

**SLOVENSKI STANDARD**  
**oSIST prEN ISO 11357-2:2019**  
**01-april-2019**

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**Polimerni materiali - Diferenčna dinamična kalorimetrija (DSC) - 2. del:  
Ugotavljanje točke posteklenitve in višine prehoda (ISO/DIS 11357-2:2019)**

Plastics - Differential scanning calorimetry (DSC) - Part 2: Determination of glass transition temperature and step height (ISO/DIS 11357-2:2019)

Kunststoffe - Dynamische Differenz-Thermoanalyse (DSC) - Teil 2: Bestimmung der Glasübergangstemperatur und der Glasübergangsstufenhöhe (ISO/DIS 11357-2:2019)

Plastiques - Analyse calorimétrique différentielle (DSC) - Partie 2: Détermination de la température et de la hauteur de palier de transition vitreuse (ISO/DIS 11357-2:2019)

**Ta slovenski standard je istoveten z: prEN ISO 11357-2**

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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 de transition vitreuse et d'hauteur d'étage*

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## ISO/DIS 11357-2:2019(E)

### 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](http://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](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

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.

# 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:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

### 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.

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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  $\pm 0,1$  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](#)).

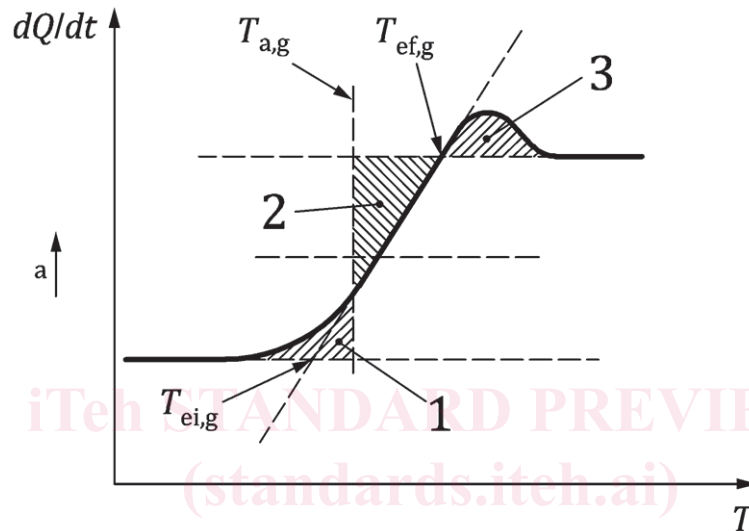
Preferably, the equal-areas method ([10.1.2](#)) shall be used for the determination of  $T_g$ . The half-step-height method ([10.1.3](#)) and the inflection point method ([10.1.4](#)) may result in significant deviations of  $T_g$  for asymmetric glass transition step profiles.

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## 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.  $1 + 3 = 2$  (see [Figure 1](#)).<sup>[7]</sup>

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 ( <a href="#">10.1.2</a> )
$T$	temperature	$T_{ei,g}$	extrapolated onset temperature of glass transition
1, 2, 3	areas between DSC trace and baselines (see <a href="#">10.1.2</a> )	$T_{ef,g}$	extrapolated end temperature of glass transition
a	Endothermic direction.		

**Figure 1 — Example of characteristic glass transition temperature determination according to [10.1.2](#)**

## 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 [Figure 2](#)).

## 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 [Figure 2](#)).

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.