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Plastics — Differential scanning calorimetry (DSC) —

Part 2: Determination of glass transition temperature and step height

Plastiques — Analyse calorimétrique différentielle (DSC) —

Partie 2: Détermination de la température et de la hauteur de palier de transition vitreuse

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Foreword

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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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For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

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 11357-2:2013), which has been technically revised. The main changes compared to the previous edition are as follows:

— revision of definition of glass transition step height;

— correction of unit of glass transition step height;

— assessment of methods for determination of T_g;

— revision of rounding of T_g;

— strong restriction of re-using crucibles.

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 2: Determination of glass transition temperature and step height

1 Scope

This document specifies methods for the determination of the glass transition temperature and the step height related to the glass transition of amorphous and 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:

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

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

3.1
glass transition temperature
T_g
characteristic value of the temperature range over which the glass transition takes place

Note 1 to entry: The assigned glass transition temperature (T_g) may vary, depending on the specific property and on the method and conditions selected to measure it.

3.2
glass transition step height
$\Delta c_p(T_g)$
difference of specific heat capacity of the upper and lower extrapolated baselines at T_g

Note 1 to entry: See Figure 1 and Figure 2.

Note 2 to entry: For partially crystalline polymers, the glass transition step height is proportional to the amorphous content.

4 Principle

The principle is specified in ISO 11357-1.

The change in heat flow rate as a function of temperature is recorded and the glass transition temperature and step height are determined from the curve thus obtained.

5 Apparatus and materials

The apparatus and materials shall be as specified in ISO 11357-1.

6 Test specimens

The test specimens shall be as specified in ISO 11357-1.

7 Test conditions and specimen conditioning

The test conditions and specimen conditioning shall be as specified in ISO 11357-1.

8 Calibration

The calibration shall be as specified in ISO 11357-1.

9 Procedure

9.1 Setting up the apparatus

The procedure for setting up the apparatus shall be as specified in ISO 11357-1.

9.2 Loading the test specimen into the crucible

The procedure for loading the test specimen into the crucible shall be as specified in ISO 11357-1.

Determine the mass of the test specimen to the nearest 0,1 mg. Unless otherwise specified in the materials standard, use a mass of between 5 mg and 20 mg. For partially crystalline materials, use a mass near the higher limit.

9.3 Insertion of crucibles

The procedure for inserting the crucibles shall be as specified in ISO 11357-1.

9.4 Temperature scan

9.4.1 Allow 5 min pre-purge prior to beginning the heating cycle.

9.4.2 Perform and record a preliminary thermal cycle at a preferred scan rate of 20 K/min, heating the cell to a temperature high enough to erase the test material's previous thermal history.

If the loading temperature is sufficiently high above the glass transition temperature, preliminary heating can be skipped, and the temperature scan continued with 9.4.3.

DSC measurements on polymers are greatly affected by the thermal history and morphology of the sample and the test specimen. A first heating scan shall be performed using the test specimen as received and measurements shall be taken preferably from the second heating 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 scan. This deviation from the standard procedure shall be recorded in the test report (see Clause 12).