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Plastics — Differential scanning calorimetry (DSC) —

Part 2:
Determination of glass transition temperature

Plastiques — Analyse calorimétrique différentielle (DSC) —

Partie 2: Détermination de la température de transition vitreuse

Reference number
ISO 11357-2:1999(E)

Contents

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11357-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

ISO 11357 consists of the following parts, under the general title *Plastics — Differential scanning calorimetry (DSC)*:

— *Part 1: General principles*

— *Part 2: Determination of glass transition temperature*

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

— *Part 4: Determination of specific heat capacity*

— *Part 5: Determination of reaction temperatures, reaction times, heats of reaction and degrees of conversion*

— *Part 6: Determination of oxidation induction time*

— *Part 7: Determination of crystallization kinetics*

— *Part 8: Determination of amount of water absorbed by polymers*

Plastics — Differential scanning calorimetry (DSC) —

Part 2
Determination of glass transition temperature

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 a method for the determination of the characteristic glass transition temperatures of amorphous and semi-crystalline plastics.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 11357. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 11357 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing*.

ISO 472:—[1)], *Plastics — Vocabulary*.

ISO 11357-1:1997, *Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles*.

3 Definitions

For the purposes of this part of ISO 11357, the definitions given in ISO 11357-1 apply, plus the following:

3.1
glass transition
the 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
the approximate midpoint of the temperature range over which the glass transition takes place

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

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1) To be published. (Revision of ISO 472:1988)

3.3 Characteristic glass transition temperatures

3.3.1
extrapolated onset temperature
T_{eig}
the point at which the extrapolated initial baseline on the low-temperature side of the curve is intersected by the tangent to the curve at the point of inflection

3.3.2
extrapolated end temperature
T_{efg}
the point at which the extrapolated initial baseline on the high-temperature side of the curve is intersected by the tangent to the curve at the point of inflection

3.3.3
midpoint temperature
T_{mg}
the point at which the curve is intersected by a line that is equidistant between the two extrapolated baselines

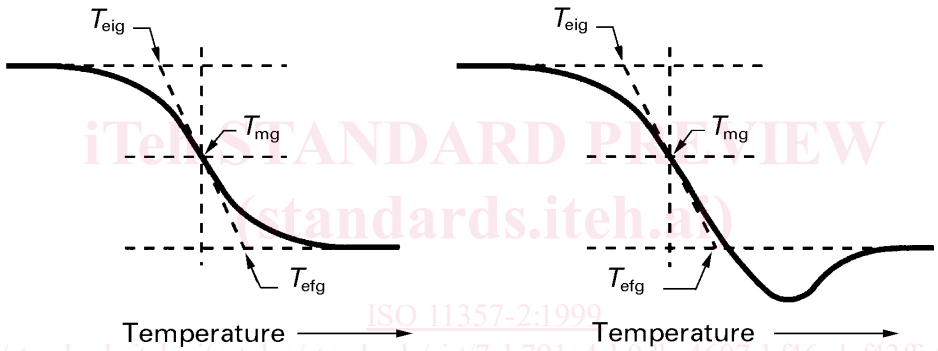

Figure 1 — Examples of characteristic glass transition temperature determinations

4 Principle

See ISO 11357-1:1997, clause 4.

The change in specific heat capacity as a function of temperature is measured and the characteristic glass transition temperatures determined from the curve thus obtained.

5 Apparatus and materials

See ISO 11357-1:1997, clause 5.

6 Test specimens

See ISO 11357-1:1997, clause 6.

7 Test conditions and specimen conditioning

See ISO 11357-1:1997, clause 7.

8 Calibration

See ISO 11357-1:1997, clause 8.

9 Procedure

9.1 Setting up the apparatus

See ISO 11357-1:1997, subclause 9.1.

Use the same purge gas flow rate that was used to calibrate the instrument. Any change in flow rate or gas requires re-calibration. Typically, nitrogen (analytical grade) at a flow rate of 50 ml/min ± 10 % is used. Other inert gases and flow rates may be used by agreement between the interested parties.

Adjust the sensitivity so that the difference in vertical height across the transition zone (step) in the curve is at least 10 % of the full-scale reading of the recorder (modern instruments do not need this adjustment).

9.2 Loading the test specimen into the pan

See ISO 11357-1:1997, subclause 9.2.

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

Ensure that the bottoms of the pans are flat. Good contact between the pans and the specimen holders is crucial to obtaining good data.

Do not handle the test material or pan with bare hands; either use tweezers or wear gloves.

9.3 Insertion of pans

See ISO 11357-1:1997, subclause 9.3.

9.4 Temperature scan

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

9.4.2 Perform and record a preliminary thermal cycle at a rate of 20 °C/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. It is important that the preliminary heat cycle is performed and that the measurements are taken from the second heat scan (see annex B of 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.

9.4.3 Hold the temperature for 5 min.

9.4.4 Quench cool to approximately 50 °C below the anticipated glass transition temperature.

9.4.5 Hold the temperature for 5 min.

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

NOTE Other heating or cooling rates 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. Appropriate selection of rate is important to the observation of subtle transitions.

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

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

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

Do not reuse pans 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 confirm that the calibration is still valid using at least one temperature and enthalpy reference standard.

9.4.10 Process the data in accordance with the instrument manufacturer's instructions.

9.4.11 Repeat testing shall be decided by the user.

10 Expression of results

Determine the transition temperatures as shown in figure 1. Often, the two baselines are not parallel. In such cases, T_{mg} is the point of intersection of the median line between the two extrapolated baselines with the curve.

The point of inflection itself can also be determined as a characteristic glass transition temperature (T_g). It is obtained either by determining the maximum in the derivative DSC signal or by measuring the steepest slope in the transition zone.

For curves showing an overshoot at the end of the transition (see the right-hand curve in figure 1), the temperature determination is identical.

11 Precision

The precision of this test method is not known because inter-laboratory data are not available. When inter-laboratory data are obtained, a precision statement will be added at the following revision.

12 Test report

See ISO 11357-1:1997, clause 10.

Include as the test results [item l)], the characteristic glass transition temperatures T_{eig}, T_{efg} and T_{mg}, in °C, rounded to the nearest whole number.

The glass transition temperature T_g corresponds to T_{mg}, but for most applications T_{eig} is more meaningful and it is often referred to as T_g. It is important when stating glass transition values that all the values T_{eig}, T_{efg} and T_{mg} are reported.

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