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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

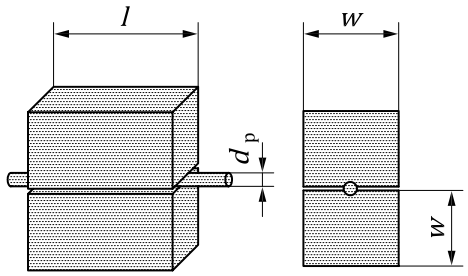
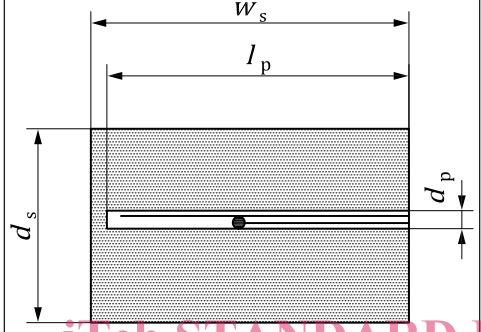
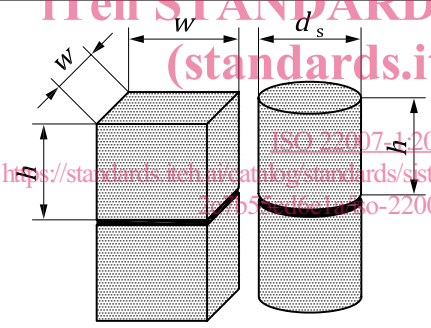
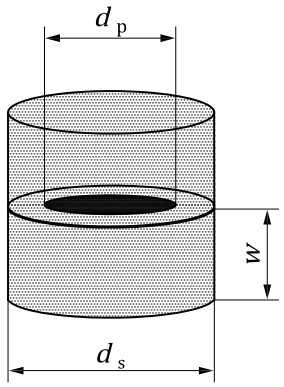
5.1 General

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

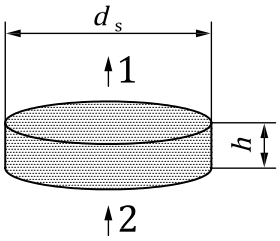
Method	Specimen set-up	Characteristic parameters	Ideal model
Hot wire[a]		l = specimen length w = specimen width, thickness d_p = wire probe diameter	$200 d_p < w$ $l > 4w$
Line source[a]		w_s = active zone l_p = probe length d_p = probe diameter d_s = specimen diameter	$w_s > 1{,}5 l_p$ $l_p > 33 d_p$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