
**Plastics — Determination of thermal
conductivity and thermal diffusivity —**

**Part 4:
Laser flash method**

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

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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.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html. (standards.itech.ai)

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This second edition cancels and replaces the first edition (ISO 22007-4:2008), *Annex C* of 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 4: Laser 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 laser 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 can be used for 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 $1 \times 10^{-7} \text{ m}^2 \cdot \text{s}^{-1} < \alpha < 1 \times 10^{-4} \text{ m}^2 \cdot \text{s}^{-1}$. Measurements can be carried out in gaseous and vacuum environments over a temperature range from $-100 \text{ }^\circ\text{C}$ to $+400 \text{ }^\circ\text{C}$.

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

2 Normative references (standards.iteh.ai)

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)*

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

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

ISO 2818, *Plastics — Preparation of test specimens by machining*

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 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 pulse width

t_p
duration for which the laser pulse intensity is larger than half of its maximum value

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

**3.2
time origin**

t_0
start of the laser 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 laser 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 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.

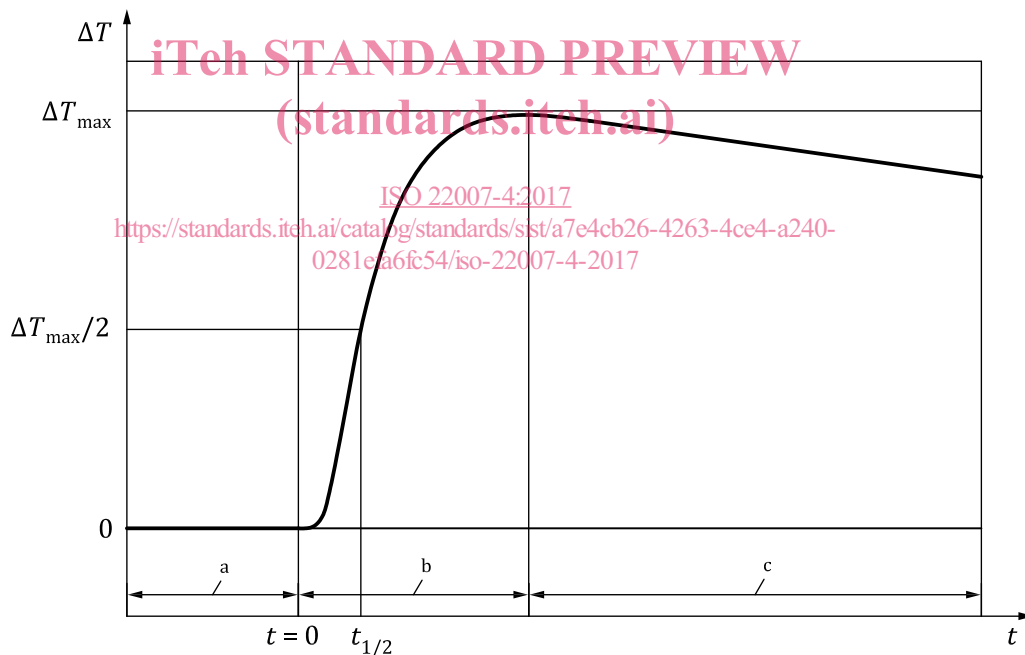
- b) It shall be capable of maintaining the test temperature constant to within $\pm 0,5$ K or less for at least 30 min.
- c) The temperature measurement device shall be capable of measuring the furnace or climatic chamber temperature with a resolution of $\pm 0,1$ K and an accuracy of $\pm 0,5$ K or 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 laser 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.

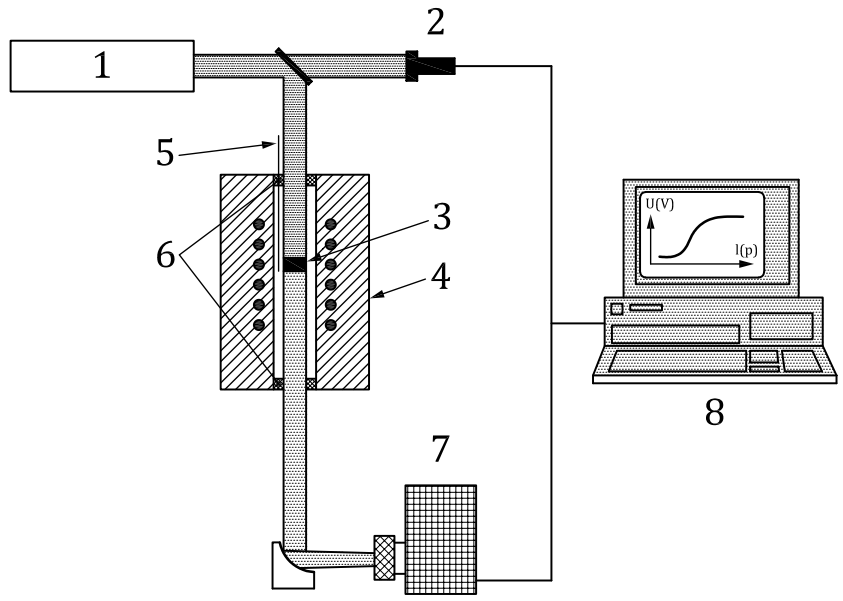
The temperature measurement device shall be designed so as not to significantly disturb the temperature field generated in the specimen by the laser pulse.



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
 - 8 control, acquisition and analysis unit

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

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 (preferably) 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 $-100\text{ }^{\circ}\text{C}$ to $+400\text{ }^{\circ}\text{C}$, photovoltaic or photoconductor detectors can be used.

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 $\pm 5\text{ }\mu\text{m}$ by a calibrated thickness measurement device having a resolution of $\pm 1\text{ }\mu\text{m}$. 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. standards.iteh.ai/catalog/standards/sist/a7e4cb26-4263-4ce4-a240-0281efa6fc54/iso-22007-4-2017

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 may alter the sample properties.

A method for sample preparation of oriented, anisotropic specimens is 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, it is recommended that the specimen be conditioned in accordance with ISO 291.

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

6.3 Coating the specimen

Specimens which are not opaque to the laser radiation at the wavelength used shall be coated with an appropriate coating (a metal coating, for example) to prevent penetration of the laser 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 \(B.1\)](#):

$$t_{1/2} = 0,13879 \frac{d^2}{\alpha} \quad (1)$$

NOTE 2 Both sides of the specimen can be coated with a thin opaque black layer (i.e. a layer of graphite) to optimize the absorption of the energy pulse and the emission of thermal radiation.

7 Calibration and verification

7.1 Calibration of apparatus

The laser flash technique 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) without calibration using reference materials. All elements of the laser 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 Armco¹⁾ iron, Poco¹⁾ graphite, Pyroceram 9606¹⁾ 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]. This certified reference material is available from IRMM (Institute for Reference Materials and Measurements)^[3].

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.

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.

- 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.
- 8.5** Set the furnace or climatic chamber to the desired temperature and wait until the stability of the specimen temperature is better than 0,05 K/s.
- 8.6** Measure the steady-state temperature of the specimen.
- 8.7** Calculate the specimen thickness at the test temperature, taking into account the change in thickness due to thermal expansion.
- 8.8** Irradiate the specimen with the laser pulse, taking adequate safety precautions. Care shall be taken to ensure that the laser beam does not cause any local degradation of the specimen. The specimen shall be inspected after removal from the instrument to check for any signs of degradation.
- 8.9** Measure and record continuously the temperature of the rear face of the specimen or a quantity directly proportional to it before, during and after the laser pulse. The thermogram obtained shall include the baseline, the transient-rise period and the cooling period (see [Figure 1](#)).
- 8.10** If necessary, measure the time form of the laser pulse applied in [8.8](#) in order to correct for the effect of the finite duration of the pulse (for guidance see [Annex A](#)).
- 8.11** From the thermogram obtained, calculate the thermal diffusivity in accordance with [Clause 9](#).
- 8.12** Repeat at least three times the procedures described from [8.8](#) to [8.11](#) at the same temperature and calculate the average value of the thermal diffusivity and the repeatability of the measurements.

The analysis assumes that the specimen is homogeneous. If there are any doubts about the homogeneity, it is recommended that measurements be performed on two specimens with a thickness ratio of about 1:2. If the same diffusivity values are obtained, the effect of inhomogeneity can be assumed to be negligible.

- 8.13** Change the test temperature to the next desired temperature and repeat steps [8.5](#) to [8.12](#).

If testing is being carried out at several temperatures, it is recommended that an increasing rather than decreasing temperature profile be used as this will minimize or avoid the effect of distortion of the specimen on the results, as distortion is more likely to occur at higher temperatures.

If testing at elevated temperatures, it is recommended that the effect of possible changes in the specimen at such temperatures be investigated. This can be done by carrying out a test at ambient temperature both before and after the required series of tests. If there is a difference in the results obtained, consideration shall be given to reducing the time at elevated temperature for each specimen.

Care shall be taken to avoid testing at temperatures at which the specimen may undergo a transformation (e.g. first- or second-order transformation, crystal transformation or glass transition) during the test.

NOTE The test temperature is a nominal temperature that can be associated with the measured thermal-diffusivity value as this value is determined under higher transient temperatures due to the heating effect of the laser pulse. An extrapolation procedure^[4] can be used to calculate the “intrinsic thermal diffusivity” at the test temperature corresponding to the thermal diffusivity that would be measured for a laser pulse of zero energy.