 80 s to 150 s.
b 75 s to 125 s.
c 30 s to 135 s.
d 75 s to 114 s.

Figure A.6 — Results of difference analysis for the pulse transient method showing the difference between the ideal model and experimental values

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