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Plastics — Differential scanning calorimetry (DSC) —

Part 1: General principles

Plastiques — Analyse calorimétrique différentielle (DSC) —

Partie 1: Principes généraux

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Foreword

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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).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*

This third edition cancels and replaces the second edition (ISO 11357-1:2009), 3.7.2 of which has been technically revised.

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 and glass transition step height*

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

— *Part 4: Determination of specific heat capacity*

— *Part 5: Determination of characteristic reaction-curve temperatures and times, enthalpy of reaction and degree of conversion*

— *Part 6: Determination of oxidation induction time (isothermal OIT) and oxidation induction temperature (dynamic OIT)*

— *Part 7: Determination of crystallization kinetics*

Introduction

ISO 11357 describes thermoanalytical DSC test methods which can be used for quality assurance purposes, for routine checks of raw materials and finished products or for the determination of comparable data needed for data sheets or databases. The procedures given in ISO 11357 apply as long as product standards or standards describing special atmospheres for conditioning of specimens do not specify otherwise.

Plastics — Differential scanning calorimetry (DSC) —

Part 1: General principles

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory requirements.

1 Scope

ISO 11357 specifies several differential scanning calorimetry (DSC) methods for the thermal analysis of polymers and polymer blends, such as

— thermoplastics (polymers, moulding compounds and other moulding materials, with or without fillers, fibres or reinforcements),

— thermosets (uncured or cured materials, with or without fillers, fibres or reinforcements), and

— elastomers (with or without fillers, fibres or reinforcements).

ISO 11357 is intended for the observation and measurement of various properties of, and phenomena associated with, the above-mentioned materials, such as

— physical transitions (glass transition, phase transitions such as melting and crystallization, polymorphic transitions, etc.),

— chemical reactions (polymerization, crosslinking and curing of elastomers and thermosets, etc.),

— the stability to oxidation, and

— the heat capacity.

This part of ISO 11357 specifies a number of general aspects of differential scanning calorimetry, such as the principle and the apparatus, sampling, calibration and general aspects of the procedure and test report common to all following parts.

Details on performing specific methods are given in subsequent parts of ISO 11357 (see Foreword).

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 472, *Plastics — Vocabulary*

ISO 80000-5, *Quantities and units — Part 5: Thermodynamics*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and ISO 80000-5 and the following apply.

3.1
differential scanning calorimetry
DSC
technique in which the difference between the rate of flow of heat into a specimen crucible containing the specimen and that into a *reference crucible* (3.3) is derived as a function of temperature and/or time while the specimen and reference are subjected to the same controlled temperature programme in a specified atmosphere using a symmetrical measurement system

Note 1 to entry: It is common practice to record, for each measurement run, a curve in which temperature or time is plotted as the abscissa and *heat flow rate* (3.4) difference as the ordinate. The endothermic and/or exothermic direction is indicated on the DSC curve.

Note 2 to entry: According to the principles of thermodynamics, energy absorbed by a system is considered positive while energy released is negative. This approach implies that the endothermic direction points upwards in the ordinate and the exothermic direction downwards (see Figures 1 and 2). It also has the advantage that the direction of thermal effects in plots of *heat flow rate* (3.4) and specific heat is consistent.

3.2
calibration material
material for which one or more of the thermal properties are sufficiently homogeneous and well established to be used for the calibration of a DSC instrument or for the assessment of a measurement method

3.3
reference crucible
crucible used on the reference side of the symmetrical crucible holder assembly

Note 1 to entry: Normally, the reference crucible is empty.

Note 2 to entry: In special cases, such as the measurement of highly filled or reinforced polymers or specimens having a heat capacity comparable to that of the crucible, a suitable material can be used inside the reference crucible. This reference material should be thermally inactive over the temperature and time range of interest and its heat capacity should be similar to that of the specimen. In the case of filled or reinforced products, the pure filler or reinforcement can be used, for example.

3.4
heat flow rate
quantity of heat transferred per unit time (dQ/dt)

Note 1 to entry: It is expressed in watts (W) or milliwatts (mW).

Note 2 to entry: The total quantity of heat transferred, Q, corresponds to the time integral of the heat flow rate:

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

3.5
change in heat
ΔQ
quantity of heat absorbed (endothermic, ΔQ positive) or released (exothermic, ΔQ negative) within a specified time, t, or temperature, T, range by a specimen undergoing a chemical or physical change and/or a temperature change:

$$\Delta Q = \int_{t_1}^{t_2} \frac{dQ}{dt} dt$$

or

$$\Delta Q = \frac{60}{\beta} \int_{T_1}^{T_2} \frac{dQ}{dt} dT$$

where

ΔQ is expressed in joules (J) or as a specific quantity, Δq, expressed in joules per amount of material in grams ($J \cdot g^{-1}$) or joules per amount of material in moles ($J \cdot mol^{-1}$);

β is the constant heating or cooling rate, dT/dt, expressed in kelvins per minute ($K \cdot min^{-1}$).

Note 1 to entry: If measurements are made at constant pressure, ΔQ corresponds to the change in enthalpy, ΔH.

3.6
specific heat capacity at constant pressure
c_p
quantity of heat necessary to raise the temperature of unit mass of material by 1 K at constant pressure:

$$c_p = \frac{1}{m} \times \left(\frac{dQ}{dT} \right)_p$$

or

$$c_p = \frac{1}{m} \times \frac{60}{\beta} \times \left(\frac{dQ}{dt} \right)_p$$

where

dQ is the quantity of heat, expressed in joules (J), necessary to raise the temperature of an amount of material of mass m, expressed in grams (g), by dT kelvins at constant pressure;

β is the heating rate, expressed in kelvins per minute ($K \cdot min^{-1}$);

c_p is expressed in joules per gram per kelvin ($J \cdot g^{-1} \cdot K^{-1}$).

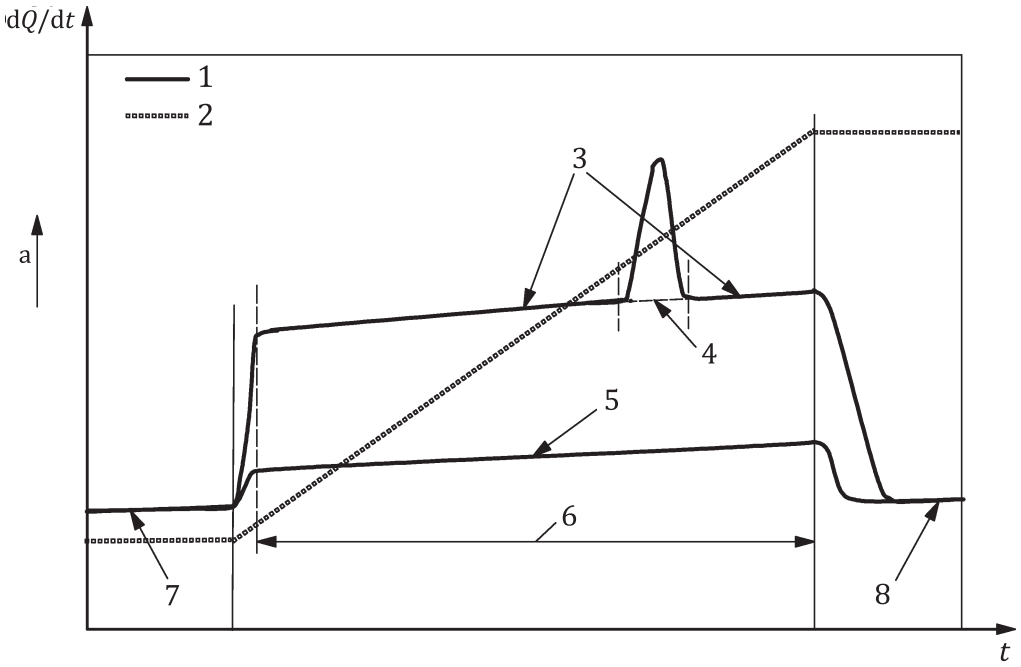
Note 1 to entry: c_p may also be expressed in joules per mole per kelvin ($J \cdot mol^{-1} \cdot K^{-1}$) when the amount of material, m, is expressed in moles.

Note 2 to entry: When analysing polymers, 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
part of the recorded curve in which no reactions or transitions take place

Note 1 to entry: This can be an isothermal baseline when the temperature is maintained constant or a dynamic baseline when the temperature is changed in accordance with a controlled temperature programme.

Note 2 to entry: The baselines defined in 3.7.1 to 3.7.3 refer to the quasi-stationary range only, i.e. when the instrument is operating under stable conditions shortly after starting and shortly before ending the DSC run (see Figure 1).



Key

dQ/dt	heat flow rate	4	virtual baseline
T	temperature	5	instrument baseline
t	time	6	quasi-stationary range
1	dQ/dt vs t (or T)	7	isothermal start baseline
2	T vs t	8	isothermal end baseline
3	specimen baselines	a	Endothermic direction.

Figure 1 — Schematic drawing showing baselines

3.7.1
instrument baseline
curve obtained using only empty crucibles of identical mass and material in the specimen and reference positions of the DSC cell

Note 1 to entry: The instrument baseline is required for heat capacity measurements.

3.7.2
specimen baseline
DSC curve obtained outside any reaction or transition zone(s) while the instrument is loaded with both the specimen in the specimen crucible and the *reference crucible* (3.3)

Note 1 to entry: In this part of the curve, the difference in *heat flow rate* (3.4) between the specimen crucible and the *reference crucible* (3.3) depends solely on the heat capacity of the specimen and the *instrument baseline* (3.7.1).

Note 2 to entry: The specimen baseline reflects the temperature dependence of the heat capacity of the specimen.

Note 3 to entry: For heat capacity determinations, a dynamic DSC curve is required and, in addition, the *instrument baseline* (3.7.1) and the isothermal start and end baselines (see Figure 1).

3.7.3
virtual baseline
imaginary line drawn through a reaction and/or transition zone assuming the heat of reaction and/or transition to be zero

Note 1 to entry: Assuming the change in heat capacity with temperature to be linear, the virtual baseline is drawn by interpolating or extrapolating the specimen baseline in a straight line. It is normally indicated on the DSC curve for convenience (see Figures 1 and 2).

Note 2 to entry: The virtual baseline drawn from peak onset, T_i, to peak end, T_f, (the peak baseline) allows the determination of the peak area from which the heat of transition can be obtained. If there is no significant change in heat capacity during the transition or reaction, the baseline can be drawn simply by connecting the peak onset and peak end by a straight line. If significant heat capacity changes occur, a sigmoidal baseline can be drawn.

Note 3 to entry: Extrapolated and interpolated virtual baselines will not necessarily coincide with each other (see Figure 2).

3.8
step
abrupt positive or negative change in the height of a DSC curve, taking place over a limited temperature range

Note 1 to entry: A step in the DSC curve can be caused by, for example, a glass transition (see Figure 2).

3.8.1
step height
difference between the heights of the extrapolated baselines before and after a step, measured at the time or temperature corresponding to the point on the DSC curve which is equidistant between the two baselines

3.9
peak
part of the DSC curve which departs from the *specimen baseline* (3.7.2), reaches a maximum or minimum, and subsequently returns to the *specimen baseline* (3.7.2)

Note 1 to entry: A peak in the DSC curve may indicate a chemical reaction or a first-order transition. The initial departure of the peak from the *virtual baseline* (3.7.3) corresponds to the start of the reaction or transition.

3.9.1
endothermic peak
peak in which the rate of flow of heat into the specimen crucible is greater than that into the *reference crucible* (3.3)

Note 1 to entry: This corresponds to a transition which absorbs heat.

3.9.2
exothermic peak
peak in which the rate of flow of heat into the specimen crucible is less than that into the *reference crucible* (3.3)

Note 1 to entry: This corresponds to a transition which releases heat.

3.9.3
peak area
area enclosed by a peak and the interpolated *virtual baseline* (3.7.3)

3.9.4
peak height
greatest distance in the ordinate direction between the interpolated *virtual baseline* (3.7.3) and the DSC curve during a peak

Note 1 to entry: The peak height, which is expressed in watts (W) or watts per gram (W/g), is not necessarily proportional to the mass of the specimen.

3.9.5
peak width
distance between the onset and end temperatures or times of a peak

3.10
characteristic temperatures, *T*, and times, *t*
values for temperature and time obtained from the DSC curve

Note 1 to entry: See Figure 2.

Note 2 to entry: For all types of DSC instrument, a distinction needs to be made between two different categories of temperature:

— the temperature at the reference position;

— the temperature at the specimen position.

The reference position temperature is the one preferred for plotting thermograms. If the specimen position temperature is used, then this information will need to be included in the test report.

Note 3 to entry: Characteristic temperatures are expressed in degrees Celsius (°C), relative temperatures and temperature differences in kelvins (K) and characteristic times in seconds (s) or minutes (min) (see Figure 2).

Note 4 to entry: The DSC curve can also be plotted using time, *t*, as the abscissa instead of temperature, *T*.