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Plastics — Differential scanning calorimetry (DSC) —

Part 8: Determination of thermal conductivity

Plastiques — Analyse calorimétrique différentielle (DSC) —

Partie 8: Détermination de la conductivité thermique

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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).

A list of all parts in the ISO 11357 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.

Introduction

The advantage of using DSC for measuring the thermal conductivity of plastics is that with the same instrument also the specific heat capacity can be obtained. This enables the determination of the thermal diffusivity by dividing the thermal conductivity by the density and specific heat capacity.

In addition, DSC instruments are widely used and available in almost all test institutes and labs. Hence, measurements of thermal conductivity can be done without need for procurement of an additional instrument.

Plastics — Differential scanning calorimetry (DSC) —

Part 8: Determination of thermal conductivity

1 Scope

This document establishes a method for determination of the thermal conductivity of solid unfilled and filled or fibre reinforced plastics and composites by means of differential scanning calorimetry (DSC).

It is applicable for materials with thermal conductivities of up to 1 W/(m·K).

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

ISO 472, *Plastics — Vocabulary*

ISO 6344-1, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

ISO 11357-1, *Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and ISO 11357-1 apply.

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

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

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

4 Principle

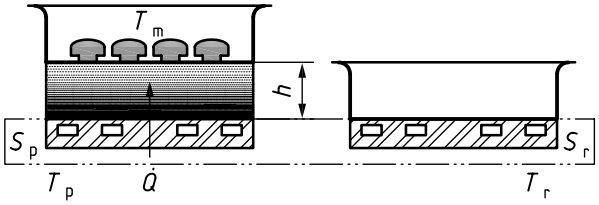
For the determination of thermal conductivity, the usual placement of the specimen in the sample holder position is modified according to a procedure proposed in References [1] and [2] Additional details on scientific background, deduction of results and performance of measurements can be found in References [3] and [4].

An empty crucible is placed in the reference position of the sample holder assembly. The test specimen is placed directly onto the sensor of the sample position and a crucible containing a substance of known melting temperature is put on top of the specimen (see Figure 1). The thermal conductivity is measured at a temperature slightly above the melting point of this substance in the small temperature range in which the slope of the melting peak with test specimen is determined (see 9.4, Figure 2).

Upon heating, a temperature gradient is created in the specimen. The temperature of the top of the specimen remains constant at the melting temperature T_m of the melting substance while the temperature of the bottom side of the specimen corresponds to the temperature of the sample side

sensor. The differential heat flow between sample and reference sensor is measured by the DSC instrument.

From the temperature difference between top and bottom of the sample (T_m - T_p), the sample height h and the heat flow rate difference $\dot{Q}$ between sample and the reference sensor, the thermal conductivity of the specimen is determined.



Key

h	height of specimen
$\dot{Q}$	heat flow rate difference between sample and reference position
S_p	sensor of sample position
S_r	sensor of reference position
T_m	melting temperature of the known substance and the temperature of the top of the specimen
T_p	temperature of the sample position and the bottom side of the specimen
T_r	temperature of the reference position

Figure 1 — Schematic layout of measurement

5 Apparatus and substances

5.1 DSC instrument

A DSC instrument in accordance with specifications given in ISO 11357-1 shall be used.

Only original raw data shall be used without any further data processing.

NOTE According to currently available results, the method has been verified with heat-flux type DSC instruments only. However, it is expected that power-compensated DSC instruments are also suitable.

5.2 Crucibles

Aluminium crucibles shall be used fitting the DSC instrument. Both, open or ventilated closed crucibles are suitable. Upon using sealed crucibles suitable precautions shall be taken to prevent excessive inside pressure.

Care shall be taken to avoid deformation of the plane crucible bottom.

NOTE Uneven crucible bottoms can result in too low thermal conductivity values.

5.3 Melting substance

Certified melting substances or melting substances having a purity of 99,999 9 % or better shall be used.

The melting temperature determines the temperature of measurement of the thermal conductivity.

Melting temperatures of specimens to be measured shall be above those of the melting substances to be used. In case of doubt, this shall be ensured by performing suitable preliminary tests.

NOTE Metals such as gallium or indium have been found suitable.

5.4 Protective lacquer

If metals that can react with the crucible material are used as melting substances, protective lacquers may be applied for coating the crucible inside surface and preventing formation of alloys with the aluminium crucibles.

NOTE 1 Gallium, for example, is known to form alloys with aluminium.

The protective coating shall be thermally stable in the measurement temperature range and shall be applied continuously on the crucible inside surface in constant thickness of not more than 30 µm.

Both, the sample crucible containing the melting substance and the reference crucible shall be coated in exactly the same manner.

NOTE 2 Suitable examples for protective lacquers are, for example, nitro lacquer or permanent marker ink.

5.5 Length measuring device

Suitable device for measuring the diameter with an accuracy of ±20 µm or better and the specimen height with an accuracy of ±10 µm or better.

6 Specimens

6.1 Geometry

Cylindrical specimens shall be used.

The diameter shall be measured with an accuracy of ±20 µm or better and shall not deviate from the diameter of the bottom of the crucible by more than 0,1 mm. The height shall be measured with an accuracy of ±10 µm or better and shall be in the range of 0,5 mm to 2,0 mm.

To ensure the best possible thermal contact to the sensor and crucible, specimens shall be free of scars and burrs and shall have plane-parallel upper and lower surfaces. Grind the upper and lower surface using ultra-fine abrasive paper of grit size P800 in accordance with the ISO/FEPA scale specified in ISO 6344-1 or better.

NOTE Higher surface roughness can result in reduced thermal contact and lower values of thermal conductivity.

6.2 Number and sampling

At least five specimens shall be measured and the results averaged.

For homogeneous filled and unfilled materials, no particular sampling procedures are required.

For fibre reinforced laminates and composites, specimens shall be taken at least 10 mm away from sheet edges. Specimens shall be taken from several homogeneously distributed locations of the sheet with exclusion of defective areas.

Depending on sample preparation and for samples with anisotropic reinforcements, the thermal conductivity can depend on the orientation of the test specimen. In such cases, the orientation of the test specimen shall be noted.

7 Conditioning

Prior to measurement specimens shall be conditioned for a minimum of 2 h in accordance with ISO 291.

Moisture sensitive materials shall be dried preferably in accordance with relevant material standards. Alternatively, drying conditions shall be agreed between involved parties.

8 Calibration

Calibration of the DSC instrument with regard to temperature and heat shall be done in accordance with ISO 11357-1.

9 Measurement procedure

9.1 Preparation of crucibles

Two identical crucibles shall be selected having a mass difference of not more than 0,1 mg.

When using materials as melting substances that can react with the crucibles and form alloys, for example gallium, the crucible inside surface shall be coated with protective lacquer in accordance with 5.4.

One of the crucibles remains empty and is used as reference. The other crucible is filled with the melting substance in accordance with 5.3.

The melting substance shall be placed centrically in the crucible. Preferably, the melting substance should cover the bottom of the crucible completely.

NOTE In case of gallium, a sample weight of approximately 60 mg to 65 mg has been found suitable.

9.2 Use of thermal contact substances

Preferably, thermal contact substances should not be used with specimens having smooth top and bottom surfaces as this can increase the standard deviation of measured thermal conductivity values and residues can remain on the sensors that can falsify subsequent measurements if not completely removed.

For specimens with very rough surfaces, for example fibre reinforced composites or laminates, thermal contact substances may exceptionally be used on the top and bottom specimen surfaces. In such cases, the same thermal contact substance shall also be used on the reference crucible.

If required, thin layers of synthetic oil shall be used as thermal contact aid. Only oil shall be used that can be removed residue-free upon heating the DSC sensors at a temperature of 500 °C.

The suitability of oil should be checked with the supplier or determined by own testing, for example, based on thermogravimetry.

9.3 Measurement of melting substance

9.3.1 Placing of crucibles in sample holder

The empty crucible according to 9.1 shall be placed in the reference position and the crucible filled with the melting substance according to 9.1 shall be placed in the sample position without test specimen. The introduction of crucibles shall be done at room temperature. The sample holder shall be closed and purged with inert gas in accordance with ISO 11357-1 to prevent oxidation of the melting substance.

9.3.2 Distribution of melting substance on crucible ground

For improved distribution of the melting substance on the crucible ground the following temperature program shall be applied.

a) Bring the sample holder to a temperature of 10 K below the melting point at a scan rate of 10 K/min.

b) Heat up to 20 K above the melting point at a scan rate of 2 K/min.

c) Hold this temperature for 10 min to ensure complete melting.

d) Cool down to 30 K below the melting point at a scan rate of 5 K/min to ensure full crystallisation.

e) Bring the sample holder to room temperature for removal of crucibles.

9.3.3 Measurement of the slope of the melting curve without test specimen

Thereafter, for evaluation of the upward slope of the melting peak of the melting substance, the following temperature program shall be applied.

a) Bring the sample holder to a temperature of 30 K below the melting point at a scan rate of 10 K/min.

b) Hold this temperature for 5 min.

c) Heat up to a temperature of 10 K below the melting point at a scan rate of 10 K/min.

d) Heat up to a temperature of 4 K below the melting point at a scan rate of 5 K/min.

e) Hold this temperature for 2 min.

f) Heat up to a temperature of 10 K above the melting point at a scan rate of 0,5 K/min.

g) Cool down to a temperature of 30 K below the melting point at a scan rate of 5 K/min.

h) Bring the sample holder to room temperature for insertion of the test specimen.

Step e) of 9.3.2 and steps a) and b) of 9.3.3 may be skipped if the measurement of the slope of the melting curve without test specimen is done immediately after distribution of the melting substance on the crucible ground.

9.3.4 Insertion of test specimen

After removing the sample crucible, the test specimen shall be placed centrically onto the sample position and then the crucible filled with the melting substance put centrically, too, on top of the test specimen.

The empty reference crucible remains on the reference position.

9.3.5 Measurement of the slope of the melting curve with test specimen

After inserting the test specimen, the following temperature program shall be applied.

a) Bring the sample holder to a temperature of 30 K below the melting point of the melting substance at a scan rate of 10 K/min.

b) Hold this temperature for 5 min.

c) Heat up to a temperature of 10 K below the melting point at a scan rate of 10 K/min.

d) Heat up to a temperature of 4 K below the melting point at a scan rate of 5 K/min.

e) Hold this temperature for 2 min.

f) Heat up to a temperature of 10 K above the melting point at a scan rate of 0,5 K/min.

g) Cool down to a temperature of 30 K below the melting point at a scan rate of 5 K/min.

h) Bring the sample holder to room temperature for exchanging the test specimen.

9.4 Evaluation of the slope of the peaks of the melting substance

The evaluation of both melting curves recorded without (see 9.3.3) and with (see 9.3.5) test specimen shall be done using the curves of recorded heat flow rate vs. sample temperature as shown in Figure 2.