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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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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)

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·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.

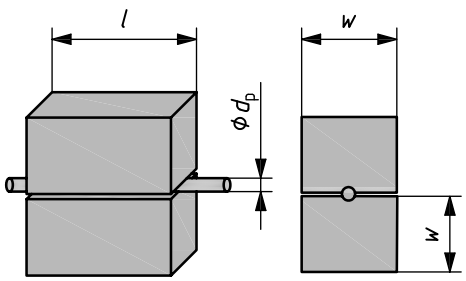
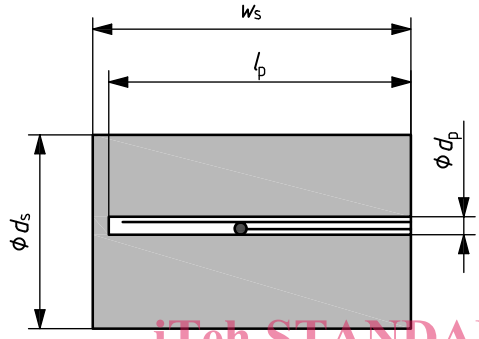
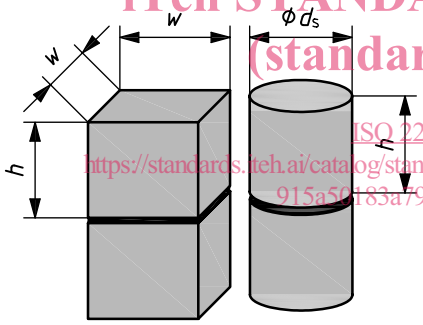
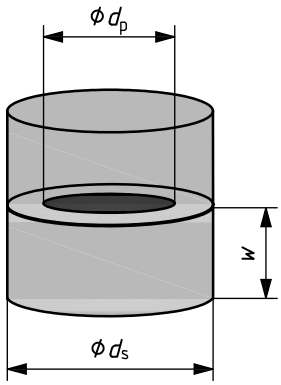
5 Test methods

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.