BS EN ISO 11357-2:2014

BSI Standards Publication

Plastics — Differential scanning calorimetry (DSC)

Part 2: Determination of glass transition temperature and glass transition step height

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National foreword

This British Standard is the UK implementation of EN ISO 11357-2:2014. It is identical to ISO 11357-2:2013. It supersedes BS ISO 11357-2:2013, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/21, Testing of plastics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Amendments/corrigenda issued since publication

Date	Text affected
30 April 2014	This corrigendum renumbers BS ISO 11357-2:2013 as BS EN ISO 11357-2:2014

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English Version

Plastics - Differential scanning calorimetry (DSC) - Part 2: Determination of glass transition temperature and glass transition step height (ISO 11357-2:2013)

Plastiques - Analyse calorimétrique différentielle (DSC) - Partie 2: Détermination de la température de transition vitreuse et de la hauteur de palier de transition vitreuse (ISO 11357-2:2013)

Kunststoffe - Dynamische Differenz-Thermoanalyse (DSC) - Teil 2: Bestimmung der Glasübergangstemperatur und Glasübergangsstufenhöhe (ISO 11357-2:2013)

This European Standard was approved by CEN on 6 March 2014.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Foreword

The text of ISO 11357-2:2013 has been prepared by Technical Committee ISO/TC 61 “Plastics” of the International Organization for Standardization (ISO) and has been taken over as EN ISO 11357-2:2014 by Technical Committee CEN/TC 249 “Plastics” the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2014, and conflicting national standards shall be withdrawn at the latest by September 2014.

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Endorsement notice

The text of ISO 11357-2:2013 has been approved by CEN as EN ISO 11357-2:2014 without any modification.

Contents

Page

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INTERNATIONAL STANDARD **ISO 11357-2:2013(E)**

Plastics — Differential scanning calorimetry (DSC) —

Part 2: Determination of glass transition temperature and glass transition step height

WARNING — The use of this part of ISO 11357 may involve hazardous materials, operations, or equipment. This part of ISO 11357 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 11357 to establish appropriate health and safety practices and to determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 11357 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, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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 11357-1 and the following apply.

3.1
glass transition
reversible change in an amorphous polymer or in amorphous regions of a partially crystalline polymer from (or to) a viscous or rubbery condition to (or from) a hard and relatively brittle one

3.2
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.

3.3
glass transition step height
$\Delta c_p(T_g)$
difference in specific heat capacity 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 measured and the glass transition temperature and step height are determined from the curve thus obtained.

5 Apparatus and materials

The apparatus and materials are specified in ISO 11357-1.

6 Test specimens

The test specimens are specified in ISO 11357-1.

7 Test conditions and specimen conditioning

The test conditions and specimen conditioning are specified in ISO 11357-1.

8 Calibration

The calibration is specified in ISO 11357-1.

9 Procedure

9.1 Setting up the apparatus

The procedure for setting up the apparatus is specified in ISO 11357-1.

9.2 Loading the test specimen into the crucible

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

9.4 Temperature scan

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

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

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).

9.4.3 Hold the temperature for 5 min unless a shorter time is required due to sample decomposition.

9.4.4 Cool down to approximately 50 °C below the anticipated glass transition temperature using a temperature scan rate of 20 K/min.

NOTE In particular cases, e.g. if cold crystallization is to be measured, quench cooling might have to be used.

9.4.5 Hold the temperature for 5 min.

9.4.6 Perform and record a second heating cycle at a temperature scan rate of 20 K/min, heating to approximately 30 °C higher than the extrapolated end temperature ($T_{ef,g}$).

NOTE Other heating or cooling rates can be used by agreement between the interested parties. Preferably, the same scan rates are intended to be used for heating and cooling cycles. In particular, high scanning rates result in better sensitivity of the recorded transition. On the other hand, low scanning rates provide better resolution. Appropriate selection of rate is important to the observation of subtle transitions.

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

9.4.8 Reweigh the crucible with the test specimen to within ±0,1 mg.

9.4.9 If any loss of mass has occurred, a chemical change should be suspected. Open the crucible and inspect the test specimen. If the specimen has degraded, discard the test results and retest, selecting a lower maximum temperature.

Do not reuse crucibles showing signs of deterioration for another measurement.

If the test specimen overflows during measurement, clean the specimen holder assembly, following the instrument manufacturer's instructions, and verify that the calibration is still valid.

9.4.10 Requirements for repeat testing shall be indicated by the referring standards or, if none, agreed between interested parties.

10 Expression of results

10.1 Determination of glass transition temperatures

10.1.1 General

Determine the glass transition temperature using one of the methods given in 10.1.2 to 10.1.4.

The type of determination of T_g shall be included in the test report (see Clause 12).

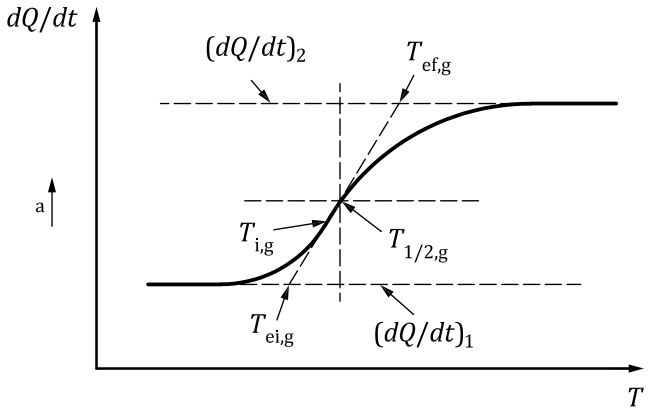
10.1.2 Half-step-height method

Assign the glass transition to the temperature, $T_{1/2,g}$, at which the measured DSC curve is intersected by a line that is equidistant between the two extrapolated baselines (see Figure 1).

10.1.3 Inflection-point method

Assign the glass transition to the temperature of inflection point, $T_{i,g}$, of the measured DSC curve in the glass transition region (see Figure 1).

The point of inflection, $T_{i,g}$, is obtained either by determining the temperature of the maximum in the derivative DSC signal or the temperature of the steepest slope in the transition zone.



Key

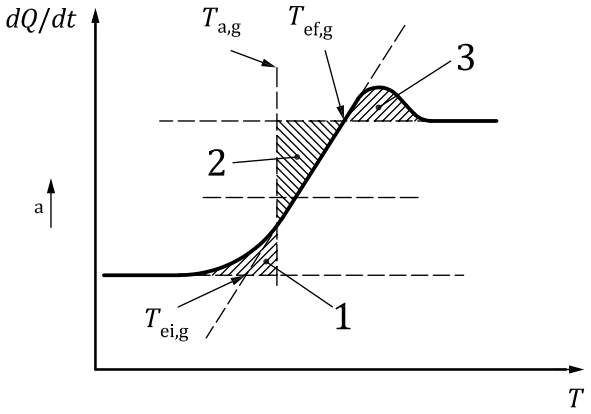
dQ/dt	heat flow rate	$T_{1/2,g}$	T_g measured by half-step-height method (10.1.2)
T	temperature	$T_{i,g}$	T_g measured by inflection-point method (10.1.3)
$(dQ/dt)_1$	heat flow rate below T_g	$T_{ei,g}$	extrapolated onset temperature of glass transition
$(dQ/dt)_2$	heat flow rate above T_g	$T_{ef,g}$	extrapolated end temperature of glass transition
a	Endothermic direction.		

Figure 1 — Examples of characteristic glass transition temperature determinations according to 10.1.2 and 10.1.3

10.1.4 Equal-areas method

Assign the glass transition to the temperature, $T_{a,g}$, obtained by drawing a vertical line such that the areas between DSC trace and baselines below and above the curve are equal, i.e. 1 + 3 = 2 (see Figure 2).[9]

NOTE As the glass transition is a kinetic phenomenon, the glass transition temperature depends on the actual used cooling rate and annealing conditions below T_g. Unperturbed glass transitions are obtained only if cooling and subsequent heating rate are the same and no significant physical ageing occurred due to annealing below T_g. If a sample is cooled significantly slower or annealed below T_g, enthalpy relaxations can occur resulting in endotherm peaks just above T_g. Peaks due to enthalpy relaxation will disappear by extrapolating to zero heating rates. The equal-areas method provides the best procedure to obtain correct glass transition temperatures in case of occurrence of enthalpy relaxations.



Key

dQ/dt	heat flow rate	$T_{a,g}$	T_g measured by equal-areas method (10.1.4)
T	temperature	$T_{ei,g}$	extrapolated onset temperature of glass transition
1, 2, 3	areas between DSC trace and baselines (see 10.1.4)	$T_{ef,g}$	extrapolated end temperature of glass transition
a	Endothermic direction.		

Figure 2 — Example of characteristic glass transition temperature determination according to 10.1.4

10.2 Determination of glass transition step height

After determining the glass transition temperature using one of the methods given in 10.1.2 to 10.1.4, extrapolate the baseline below the glass transition towards higher temperatures and the baseline above the glass transition towards lower temperatures. From the difference of heat flow rate above and below the glass transition both extrapolated to T_g, $(dQ/dt)_2$ - $(dQ/dt)_1$, the change of specific heat capacity $\Delta c_p(T_g)$ corresponding to the glass transition shall be obtained.

11 Precision

The precision of this test method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added in a revision of this part of ISO 11357.

12 Test report

The test report is specified in ISO 11357-1.

Include as the test results [item m)], the method used for determination of T_g, the characteristic glass transition temperatures $T_{ei,g}$, $T_{ef,g}$, and $T_{1/2,g}$, $T_{i,g}$, or $T_{a,g}$, as applicable, in degrees Celsius, rounded to the nearest whole number, and, if applicable, the glass transition temperature step height $\Delta c_p(T_g)$, in watts or milliwatts, rounded to the nearest two significant digits.

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