FINAL DRAFT

INTERNATIONAL STANDARD

ISO/FDIS 11357-4

ISO/TC 61/SC 5

Secretariat: DIN

Voting begins on: **2020-11-02**

Voting terminates on: 2020-12-28

Plastics — Differential scanning calorimetry (DSC) —

Part 4: Determination of specific heat capacity

iTeh STPlastiques – Analyse calorimétrique différentielle (DSC) – Partie 4: Détermination de la capacité thermique massique

ISO/FDIS 11357-4 https://standards.iteh.ai/catalog/standards/sist/03fc802c-6d06-41ac-9d28-9d4a19375007/iso-fdis-11357-4

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Published in Switzerland

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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).

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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 11357-4:2014), which has been technically revised. The main changes compared to the previous edition are as follows:

- the measurement procedure has been updated;
- reference data of α -alumina have been updated.

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 <u>www.iso.org/members.html</u>.

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 **Standards.iteh.ai**)

3 Terms and definitions ISO/FDIS 11357-4

For the purposes of this document, the terms and definitions given in ISO 472, 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 <u>https://www.iso.org/obp</u>

— 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

 q^{ν} 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:

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

where

- c_p is the specific heat capacity and is expressed in kilojoules per kilogram per K (kJ·k- g^{-1} ·K⁻¹) or in joules per gram per K (J· g^{-1} ·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·K⁻¹) or in joules per K (J·K⁻¹); subscript *p* indicates an isobaric process;
- $\left(\frac{dQ}{dT}\right)_p$ is the quantity of heat dQ necessary to raise the temperature of the material by dT, expressed in kilojoules per K (kJ·K⁻¹) or in joules per K (J·K⁻¹), measured at constant pressure.

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

The quotient
$$\left(\frac{dQ}{dT}\right)$$
 can be obtained by dividing the heat flow rate by the heating rate:
 $\left(\frac{dQ}{dT}\right) = \frac{\left(\frac{dQ}{dt}\right)}{\left(\frac{dT}{dt}\right)}$

where

(dQ/dt) is the heat flow rate, expressed in kilojoules per second (kJ·s⁻¹) or in joules per second (J·s⁻¹) or in watts (W);

(*dT/dt*) is the heating rate, expressed in kelvins (K) per second (s);

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.

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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).



Кеу

- 1 blank run
- 2 calibration run
- 3 specimen run
- I isothermal baseline at start temperature, **DARD PREVIEW**
- II isothermal baseline at end temperature Tlards.iteh.ai)
- ^a Endothermic direction.

ISO/FDIS 11357-4

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 at constant pressure given in <u>3.2</u>, the following relations shown as <u>Formulae (1)</u> and <u>(2)</u> can be obtained:

$$m^{\rm sp} \cdot c_p^{\rm sp} \propto P_{\rm sp} - P_{\rm bl} \tag{1}$$

$$m^{\rm cal} \cdot c_p^{\rm cal} \propto P_{\rm cal} - P_{\rm bl} \tag{2}$$

where

- *P* is the heat flow rate (dQ/dt);
- ^{sp} is the specimen;
- ^{bl} is an empty crucible (blank run);
- ^{cal} is the calibration material.

See <u>Figure 1</u>.

When P_{spn} , P_{ca} and P_{bl} are measured, c_p^{sp} can be calculated using Formula (4), since the values of c_p^{cal} , m^{sp} and m^{cal} are known:

$$\frac{m^{\rm sp} \cdot c_p^{\rm sp}}{m^{\rm cal} \cdot c_p^{\rm cal}} = \frac{P_{\rm sp} - P_{\rm bl}}{P_{\rm cal} - P_{\rm bl}}$$
(3)

$$c_p^{\rm sp} = c_p^{\rm cal} \cdot \frac{m^{\rm cal} \left(P_{\rm sp} - P_{\rm bl} \right)}{m^{\rm sp} \left(P_{\rm cal} - P_{\rm bl} \right)} \tag{4}$$

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 3.2):

$$m^{\rm sp} \cdot c_p^{\rm sp} \propto \left(\frac{\Delta Q}{\Delta T}^{\rm sp}\right)_p - \left(\frac{\Delta Q}{\Delta T}^{\rm bl}\right)_p \tag{5}$$
$$m^{\rm cal} \cdot c_p^{\rm cal} \propto \left(\frac{\Delta Q}{\Delta T}^{\rm cal}\right)_p - \left(\frac{\Delta Q}{\Delta T}^{\rm bl}\right)_p \textbf{STANDARD PREVIEW} \tag{6}$$

Keeping the temperature intervals ΔT constant, combining Formula (5) and Formula (6) results in:

 $c_{p}^{\rm sp} = c_{p}^{\rm cal} \cdot \frac{m^{\rm cal}}{m^{\rm sp}} \cdot \frac{\Delta Q^{\rm sp} - \Delta Q^{\rm bl}}{\Delta Q^{\rm cal} - \Delta Q^{\rm bl}} \underbrace{ISO/FDIS 11357-4}_{9d4a19375007/iso-fdis-11357-4}$ (7)

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 $c_{p, \text{crucible}}(T)\beta\Delta m$ to the heat flow rate of the calibration run, where $c_{p,\text{crucible}}(T)$ 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.

8 Procedure

8.1 Selection of crucibles

Prepare three crucibles and their lids and weigh the crucibles together with their lids. The total mass shall not differ by more than 0,1 mg (see <u>5.2</u>). In other respects, such as material, size, crucible type (open or sealed), the crucibles shall be identical.

NOTE It is also possible to use the same crucible in the sample holder for blank run, calibration run, and specimen run if the lid is just laid upon the crucible for blank run and calibration run. For the specimen run, the crucible can be hermetically sealed.

8.2 Setting up the apparatus and adjustment of isothermal baselines

8.2.1 Place a pair of empty crucibles with lids in the DSC sample and reference holders.

- **8.2.2** If using a continuous-scan programme:
- a) Set the start and end temperatures (T_s and T_f). The start temperature T_s should be at least 30 K lower than that at which data are first required. PREVE

When more precise results are **required over a wide temper**ature range, the overall range may be divided into two (or more) smaller ranges, each 50 K to 100 K wide. The start temperature T_s of the second range should be 30 K lower than the end temperature T_f of the first temperature range to ensure sufficient overlap.

sufficient overlap. https://standards.iteh.ai/catalog/standards/sist/03fc802c-6d06-41ac-9d28-9d4a19375007/iso-fdis-11357-4

- b) Set the scanning rate.
- c) Set the dwell time of the isothermal stages I and II (see <u>Figure 1</u>) and allow the respective isothermal baselines to stabilize. This dwell time will usually be between 2 min and 10 min.

NOTE Some calorimeters, such as those of the Calvet type, can require up to 30 min before the baseline stabilizes.

8.2.3 If using a stepwise-scan programme:

When the specific heat capacities of the samples do not significantly depend on the temperature, the stepwise-scanning method may be used in which the integration of the heat flow over small temperature intervals gives a set of individual specific heat values for the temperature intervals considered. Attention shall be paid to the following points.

- a) The dwell time of the isothermal stages shall be sufficiently long to obtain a stable baseline.
- b) This method shall not be used over a temperature range in which first-order phase transitions occur.

The stepwise scan is performed as follows.

- Set the start and end temperatures (T_s and T_f).
- Set the temperature increment preferably to 5 K or 10 K.
- Set the temperature-scanning rate to 5 K·min⁻¹ or 10 K·min⁻¹.
- Set the dwell time of the isothermal stages, usually to between 2 min and 10 min.