SLOVENSKI STANDARD
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Polimerni materiali - Diferenčna dinamična kalorimetrija (DSC) - 4. del: Ugotavljanje specifične toplotne kapacitete (ISO/DIS 11357-4:2020)

Plastics - Differential scanning calorimetry (DSC) - Part 4: Determination of specific heat capacity (ISO/DIS 11357-4:2020)

Kunststoffe - Dynamische Differenz-Thermoanalyse (DSC) - Teil 4: Bestimmung der spezifischen Wärmekapazität (ISO/DIS 11357-4:2020)

Plastiques - Analyse calorimétrique différentielle (DSC) - Partie 4: Détermination de la capacité thermique massique (ISO/DIS 11357-4:2020)

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Part 4: Determination of specific heat capacity

Plastiques — Analyse calorimétrique différentielle (DSC) —

Partie 4: Détermination de la capacité thermique massique

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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 of 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 www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This third edition cancels and replaces the second edition (ISO 11357-4:2014), which has been technically revised. The main changes compared to the previous edition are as follows:

a) all normative references were changed into undated ones;

b) the term "pan" was replaced by "crucible" within the whole text;

c) the endothermic direction, a, was added in all figures and key.

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.

Plastics — Differential scanning calorimetry (DSC) —

Part 4: Determination of specific heat capacity

1 Scope

This document specifies methods for determining the specific heat capacity of plastics by differential scanning calorimetry.

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*

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

ISO 80000-1, *Quantities and units — Part 1: General*

3 Terms and definitions

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

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

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

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

3.1
calibration material
material of known specific heat capacity

Note 1 to entry: Usually, α-alumina (such as synthetic sapphire) of 99,9 % or higher purity is used as the calibration material.

3.2
specific heat capacity (at constant pressure)
c_p
quantity of heat necessary to raise the temperature of unit mass of material by 1 K at constant pressure

Note 1 to entry: It is given by the following formula:

$$c_p = m^{-1} C_p = m^{-1} \left(\frac{dQ}{dT} \right)_p \quad (1)$$

where

c_p is the specific heat capacity and is expressed in kilojoules per kilogram per K ($kJ \cdot kg^{-1} \cdot K^{-1}$) or in joules per K ($J \cdot g^{-1} \cdot K^{-1}$); subscript p indicates an isobaric process;

m is the mass of material, expressed in kilogram (kg) or gram (g);

C_p is the total heat capacity and is expressed in kilojoules per K ($kJ \cdot K^{-1}$) or in joules per K ($J \cdot K^{-1}$); subscript p indicates an isobaric process;

dQ is the quantity of heat necessary to raise the temperature of the material by dT, expressed in kilojoules per K ($kJ \cdot K^{-1}$) or in joules per K ($J \cdot K^{-1}$).

This formula is valid in a temperature range where a material shows no first-order phase transition.

$$(dQ/dT) = (dt/dT) \times (dQ/dt) = (\text{heatingrate})^{-1} \times (\text{heatflowrate}) \quad (2)$$

Note 2 to entry: At phase transitions, there is a discontinuity in the heat capacity. Part of the heat is consumed to produce a material state of higher energy and it is not all used in raising the temperature. For this reason, the specific heat can only be determined properly outside regions of phase transitions.

4 Principle

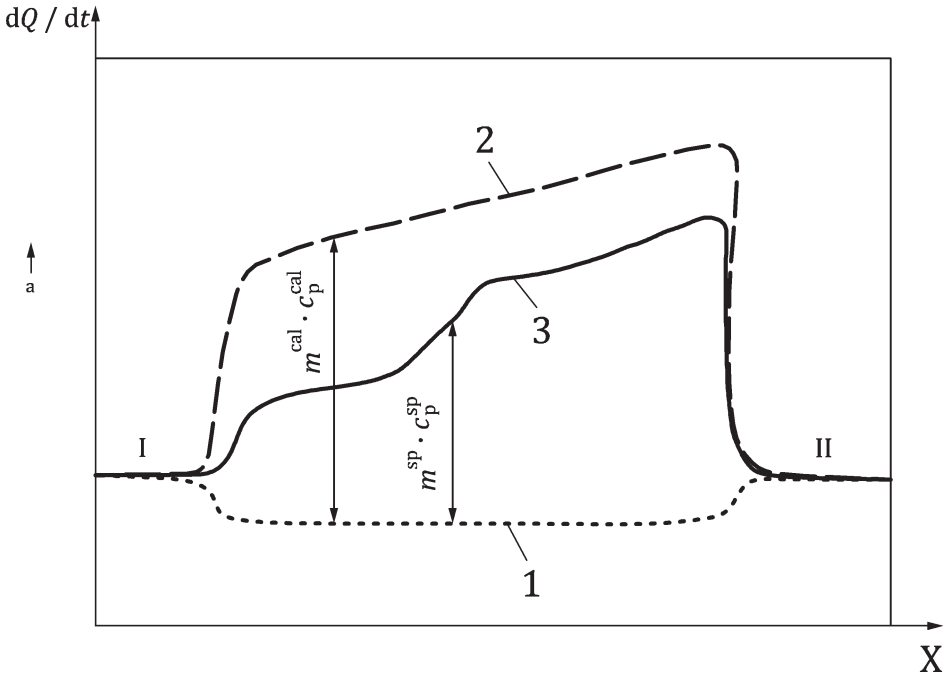
4.1 General

Each measurement consists of three runs at the same scanning rate (see Figure 1):

a) a blank run (empty crucibles in sample and reference holders);

b) a calibration run (calibration material in sample holder crucible and empty crucible in reference holder);

c) a specimen run (specimen in sample holder crucible and empty crucible in reference holder).



Key

X time t
1 blank run
2 calibration run
3 specimen run
I isothermal baseline at start temperature T_s
II isothermal baseline at end temperature T_f
a Endothermic direction.

Figure 1 — Schematic drawing of typical DSC curves for specific heat capacity measurement (blank, calibration and specimen runs) after baseline adjustment

4.2 Continuous-scanning method

Based on the DSC principle (see ISO 11357-1) and the definition of specific heat capacity given in 3.2, the following relations can be obtained:

$$m^{sp} \cdot c_p^{sp} \propto P_{specimenrun} - P_{blankrun} \tag{3}$$

$$m^{cal} \cdot c_p^{cal} \propto P_{calibrationrun} - P_{blankrun} \tag{4}$$

where P is the heat flow rate (dQ/dt); superscripts sp and cal represent specimen and calibration material, respectively (see Figure 1).

When $P_{specimen\ run}$, $P_{calibration\ run}$ and $P_{blank\ run}$ are measured, c_p^{sp} can be calculated using Formula (6), since the values of c_p^{cal}, m^{sp} and m^{cal} are known:

$$\frac{m^{sp} \cdot c_p^{sp}}{m^{cal} \cdot c_p^{cal}} = \frac{P_{specimenrun} - P_{blankrun}}{P_{calibrationrun} - P_{blankrun}} \tag{5}$$

$$c_{\mathrm{p}}^{\mathrm{sp}} = c_{\mathrm{p}}^{\mathrm{cal}} \cdot \frac{m^{\mathrm{cal}} \left(P_{\mathrm{specimenrun}} - P_{\mathrm{blankrun}} \right)}{m^{\mathrm{sp}} \left(P_{\mathrm{calibrationrun}} - P_{\mathrm{blankrun}} \right)} \tag{6}$$

4.3 Stepwise-scanning method

In the stepwise-scanning method, the total temperature range to be scanned is divided into small intervals and a complete determination consisting of the three runs mentioned in 4.1 is performed for each temperature interval. Upon integration of the heat flow rate curve, the total heat ΔQ consumed in the interval can be obtained. Dividing ΔQ by the temperature interval ΔT and the mass of the specimen gives the specific heat [see Formula (1)]:

$$m^{\mathrm{sp}} \cdot c_{\mathrm{p}}^{\mathrm{sp}} \propto \left(\frac{\Delta Q^{\mathrm{sp}}}{\Delta T} \right)_{\mathrm{p}} - \left(\frac{\Delta Q^{\mathrm{blank}}}{\Delta T} \right)_{\mathrm{p}} \tag{7}$$

$$m^{\mathrm{cal}} \cdot c_{\mathrm{p}}^{\mathrm{cal}} \propto \left(\frac{\Delta Q^{\mathrm{cal}}}{\Delta T} \right)_{\mathrm{p}} - \left(\frac{\Delta Q^{\mathrm{blank}}}{\Delta T} \right)_{\mathrm{p}} \tag{8}$$

Keeping the temperature intervals ΔT constant, combining Formula (7) and Formula (8) results in:

$$c_{\mathrm{p}}^{\mathrm{sp}} = c_{\mathrm{p}}^{\mathrm{cal}} \cdot \frac{m^{\mathrm{cal}}}{m^{\mathrm{sp}}} \cdot \frac{\Delta Q^{\mathrm{sp}} - \Delta Q^{\mathrm{blank}}}{\Delta Q^{\mathrm{cal}} - \Delta Q^{\mathrm{blank}}} \tag{9}$$

5 Apparatus

5.1 DSC apparatus. See ISO 11357-1.

5.2 Crucibles. See ISO 11357-1.

The crucibles for the test specimen and the reference specimen (calibration material) shall be of the same shape and material and their masses shall not differ by more than 0,1 mg.

NOTE The same blank run and calibration run can be used for several measurements, if the instrument is sufficiently stable and the difference in mass between the calibration material crucible and the empty crucible is corrected for. An adequate correction can be obtained by adding the term $c_{\mathrm{p,\,crucible}}(T)\beta\Delta m$ to the heat flow rate of the calibration run, where $c_{\mathrm{p,crucible}}(T)$ is the specific heat capacity of the calibration crucible as a function of temperature, β is the heating rate and Δm is the difference in mass between the calibration crucible and the empty crucible. The same procedure can also be used for correcting differences in mass between the specimen run and the blank run.

5.3 Analytical balance. See ISO 11357-1.

6 Test specimen

See ISO 11357-1.

7 Test conditions and specimen conditioning

See ISO 11357-1.