SLOVENSKI STANDARD
oSIST prEN ISO 11357-3:2017

01-junij-2017

Polimerni materiali - Diferenčna dinamična kalorimetrija (DSC) - 3. del: Ugotavljanje temperature in entalpije taljenja in kristalizacije (ISO/DIS 11357-3:2017)

Plastics - Differential scanning calorimetry (DSC) - Part 3: Determination of temperature and enthalpy of melting and crystallization (ISO/DIS 11357-3:2017)

Kunststoffe - Dynamische Differenz-Thermoanalyse (DSC) - Teil 3: Bestimmung der Schmelz- und Kristallisationstemperatur und der Schmelz- und Kristallisationsenthalpie (ISO/DIS 11357-3:2017)

Plastiques - Analyse calorimétrique différentielle (DSC) - Partie 3: Détermination de la température et de l'enthalpie de fusion et de cristallisation (ISO/DIS 11357-3:2017)

Ta slovenski standard je istoveten z: **prEN ISO 11357-3**

ICS:

17.200.10	Toplota. Kalorimetrija	Heat. Calorimetry
83.080.01	Polimerni materiali na splošno	Plastics in general

oSIST prEN ISO 11357-3:2017 **en,fr,de**

DRAFT INTERNATIONAL STANDARD

ISO/DIS 11357-3

ISO/TC **61**/SC **5** Secretariat: **DIN**

Voting begins on: **2017-04-06**

Voting terminates on: **2017-06-28**

Plastics — Differential scanning calorimetry (DSC) —

Part 3: Determination of temperature and enthalpy of melting and crystallization

Plastiques — Analyse calorimétrique différentielle (DSC) —

Partie 3: Détermination de la température et de l'enthalpie de fusion et de cristallisation

ICS: 83.080.01

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 11357-3:2017(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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.

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 11357-3:2011), which has been technically revised.

A list of all parts in the ISO 11357 series can be found on the ISO website.

Plastics — Differential scanning calorimetry (DSC) — Part 3: Determination of temperature and enthalpy of melting and crystallization

1 Scope

This document specifies a method for the determination of the temperatures and enthalpies of melting and crystallization of crystalline or partially crystalline plastics.

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*

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:

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

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

3.1
melting
transition stage between a fully crystalline or partially crystalline solid state and an amorphous liquid of variable viscosity

Note 1 to entry: The transition, also referred to as "fusion", is characterised by an endothermic peak in the DSC curve. An exception to this definition is the case of liquid crystals, where the term "amorphous liquid" is replaced by "ordered liquid".

3.2
crystallization
transition stage between an amorphous liquid state and a fully crystalline or partially crystalline solid state

Note 1 to entry: The transition is characterised by an exothermic peak in the DSC curve. An exception to this definition is the case of liquid crystals, where the term "amorphous liquid" is replaced by "ordered liquid".

3.3
enthalpy of fusion
heat required to melt a material at constant pressure

Note 1 to entry: It is expressed in kilojoules per kilogram (kJ/kg) or joules per gram (J/g).

3.4
enthalpy of crystallization
heat released by the crystallization of a material at constant pressure

Note 1 to entry: It is expressed in kilojoules per kilogram (kJ/kg) or joules per gram (J/g).

4 Principle

See ISO 11357-1.

5 Apparatus and materials

Apparatus and materials shall be in accordance with ISO 11357-1.

6 Test specimen

The test specimen shall be in accordance with ISO 11357-1.

7 Test conditions and specimen conditioning

The test conditions and specimen conditioning shall be in accordance with ISO 11357-1.

8 Calibration

Calibration shall be in accordance with ISO 11357-1.

9 Procedure

9.1 Setting up the apparatus

The setting up of the apparatus shall be in accordance with ISO 11357-1.

9.2 Loading the test specimen into the crucible

The loading of the test specimen shall be in accordance with ISO 11357-1.

Unless otherwise specified in the material standard, preferably use a mass of 5 mg to 10 mg for the measurement. In the case of high or low heats of transition, masses lower or higher than 5 mg to 10 mg, respectively, may be used.

9.3 Insertion of crucibles

The insertion shall be in accordance with ISO 11357-1.

9.4 Temperature scan

9.4.1 Heating and cooling rates other than those recommended here may be used by agreement between the interested parties. In particular, high scanning rates result in better sensitivity of the recorded transition. On the other hand, low scanning rates provide better resolution in temperature and may be appropriate in the resolution of closely overlapping transitions.

9.4.2 Allow 5 min for a nitrogen pre-purge prior to beginning the heating cycle.

9.4.3 Perform and record a first heating run, heating the cell to a temperature high enough to erase the test material's previous thermal history, typically 30 K above the extrapolated end melting temperature, T_{efm}. Preferred heating rates for the first heating run are 10 K/min or 20 K/min, but also higher heating rates may be used to delete the thermal history of a sample within the first heating run.

DSC measurements on polymers are greatly affected by the thermal history and morphology of the sample and the test specimen. It is important that the preliminary heat cycle be performed and the measurements be taken from the second heat scan (see ISO 11357-1). In cases where the material is reactive or where it is desired to evaluate the properties of a specially pre-conditioned specimen, data may be taken during the first heating cycle. This deviation from the standard procedure shall be recorded in the test report.

9.4.4 Hold the temperature for 5 min.

NOTE Longer times may be acceptable or needed provided degradation of the polymer does not result.

9.4.5 Perform and record a cooling run at preferably 10 K/min or 20 K/min to approximately 50 K below the extrapolated end crystallization temperature, T_{efc}.

NOTE 1 Because of supercooling, crystallization does not occur until a sufficient temperature gradient is available, usually significantly below the melting temperature.

NOTE 2 If glass transitions are to be evaluated, too, cooling should be done 50 K below the glass transition temperature.

9.4.6 Hold the temperature for 5 min.

9.4.7 Perform and record a second heating run at preferably the same heating rate as the cooling run (see 9.4.5) to approximately 30 K higher than the extrapolated end melting temperature, T_{efm}.

NOTE It is important to create a defined thermal history in order to evaluate correct results.

9.4.8 Bring the apparatus to ambient temperature and remove the crucibles to determine if deformation of the crucible or specimen overflow has occurred.

9.4.9 Reweigh the crucible with the test specimen, unless it is known that the material will suffer no loss in mass during the experiment.

In case of significant weight loss repetition of measurements with stronger dried specimens shall be considered.

10 Expression of results

10.1 Determination of transition temperatures

Scale the plot so that the peak covers at least 25 % of full scale. Construct a baseline to the peak (see Figure 1) by joining the peak initiation temperature, T_{im}, and end temperature, T_{fm}, at which the peak (endothermic peak for fusion, exothermic peak for crystallization) begins to deviate from the relatively straight baseline. If multiple peaks are present, a baseline shall be drawn, covering all peaks. If possible,the evaluation shall then be divided between each peak, in order to get the most accurate enthalpy. Suitable peak separation techniques may be used and mentioned in the test report.

For a melting transition curve, measure and report the peak melting temperature, T_{pm}, for each peak.

Reporting onset of melting is acceptable if requested.

For a crystallization transition curve, measure and report for each peak:

— the extrapolated onset crystallization temperature, T_{eic};

— the peak crystallization temperature, T_{pc}.

Extrapolated onset and end temperatures need to be reported if the width of the peak is of interest.

10.2 Determination of enthalpies (see Figure 1)

Measure the area under the peak to the baseline constructed in accordance with 10.1.

Calculate the enthalpy of fusion, ΔH_f [enthalpy of crystallization, ΔH_C], in kilojoules per kilogram (kJ/kg), using the following equation:

$$\Delta H = \Delta H_C \cdot \frac{A \cdot m_C}{A_C \cdot m}$$

where

ΔH is the enthalpy of fusion or crystallization of the specimen (kJ/kg);

ΔH_C is the enthalpy of fusion or crystallization of the calibration material (kJ/kg);

A is the peak area for the specimen (mm^2);

A_C is the peak area for the calibration material (mm^2);

m is the mass of the specimen (mg);

m_C is the mass of the calibration material (mg).

NOTE 1 The enthalpy calculation may be facilitated using computer aided analysis.

NOTE 2 In the event of significant differences between the specific heat capacities of the solid and liquid states of the polymer, the use of special types of baseline, such as sigmoidal baselines, may improve the results.

