



SLOVENSKI STANDARD
SIST EN ISO 11357-1:1999
01-maj-1999

Polimerni materiali – Diferenčna dinamična kalorimetrija (DSC) - 1. del: Splošna načela (ISO 11357:1997)

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

Kunststoffe - Dynamische Differenz-Thermoanalyse (DSC) - Teil 1: Allgemeine Grundlagen (ISO 11357-1:1997)

Plastiques - Analyse calorimétrique différentielle (DSC) - Partie 1: Principes généraux (ISO 11357-1:1997)

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EUROPEAN STANDARD

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English version

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

Plastiques - Analyse calorimétrique différentielle (DSC) - Partie 1: Principes généraux (ISO 11357-1:1997) Kunststoffe - Dynamische Differenz-Thermoanalyse (DSC) - Teil 1: Allgemeine Grundlagen (ISO 11357-1:1997)

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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EN ISO 11357-1:1997

Foreword

The text of the International Standard ISO 11357-1:1997 has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

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of an identical text or by endorsement, at the latest by October 1997, and conflicting national standards shall be withdrawn at the latest by October 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 11357-1:1997 was approved by CEN as a European Standard without any modification.

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INTERNATIONAL
STANDARD

ISO
11357-1

First edition
1997-04-15

**Plastics — Differential scanning
calorimetry (DSC) —**

**Part 1:
General principles**

Plastiques — Analyse calorimétrique différentielle (DSC) —

Partie 1: Principes généraux

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Reference number
ISO 11357-1:1997(E)

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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 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 polymerization temperatures and/or times and polymerization kinetics*
- *Part 6: Determination of oxidation induction time*
- *Part 7: Determination of crystallization kinetics*

Annexes A, B and C of this part of ISO 11357 are for information only.

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Plastics — Differential scanning calorimetry (DSC) —

Part 1:

General principles

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

1 Scope

This International Standard specifies a method for the thermal analysis of polymers such as thermoplastics and thermosetting plastics, including moulding materials and composite materials, using differential scanning calorimetry (DSC).

Various determinations can be made on polymers by using differential scanning calorimetry. These determinations are dealt with in parts 2 to 7 of this standard (see the foreword).

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 11357. At the time of publication, the edition indicated was 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 edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291:—1), *Plastics — Standard atmospheres for conditioning and testing*.

3 Definitions

For the purposes of this International Standard, the following definitions apply:

3.1 differential scanning calorimetry (DSC): A technique in which the difference between the heat flux (power) into a test specimen and that into a reference specimen is measured as a function of temperature and/or time while the test specimen and the reference specimen are subjected to a controlled temperature programme.

It is common practice to record, for each measurement, a curve in which temperature or time is plotted on the x -axis and heat flux difference is plotted on the y -axis.

1) To be published. (Revision of ISO 291:1977)

3.2 reference specimen: A known specimen which is usually thermally inactive over the temperature and time range of interest.

NOTE — Generally an empty pan identical to the one containing the test specimen is used as the reference specimen.

3.3 standard reference material: A material for which one or more of the thermal properties are sufficiently homogeneous and well established to be used for the calibration of DSC apparatus, for the assessment of a measurement method or for assigning values to materials.

3.4 heat flux; thermal power: The amount of heat transferred per unit time (dQ/dt).

NOTE — The total quantity of heat transferred Q corresponds to the time integral of the heat flux

$$Q = \int \frac{dQ}{dt} dt$$

where Q is expressed in joules or in joules per unit mass ($J \cdot kg^{-1}$ or $J \cdot g^{-1}$).

3.5 change in enthalpy, ΔH : The quantity of heat absorbed (ΔH positive) or released (ΔH negative) by a test specimen undergoing a chemical or physical change, and/or a temperature change, at constant pressure. ΔH is expressed in joules or in joules per unit mass ($J \cdot kg^{-1}$ or $J \cdot g^{-1}$):

$$\Delta H = \int_{T_1}^{T_2} \frac{dH}{dT} dT$$

3.6 specific heat capacity at constant pressure, c_p : The quantity of heat necessary to raise the temperature of unit mass of material by 1 °C at constant pressure, with all other intensive parameters constant:

$$c_p = \frac{1}{m} \times \left(\frac{\partial Q}{\partial T} \right)_p$$

where

∂Q is the quantity of heat, expressed in joules, necessary to raise the temperature of material of mass m by ∂T degrees Celsius at constant pressure;

c_p is expressed in joules per kilogram degree Celsius [$J/(kg \cdot ^\circ C)$] or joules per gram degree Celsius [$J/(g \cdot ^\circ C)$].

When analysing polymers, care must be taken to ensure that the measured specific heat capacity does not include any heat change due to a chemical reaction or a physical transition.

3.7 baseline: The part of the recorded curve outside, but adjacent to, the reaction or transition zone. In this part of the recorded curve, the heat flux difference is approximately constant.

3.8 virtual baseline: An imaginary line drawn through a reaction and/or transition zone assuming that the heat of reaction and/or transition is zero. It is commonly drawn by interpolating or extrapolating the recorded baseline. It is normally indicated on the DSC curve for convenience (see figure 1).

3.9 peak: The part of the DSC curve which departs from the baseline, reaches a maximum, and subsequently returns to the baseline.

NOTE — The start of the peak corresponds to the start of the reaction or transition.

3.9.1 endothermic peak: A peak in which the energy supplied to the test specimen is greater than the energy corresponding to the virtual baseline.

3.9.2 exothermic peak: A peak in which the energy supplied to the test specimen is less than the energy corresponding to the virtual baseline.

NOTE — In accordance with the accepted conventions of thermodynamics, the enthalpy change is negative when the reaction or transition is exothermic and positive when the reaction or transition is endothermic. The direction corresponding to exothermic or endothermic is normally indicated on the DSC curve.

3.9.3 peak height: The distance between the virtual baseline and the point of maximum height of a peak. The height is expressed in milliwatts. The height is not proportional to the mass of the test specimen.

3.10 characteristic temperatures: The following are the characteristic temperature on a DSC curve:

— onset temperature	T_i
— extrapolated onset temperature	T_{ei}
— peak temperature	T_p
— extrapolated end temperature	T_{ef}
— end temperature	T_f

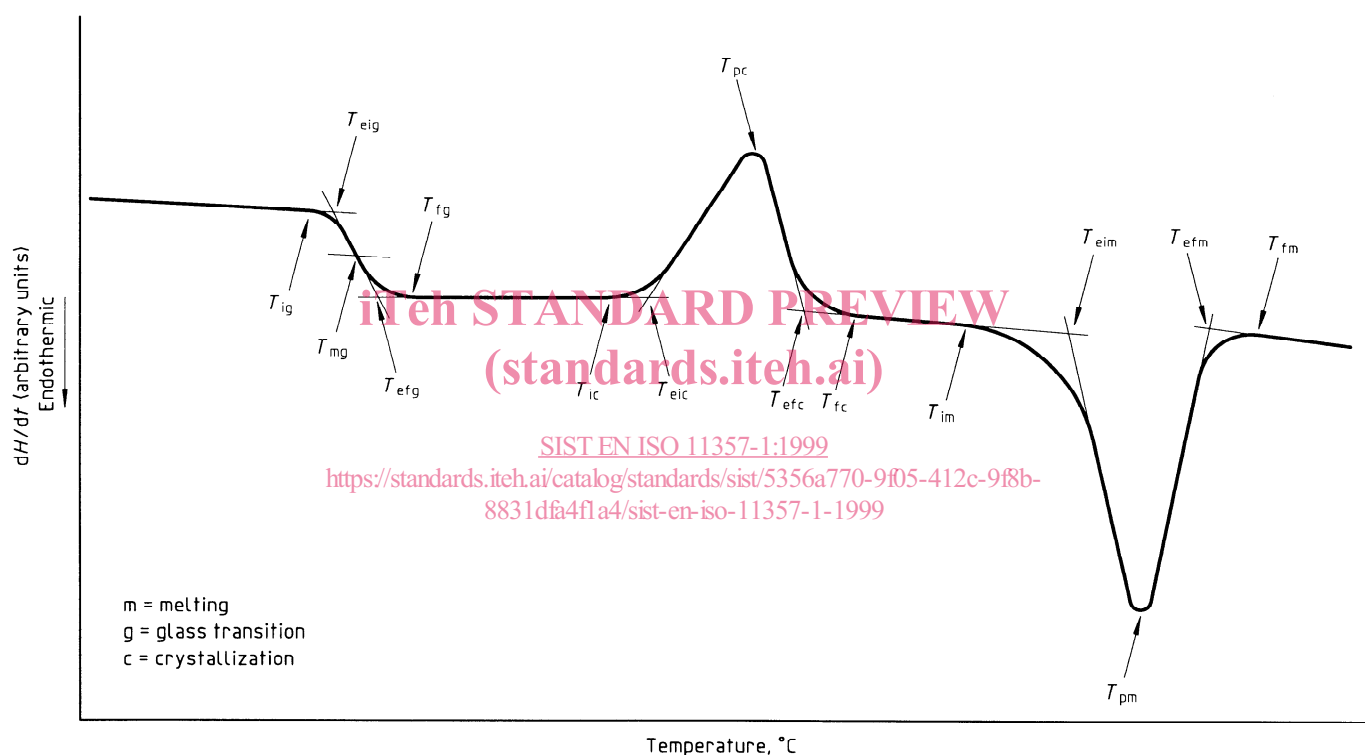


Figure 1 — Typical DSC curve

4 Principle

The difference between the heat flux into a test specimen and that into a reference specimen is measured as a function of temperature and/or time, while the test specimen and the reference specimen are subjected to a controlled temperature programme under a specified atmosphere.

NOTE — Two types of DSC, power-compensation DSC and heat-flux DSC, may be carried out. They are distinguished by the design of instrumentation used for measurement, as follows:

- Power-compensation DSC: The difference between the heat flux into the test specimen and that into the reference specimen is measured as a function of temperature or time while varying the temperature of the specimens in accordance with a controlled programme, keeping the temperature of both specimens equal.
- Heat-flux DSC: The difference in heat flux derived from the temperature difference between a test specimen and a reference specimen is measured as a function of temperature or time while varying the temperature of the specimens in accordance with a controlled programme. In this type of measurement, the difference in temperature between the test specimen and reference specimen is proportional to the difference in heat flux.