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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 flash lumineux

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Foreword

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— the term laser flash has been replaced by the more general term light flash.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

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 $1 \times 10^{-7}\ \text{m}^2\cdot\text{s}^{-1} < \alpha < 1 \times 10^{-4}\ \text{m}^2\cdot\text{s}^{-1}$. Measurements can be carried out in gaseous and vacuum environments over a temperature range from −100 °C to +400 °C.

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

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)*

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).

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

$\Delta T_{\max}$

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 $\Delta T_{\max}$

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).

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 ±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.

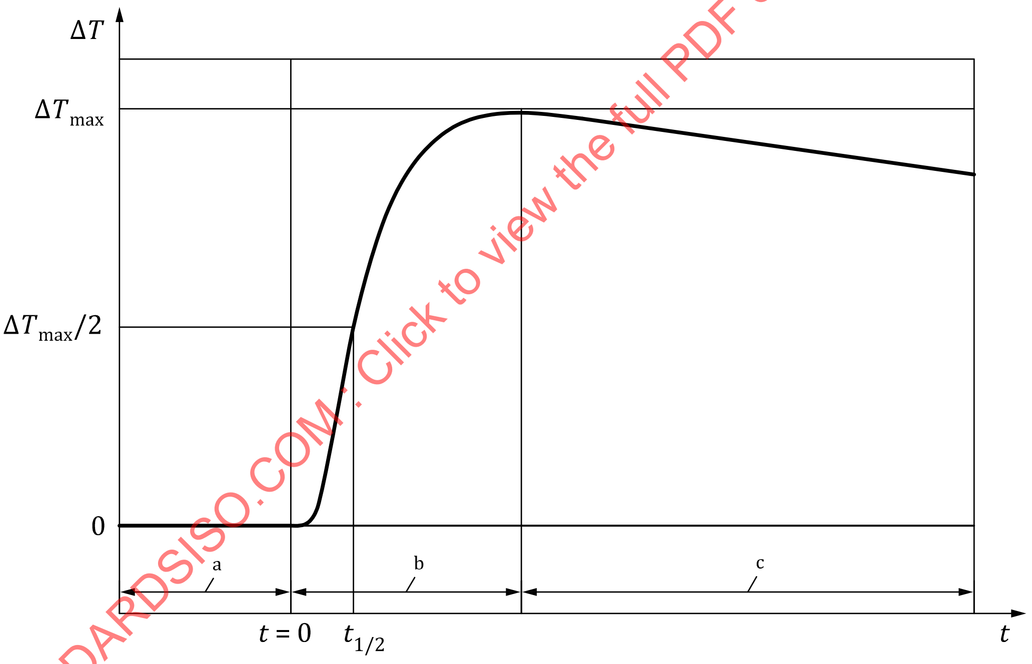
e) If required, the test environment shall be vacuum or inert-gas atmosphere to avoid oxidative degradation during heating and testing of the specimen. For cryoscopic measurements, care shall be taken to avoid water condensation on the windows.

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.



Key

t time

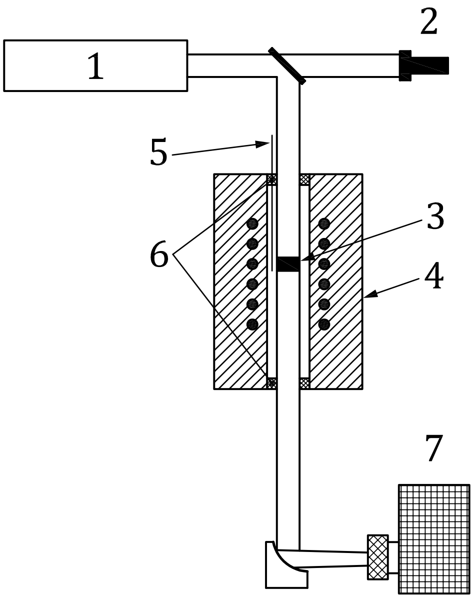
ΔT temperature rise

[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

Figure 2 — Schematic diagram of light 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 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.

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 ±1 × 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.

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.

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.

[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.

8.6 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 light pulse, taking adequate safety precautions. Care shall be taken to ensure that the light 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 light 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 light 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 can 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 light pulse. An extrapolation procedure[3] can be used to calculate the “intrinsic thermal diffusivity” at the test temperature corresponding to the thermal diffusivity that would be measured for a light pulse of zero energy.

9 Data analysis

The analysis of the rear-face temperature curve and the calculation of the thermal diffusivity shall be carried out as follows.

a) If the half-rise time, $t_{1/2}$, is shorter than 100 times the pulse width, correct the time scale for the finite pulse time effect (for guidance, see Annex A).

b) Determine the baseline and the maximum of the curve to calculate the maximum temperature rise, $\Delta T_{\max}$. If the baseline shows a drift, correct the thermogram by subtracting the extrapolated baseline.

c) Normalize the rear-face temperature rise curve by dividing all temperature rise values by $\Delta T_{\max}$.

d) Calculate the thermal diffusivity from the normalized thermogram, using the partial time moments method[4] described below, or any other suitable method, as well as the thickness, d, corrected for thermal expansion.

The “partial time moments” method recommended in this document has a low sensitivity to measurement noise. It uses only the rising part of the thermogram, which is highly sensitive to thermal diffusivity. Other methods may be used as long as their uncertainties are calculated. Annex B gives examples of such methods of calculation.

NOTE 1 Additional information on calculation methods, data analysis and sources of error can be found in ISO 18755[1]. Since ISO 18755 refers to ceramics, not all the details presented therein are necessarily relevant to the testing of plastics.

In the partial time moments method, the thermal diffusivity is given by Formula (2):

$$\alpha = \frac{d^2 F(m_{-1})}{m_0} \tag{2}$$

The two partial time moments, m_0 and m_{-1}, are defined by Formulae (3) and (4):

$$m_0 = \int_{t_{0,1}}^{t_{0,8}} \frac{\Delta T(t)}{\Delta T_{\max}} \mathrm{d}t \tag{3}$$

and

$$m_{-1} = \int_{t_{0,1}}^{t_{0,8}} \frac{1}{t} \times \frac{\Delta T(t)}{\Delta T_{\max}} \mathrm{d}t \tag{4}$$

where

$\Delta T(t)$	is obtained from the experimental thermogram;
$\Delta T_{\max}$	is the maximum temperature rise;
$t_{0,1}$ and $t_{0,8}$	are the times required from the initiation of the pulse for the rear face of the specimen to reach 10 % and 80 % of $\Delta T_{\max}$.

The function $F(m_{-1})$ is defined by Formulae (5) and (6):

For $0{,}44 > m_{-1} > 0{,}27$: $$F(m_{-1}) = 0{,}085\,48 - 0{,}314\,(0{,}548\,6 - m_{-1}) + 0{,}500\,(0{,}548\,6 - m_{-1})^{2{,}63} \tag{5}$$

For $m_{-1} > 0{,}44$: $$F(m_{-1}) = -0{,}081\,9 + 0{,}305\,m_{-1} \tag{6}$$

NOTE 2 Values of m_{-1} less than 0,27 do not have any physical significance.

This calculation method may be used if the following conditions are satisfied:

- the duration of the light pulse is short compared with the time characteristic for thermal diffusion (i.e. pulse width <1 % of $t_{1/2}$) or, if not, a correction is applied to the time scale in accordance with Annex A;
- the front face of the specimen is uniformly heated by the light pulse;
- the specimen is homogeneous;
- the specimen is opaque to the light radiation at the wavelength used.

NOTE 3 The potential effect of non-uniformity of the pulse on the calculated thermal diffusivity can usually be assumed to be negligible due to the use of an IR detector to detect the transient temperature rise of the specimen rear face. However, if needed, the light beam can be made more uniform by using beam-homogenizing optics.

NOTE 4 The assumption of opacity of the specimen is implicitly satisfied by the deposit of an appropriate coating on its two faces.

10 Uncertainty

The relative expanded uncertainty (confidence level of 95 %) of the thermal-diffusivity determination by the light flash method with the partial time moments method is estimated to be from ±3 % to ±5 %[5] between ambient temperature and 400 °C (depending on the material and the temperature). It has been calculated in accordance with the ISO/IEC Guide 98-3[6]. On the basis of the results of five successive measurements performed under the same conditions, the repeatability of the method is better than 1 %.

11 Test report

The test report shall include the following information:

a) a reference to this document, including the year of publication;

b) the date of the test;

c) all details necessary for complete identification of the sample tested (type, batch number, etc.), including its thermal history;

d) the shape and dimensions (diameter and thicknesses at room temperature and at each test temperature, giving the method of calculation of the thicknesses at the test temperatures) of the specimens and the number of specimens tested;

e) details of sample and specimen preparation;

f) whether a coating was used and, if so, the coating material and coating procedure and the thickness of the coating);

g) the type of the light flash source, the wavelength and duration of the light pulse;

h) the type of detector used for measuring the transient temperature rise of the specimen rear face;

i) the type of furnace used and its temperature range (or a statement to the effect that a climatic chamber was used);

j) the measurement conditions, such as the test temperature(s), in degrees Celsius, and the furnace or climatic chamber atmosphere;

k) the method(s) used to calculate the thermal diffusivity and estimate corrections (e.g. due to heat losses and the finite pulse duration);

l) the thermal diffusivity value(s) obtained, in square metres per second;

m) any additional information or details of operations not specified in this document, which can be important for assessment of the results.

Annex A
(informative)

Correction for finite pulse duration

All the methods used to calculate thermal diffusivity assume that the light pulse is similar to a Dirac function. However, if the duration of the pulse cannot be neglected (see Clause 9), the effect of the finite pulse time should be corrected for.

For the methods of calculation based on the use of the half-rise time, $t_{1/2}$, a first approach consists in calculating the thermal diffusivity, assuming a Dirac function pulse, and applying a correction to the value obtained. By specifying the shapes of pulses, several authors[7],[8],[9] have proposed analytical relationships to estimate this correction.

A more general method consists in shifting the time origin to the centroid, t_g, of the light pulse and calculating the thermal diffusivity considering the pulse as a Dirac function. In this case, the waveform of the light pulse is measured by a detector having a response faster than 10 µs and the position of the centroid is calculated directly from the observed waveform. This method, proposed initially by Azumi and Takahashi[10] for adiabatic experiments, has been extended to non-adiabatic ones by Degiovanni[11].

Annex B
(informative)

Alternative methods of calculating thermal diffusivity

All methods of calculating thermal diffusivity are based on the mathematical solution of the heat conduction equation with appropriate initial and boundary conditions. The original method proposed by Parker, et al.[12] is based on an analytical model corresponding to an adiabatic experiment, on the basis of which the thermal diffusivity is calculated from the half-rise time, $t_{1/2}$.

In the case of an adiabatic experiment, the thermal diffusivity is calculated simply from the thickness, d, and the half-rise time, $t_{1/2}$, as follows:

$$\alpha = 0{,}138\ 79 \frac{d^2}{t_{1/2}} \qquad \text{(B.1)}$$

The use of this method, which neglects heat losses, can generate significant systematic errors in thermal diffusivity determinations. It is not a suitable method for polymers because of their low thermal diffusivity and the test cannot really be considered adiabatic.

Some methods are based on an improvement of Parker's method, introducing correction factors in the calculation of the thermal diffusivity to take into account the unavoidable heat losses. They use one or several characteristic points on the experimental curve. Among them, Cape and Lehman's method[13], Cowan's method[14] and Clark and Taylor's method[15] are the most commonly used.

With the advances in modern data acquisition, methods based on the analysis of a part (i.e. the partial time moments method of Degiovanni and Laurent[4]) or the whole of the temperature response (i.e. the technique proposed by Gembarovic, et al.[16] using a least-squares method) are increasingly utilized. These calculation methods are generally based on minimizing the difference between the measured values and the theoretical values obtained from a mathematical model. They differ either by the analytical model used or by the way in which they compare the measured temperature rise versus time recordings with the analytical curve.

Annex C
(normative)

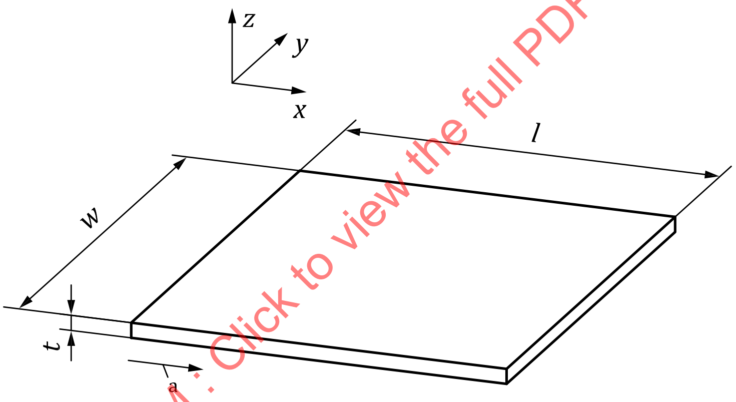
Samples and test specimen preparation for injection mouldable thermoplastics and thermosets

C.1 General

The purpose of this annex is to provide a comparable sample preparation for the measurement of the directional thermal conductivity of injection mouldable thermoplastics and thermoset. This is necessary, because the thermal conductivity and thermal diffusivity values are influenced by the orientation of the filler particles, the sample shape, dimension and preparation. The preparation of the test specimen out of these samples (plates) also has an influence on the above values.

C.2 Sample shape and dimension

Figure C.1 shows an example of a plate.



Key

w	width	z	measuring direction: through-plane
l	length	y	measuring direction: in-plane — perpendicular
t	thickness	x	measuring direction: in plane — parallel
[a]	Flow direction (injection direction).		

Figure C.1 — Example of sample

Minimum dimensions of the plates:

- width w: 40 mm
- length l: 50 mm
- thickness t: 2 mm

The film-gate shall be over the whole width w of the sample (plate).

C.3 Sample manufacturing

Plates shall preferably be injection-moulded from granulate. The injection moulding conditions shall be in accordance with the relevant International Standard for the material or, if none exists, agreed between the interested parties.

Strict control of all conditions of the samples (plates) preparation is essential to ensure that all test specimens in a set are actually in the same state.

Plates shall be coplanar and of uniform thickness. All surfaces shall be free from visible flaws, scratches or other imperfections.

Test specimens from finished goods shall be taken from flat areas or zones having minimum curvature.

Plates should not be machined to reduce their thickness. Test specimens with machined surfaces will not give results comparable to specimens having non-machined surfaces.

If samples (plates) have to be compression-moulded, extruded or machined in accordance with ISO 2818[17], it shall be noted in the test report. In general injection moulded samples are preferred.

C.4 Sample inspection

The sample inspection shall be done in accordance with ISO 527-1.

C.5 Sample anisotropy

Injection moulded samples (plates), as well as finished component parts, exhibit some degree of anisotropy as a result of flow-induced orientation.

The direction dependence of the thermal properties can be assessed by cutting specimens parallel and normal to the flow direction characterizing the moulding process. In the absence of information on such directions, specimens shall be machined in directions as agreed between the interested parties.

- in flow-direction, in-plane: x-direction (see Figure C.1);
- perpendicular to flow-direction, in-plane: y-direction (see Figure C.1);
- perpendicular to flow-direction, through-plane: z-direction (see Figure C.1).

C.6 Location and preparation of the test specimen

C.6.1 General

All test specimens shall be prepared from the middle of the sample with regard to the direction of flow (see Figure C.2 and Figure C.3).