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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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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).

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 preferred scan rate of 20 K/min.

NOTE In particular cases, for example 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 preferred scan rate of 20 K/min, heating to approximately 30 K higher than the extrapolated end temperature ($T_{ef,g}$).

If melt transitions are to be evaluated, too, heating shall be done up to 30 K higher than the extrapolated end melting temperature.

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.

NOTE Usually, a scan rate of 20 K/min is applied which generally results in the best compromise between accuracy of glass transition temperature and sufficiently high glass transition step height.

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.

Preferably, new crucibles shall be used for each new specimen. Re-use of crucibles shall be limited to exceptional cases and mentioned in the test report including justification.

Do not re-use 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.

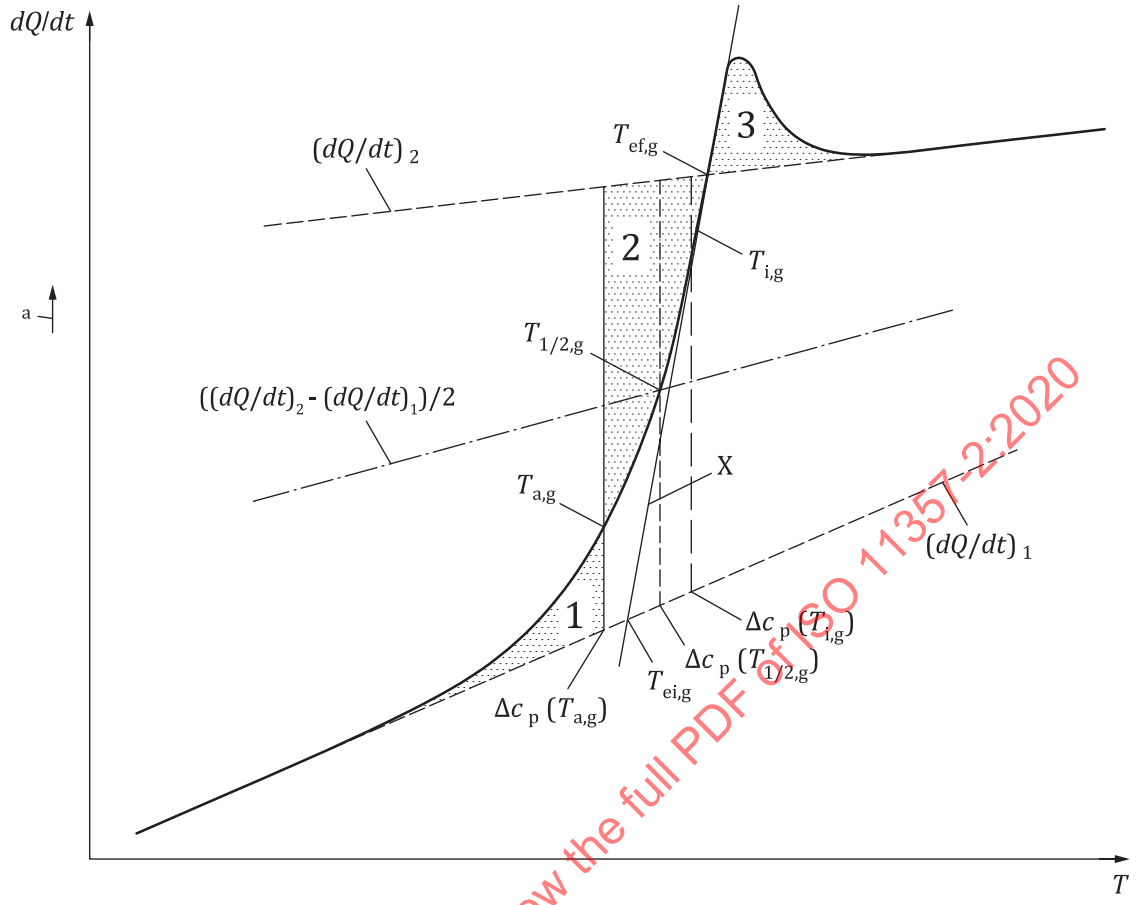
The type of determination of T_g shall be included in the test report (see Clause 12).

Preferably, the equal-areas method (see 10.1.2) shall be used for the determination of T_g. The half-step-height method (see 10.1.3) and the inflection point method (see 10.1.4) may result in significant

deviations of T_g for asymmetric glass transition step profiles, in particular for samples showing enthalpy relaxations (see Figure 1).

10.1.2 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 for samples with enthalpy relaxation (see Figure 1) or 1 = 2 for samples without enthalpy relaxation (see Figure 2)[8].



Key

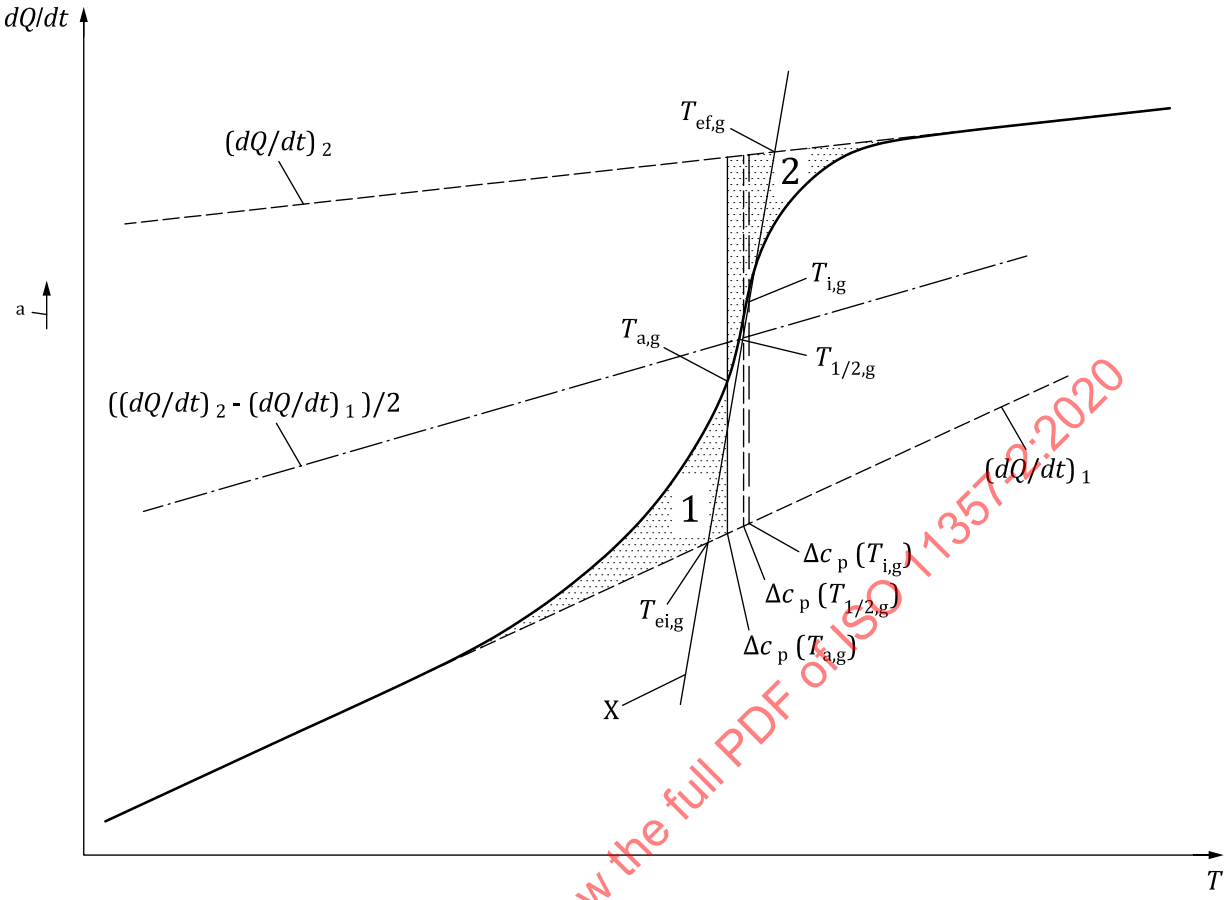
dQ/dt	heat flow rate	$T_{ei,g}$	extrapolated onset temperature of glass transition
T	temperature	$T_{ef,g}$	extrapolated end temperature of glass transition
1, 2, 3	areas between DSC trace and baselines (see 10.1.2)	$T_{a,g}$	T_g measured by equal-areas method (10.1.2)
$(dQ/dt)_1$	extrapolated heat flow rate below T_g	$T_{1/2,g}$	T_g measured by half-step-height method (10.1.3)
$(dQ/dt)_2$	extrapolated heat flow rate above T_g	$T_{i,g}$	T_g measured by inflection-point method (10.1.4)
$\frac{(dQ/dt)_2 - (dQ/dt)_1}{2}$	equidistant line between extrapolated heat flow rates above and below T_g		
X	line of steepest slope	$\Delta c_p(T_{a,g})$	glass transition step height measured by equal-areas method (10.1.2)
a	Endothermic direction.	$\Delta c_p(T_{1/2,g})$	glass transition step height measured by half-step-height method (10.1.2)
		$\Delta c_p(T_{i,g})$	glass transition step height measured by inflection-point method (10.1.2)

Figure 1 — Example of characteristic glass transition temperature determination for a sample with enthalpy relaxation

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.

10.1.3 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 Figures 1 and 2).



Key

dQ/dt	heat flow rate	$T_{ei,g}$	extrapolated onset temperature of glass transition
T	temperature	$T_{ef,g}$	extrapolated end temperature of glass transition
1, 2	areas between DSC trace and baselines (see 10.1.2)	$T_{a,g}$	T_g measured by equal-areas method (10.1.2)
$(dQ/dt)_1$	extrapolated heat flow rate below T_g	$T_{1/2,g}$	T_g measured by half-step-height method (10.1.3)
$(dQ/dt)_2$	extrapolated heat flow rate above T_g	$T_{i,g}$	T_g measured by inflection-point method (10.1.4)
$\frac{(dQ/dt)_2 - (dQ/dt)_1}{2}$	equidistant line between extrapolated heat flow rates above and below T_g		
X	line of steepest slope	$\Delta c_p(T_{a,g})$	glass transition step height measured by equal-areas method (10.1.2)
a	Endothermic direction.	$\Delta c_p(T_{1/2,g})$	glass transition step height measured by half-step-height method (10.1.3)
		$\Delta c_p(T_{i,g})$	glass transition step height measured by inflection-point method (10.1.2)

Figure 2 — Examples of characteristic glass transition temperature determinations for a sample without enthalpy relaxation

10.1.4 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 Figures 1 and 2).