INTERNATIONAL STANDARD

ISO 22007-4

Third edition

Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 4: Light flash method

Plastiques — Détermination de la conductivité thermique et de la diffusivité thermique —
Partie 4: Méthode par flash lumineux

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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 document 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,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 22007-4:2017), which has been technically revised.

The main changes are as follows:

— the term laser flash has been replaced by the more general term light flash.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 4: Light flash method

1 Scope

This document specifies a method for the determination of the thermal diffusivity of a thin solid disc of plastics in the thickness direction by the light flash method. This method is based upon the measurement of the temperature rise at the rear face of the thin-disc specimen produced by a short energy pulse on the front face.

The method is applicable to homogeneous solid plastics as well as composites having an isotropic or orthotropic structure. In general, it covers materials having a thermal diffusivity, α, in the range 1×10^{-7} $m^2 \cdot s^{-1} < \alpha < 1 \times 10^{-4}$ $m^2 \cdot s^{-1}$. Measurements can be carried out in gaseous and vacuum environments over a temperature range from −100 °C to +400 °C.

NOTE For inhomogeneous specimens, the measured values can be specimen thickness dependent.

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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/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

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ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 527-1, *Plastics — Determination of tensile properties — Part 1: General principles*

ISO 22007-1, *Plastics — Determination of thermal conductivity and thermal diffusivity — Part 1: General principles*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 22007-1 and the following apply.

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

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

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

3.1
pulse width
t_p
duration for which the light pulse intensity is larger than half of its maximum value

Note 1 to entry: It is expressed in seconds (s).

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3.2
time origin
t_0
start of the light pulse

Note 1 to entry: It is expressed in seconds (s).

3.3
maximum temperature rise
ΔT_{max}
difference between the maximum temperature reached by the rear face of the specimen after the light pulse has passed and its steady temperature before the pulse

Note 1 to entry: It is expressed in kelvins (K).

3.4
half-rise time
$t_{1/2}$
time from the *time origin* (3.2) until the rear-face temperature increases by one-half of ΔT_{max}

Note 1 to entry: It is expressed in seconds (s).

3.5
thermogram
temperature versus time curve for the rear face of the specimen

3.6
thickness
d
dimension of the test specimen in the direction of heat transfer measurement

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

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4 Principle

One side of a flat-sheet test specimen is subjected to an energy pulse which has a very short duration compared with the half-rise time (see 6.1) and a uniform spatial energy distribution. The transient temperature rise on the opposite face (rear face) is recorded as a function of time (see Figure 1). The thermal diffusivity is obtained by comparing the experimental thermogram with a theoretical model (see Clause 9 and Annex B).

5 Apparatus

5.1 General

The apparatus shall be designed to obtain the thermal diffusivity as described in Clause 4 and shall consist of the following main components as shown in Figure 2. These are the furnace or climatic chamber with a specimen holder and temperature measurement device (e.g. thermocouple), the flash source (e.g. laser), the pulse detector, the transient detector (IR detector) and the control, data acquisition and analysis unit.

5.2 Furnace or climatic chamber

The furnace or climatic chamber shall meet the following requirements.

a) The temperature range shall be appropriate to the range of materials to be studied. Depending on the range of temperature, the specimen is maintained at a constant temperature by a cryostat or by a furnace.

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b) It shall be capable of maintaining the test temperature constant to within ±0,5 K or less for at least 30 min.

c) The temperature measurement device shall be capable of measuring the furnace or climatic chamber temperature with a resolution of ±0,1 K and an accuracy of ±0,5 K or better.

d) The furnace or climatic chamber shall be fitted with two windows, one transparent to the pulse radiation and the other transparent to the working wavelength range of the IR detector.

e) If required, the test environment shall be vacuum or inert-gas atmosphere to avoid oxidative degradation during heating and testing of the specimen. For cryoscopic measurements, care shall be taken to avoid water condensation on the windows.

NOTE Measurement under vacuum will eliminate convection effects.

The specimen holder shall be designed to minimize thermal contact with the specimen and to suppress stray light transmitted from the light beam to the IR detector.

The test temperature shall be measured using a calibrated temperature measurement device that is preferably in contact with the specimen or the specimen holder but at least within 1 mm of the specimen holder.

The temperature measurement device shall be designed so as not to significantly disturb the temperature field generated in the specimen by the light pulse.

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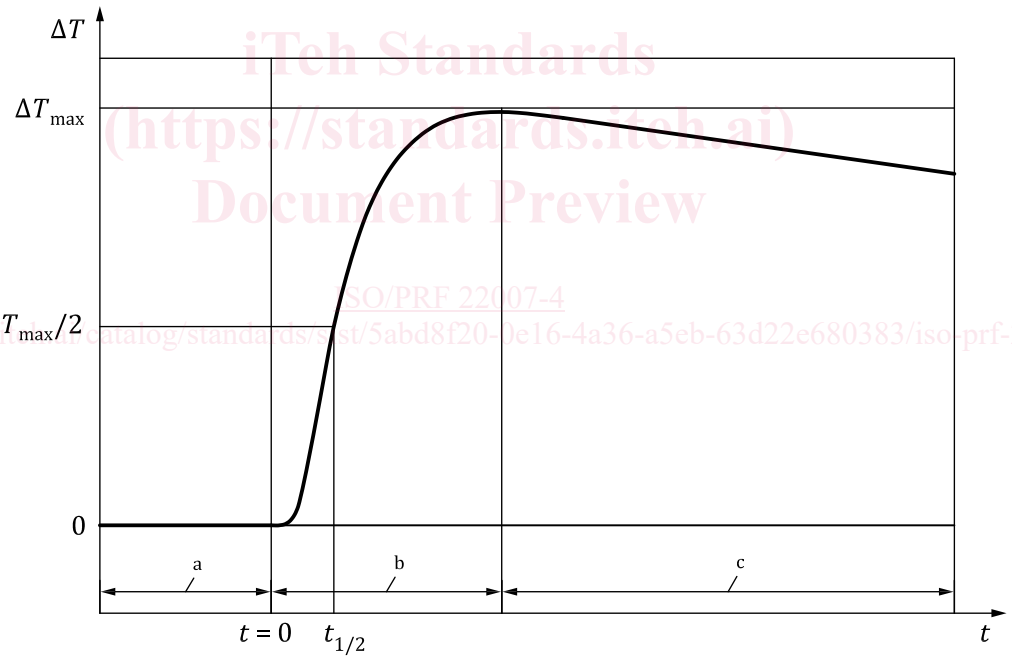


Key

t time

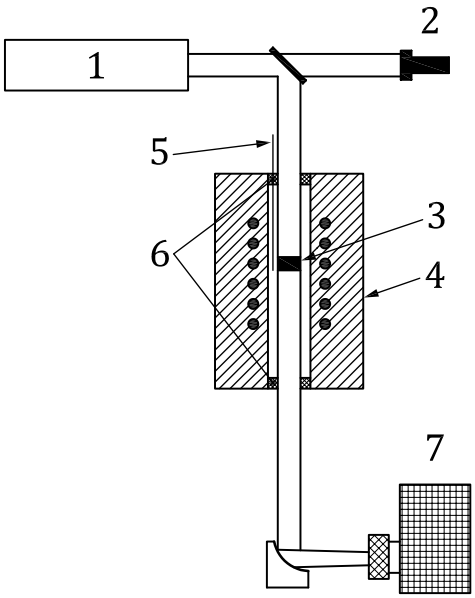
ΔT temperature rise

a Baseline.

b Transient-rise period.

c Cooling period.

Figure 1 — Example of thermogram



Key

1 flash source
2 pulse detector
3 specimen
4 furnace or climatic chamber
5 temperature measurement device
6 windows
7 transient detector

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Figure 2 — Schematic diagram of light flash set-up for measuring thermal diffusivity

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5.3 Flash source

The energy level of the flash source shall produce a temperature rise not exceeding 3 K at the rear face of the specimen.

The spatial energy distribution of the pulse heating shall be as uniform as possible over the front face of the specimen.

The pulse duration shall be shorter than 1 ms.

The heat pulse source may be a laser or a flash tube.

A photodiode can be used to determine the duration and form of the pulse and the time origin.

5.4 Transient detectors

The transient temperature rise at the rear face of the specimen shall be measured with an IR detector. The transient detector shall be able to detect a variation of 5 mK in the specimen rear face temperature. Its response shall be linear with temperature over a temperature range of at least 3 K.

The frequency response of the detector and its associated electronics (amplifiers, analogue/digital converters, etc.) shall be faster than 10 kHz. If electronic filters are used, they shall meet the requirements defined above and shall not decrease the accuracy of temperature measurement, otherwise they could distort the shape of the temperature-time curve.

NOTE The choice of IR detector depends also on the temperature range. For the range –100 °C to +400 °C, photovoltaic or photoconductor detectors can be used.

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The temperature of the rear face, or a quantity directly proportional to it (e.g. voltage), shall be measured and recorded continuously over the duration of the test. The data acquisition system, which may be analogue or digital, shall be able to sample more than 1 000 data points on the thermogram with a sampling frequency higher than $100/t_{1/2}$. The accuracy of the time base shall be better than $\pm 1 \times 10^{-5}$ s.

5.5 Thickness measurement device

The specimen thickness shall be measured with an accuracy of ±5 µm by a calibrated thickness measurement device having a resolution of ±1 µm. For soft materials, a micrometer with reproducibly low compression is required.

6 Test specimen

6.1 Shape and dimension of the specimen

The specimen shall be a thin disc. The specimen diameter is usually from 5 mm to 20 mm. The specimen thickness shall be chosen according to the pulse width and the thermal diffusivity of the material. It shall be selected such that the pulse width is less than 0,01 of the half-rise time. Typically, the thickness will be between 0,5 mm and 3 mm. The aspect ratio of the specimen shall be chosen such that 2D effects are negligible during the test. The ratio of the diameter to the thickness shall be larger than 3:1.

The faces shall be flat and parallel. Any variation in the thickness of the specimen should preferably be less than 1 % of the mean thickness. The effect of greater non-uniformity can be estimated in the measurement uncertainty.

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6.2 Preparation and conditioning of test specimen

The test specimen shall be representative of the material being examined and shall be prepared and handled with care. If the specimen is taken from sample pieces by cutting, care shall be taken to prevent heating, changes in molecular orientation or any other effect that can alter the sample properties.

The preparation of specimens of oriented, anisotropic samples shall be done as specified in Annex C.

The test specimen shall be conditioned prior to the measurement as specified in the relevant material standard or by a method agreed between the parties involved. Unless other conditions are specified, the specimen shall be conditioned in accordance with ISO 291.

NOTE Depending on the material and its thermal history, the method of test specimen preparation can be crucial to the consistency of the results and their significance.

6.3 Coating the specimen

Specimens which are not opaque to the light radiation at the wavelength used shall be coated with an appropriate coating (a metal or graphite coating, for example) to prevent penetration of the light beam into the specimen. The influence of the coating on the heat transfer shall be negligible (i.e. it shall have a high diffusivity and low thickness in comparison with the specimen). The total thickness of the coating shall be chosen such that the half-rise time for the coating alone is less than 2 % of the total half-rise time for the specimen.

NOTE 1 The half-rise time, $t_{1/2}$, for the coating can be simply calculated from its thickness, d, and thermal diffusivity, α, using Formula (1), a rearranged form of Formula (B.1):

$$t_{1/2} = 0{,}138\ 79 \frac{d^2}{\alpha} \tag{1}$$

NOTE 2 Both sides of the specimen can be coated with a thin opaque layer as mentioned above to optimize the absorption of the energy pulse and the emission of thermal radiation.

7 Calibration and verification

7.1 Calibration of apparatus

The light flash technique for the determination of the thermal diffusivity is an absolute method which allows the user to perform measurements that are directly traceable to primary SI units (such as temperature, time, length and voltage). All elements of the light flash apparatus shall be calibrated separately, as follows.

— Calibrate the micrometer used to measure the specimen thickness.

— Calibrate the temperature measurement device used to measure the steady-state temperature of the specimen.

— Calibrate the time base and the voltage of the data acquisition system used to measure the signal coming from the IR detector.

— Calibrate the IR detector in order to be able to observe the transient temperature change at the rear face of the specimen rather than the transient spectral irradiance. Perform this calibration mainly for temperatures lower than 200 °C where nonlinear behaviour of the output temperature cannot be neglected (see ISO 18755[1]). If this calibration is not possible (e.g. because the IR detector is inaccessible or due to differences between the optical paths of the IR detector calibration configuration and the thermal-diffusivity measurement configuration), the effects of nonlinearity have to be taken into account in the uncertainty of measurement.

7.2 Verification of apparatus

The apparatus should preferably be verified periodically by measuring the thermal diffusivity of one or more reference materials covering the range of thermal diffusivities of the materials to be tested. If the measured values differ by more than 5 % from the reference values, recalibrate the various items of apparatus in accordance with 7.1.

Verification can be carried out by measurements on materials which have well-defined and reproducible thermal properties such as Armco[1)] iron, Poco[1)] graphite, Pyroceram 9606[1)] or poly(methylmethacrylate) (PMMA). Pyroceram 9606 (a ceramic material) has been certified for thermal diffusivity measurements as part of an international programme involving national metrology institutes[2].

It is recommended that the reference materials be chosen so that their properties (half-rise time and thermal diffusivity) are close to those of the materials to be tested.

8 Procedure

8.1 Switch on the equipment at least 1 h prior to any testing to allow it to reach equilibrium.

8.2 Measure the thickness of the specimen at ambient temperature, using a calibrated thickness measurement device. If a specimen coating is used, the specimen-thickness measurement shall be made before coating.

8.3 Mount the specimen in its holder and put this assembly in the furnace or climatic chamber.

8.4 If required, establish a vacuum or an inert-gas environment in the furnace or climatic chamber.

1) Armco, Poco and Pyroceram 9606 are examples of products available commercially. This information is given for the convenience of users of this document, and does not constitute an endorsement by ISO of the products named.

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