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Plastics — Differential scanning calorimetry (DSC) —

Part 4:
Determination of specific heat capacity

Plastiques — Analyse calorimétrique différentielle (DSC) —

Partie 4: Détermination de la capacité thermique massique

ICS 83.080.01

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 11357-4 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*

Annex A is for information only.

Introduction

A list of standards related to this part of ISO 11357 is given in the bibliography.

Plastics — Differential scanning calorimetry (DSC) – Part 4: Determination of specific heat capacity

1 Scope

This International Standard specifies the method for determining of specific heat capacity of plastics by differential scanning calorimetry.

2 Normative references

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

ISO 472:1999, *Plastics – Vocabulary.*

ISO11357-1, *Plastics-Differential Scanning Calorimetry (DSC) - Part 1: General principles.*

ISO31-0, *General Principles.*

3 Definitions

For the purposes of this International Standard, the terms and definitions given in ISO 11357-1 and the following apply.

3.1
Calibration material
materials whose specific heat capacity values have been established are used

NOTE Usually, α-alumina (such as synthetic sapphire) of 99.9% or higher purity is used as a calibration material.

3.2
Specific heat capacity (at constant pressure)
The quantity of heat necessary to raise the temperature of unit mass of material by 1 K at constant pressure. It is indicated by c_p, and the following equation is obtained:

$$c_p = m^{-1} \cdot C_p = m^{-1} \cdot (\mathrm{d}Q / \mathrm{d}T)_p \qquad (1)$$

where m is the mass of the material, C_p is the heat capacity and dQ is the heat quantity necessary to raise the temperature of the material by dT. Subscript p indicates an isobaric process. c_p is expressed in kilojoules per kilogram per K (kJ · kg^{-1} · K^{-1}) or in joule per gram per K (J · g^{-1} · K^{-1}).

This equation is valid in a temperature range where a material shows no first order phase transition.

$$(\mathrm{d}Q / dT) = (\mathrm{d}t / dT) \cdot (\mathrm{d}Q / \mathrm{d}t)$$
$$= (\text{heating rate})^{-1} \cdot (\text{heat flow rate}) \qquad (2)$$

4 Principle

Each measurement consists of three test at the same scanning rate runs as follow (figure 1) (Annex A[1,2]):

(1) blank run (set of empty pans in DSC holders)

(2) calibration run (calibration material and empty pan)

(3) specimen run (specimen and empty pan).

Based on the DSC principle (ISO 11357-1) and the definition of the specific heat capacity as described in 3.2, the following relations are obtained, where P is dQ / dt , specimen is a part of sample for measurement and the superscrips of sp and cal represent specimen and calibration:

$$m^{sp}\, c_p^{\,sp} \propto P_{specimen\ \ run} - P_{blank\ \ run} \qquad (3)$$

$$m^{cal}\, c_p^{\,cal} \propto P_{calibration\ \ run} - P_{blank\ \ run} \qquad (4)$$

Where P_{run} shows the DSC signal, when $P_{specimen\ \ run}$, $P_{calibration\ \ run}$ and $P_{blank\ run}$ are measured, $c_p^{\,sp}$ can be calculated using equation (6), since $c_p^{\,cal}$, m^{cal} and m^{sp} are known values

$$\frac{m^{sp}\, c_p^{\,sp}}{m^{cal}\, c_p^{\,cal}} = \frac{P_{specimen\ \ run} - P_{blank\ \ run}}{P_{calibration\ \ run} - P_{blank\ \ run}} \qquad (5)$$

$$c_p^{\,sp} = c_p^{\,cal}\, \frac{m^{cal}\,(P_{specimen\ \ run} - P_{blank\ \ run})}{m^{sp}\,(P_{calibration\ \ run} - P_{blank\ \ run})} \qquad (6)$$

NOTE Certain types of DSC can be calibrated using electrical calibration (Joule effect). In this case, it is not needed to refer to a calibration run.

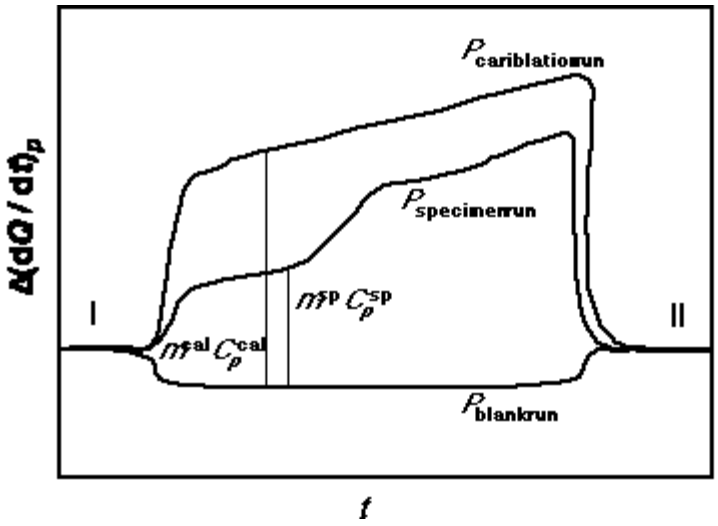

Figure 1 — Typical DSC curve on specific heat capacity measurement after baseline adjuststment

I : isothermal baseline at T_b, II : isothermal baseline at T_f

5 Apparatus

5.1 DSC apparatus

See ISO 11357-1, clause 5.1

5.2 Pan

(See ISO 11357-1, clause 5.2)

NOTE 1 Pans for the test specimen and the reference should be of the same shape, material and the mass should be equal.

NOTE 2 The same blank run and calibration run can be used for several measurements, if the instrument is sufficiently stable and the mass difference between calibration material pan and empty pan is corrected. An adequate correction can be obtained by adding the term $c_{p\ pan}(T)\ \beta\ \Delta m$ to the heat flow rate signal of the sample, where $c_{p\ pan}$ is specific heat capacity of pan as a function of temperature, β is the heating rate and Δm is the mass difference between calibration and blank pan . The same procedure can also be used for mass differences between specimen and blank run.

5.3 Analytical balance

See ISO 11357-1

6 Test specimen

See also ISO 11357-1, clause 6