BSI Standards Publication

Plastics — Temperature modulated DSC

Part 2: Measurement of specific heat capacity c_p

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National foreword

This British Standard is the UK implementation of ISO 19935-2:2020.

The UK participation in its preparation was entrusted to Technical Committee PRI/21, Testing of plastics.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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INTERNATIONAL STANDARD

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Plastics — Temperature modulated DSC —

Part 2:
Measurement of specific heat capacity c_p

Plastiques — DSC à température modulée —

Partie 2: Mesurage précis de la chaleur spécifique c_p

Contents

Page

Foreword

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

A list of all parts in the ISO 19935 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document describes the realization of standardized thermoanalytical test methods which can be used for the determination of specific heat capacity data needed for data sheets or databases as well as for research purposes. It can also be applied to quality assurance or to routine checks of raw materials and finished products, if desired. The procedures mentioned in this document apply as long as special product standards or standards describing special atmospheres for conditioning of samples do not require alternate regulations.

For scientific investigations or resolution of special analytical problems, all technical capabilities of the instruments beyond the regulations of this document may be used.

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INTERNATIONAL STANDARD

Plastics — Temperature modulated DSC —

Part 2:
Measurement of specific heat capacity

1   Scope

This document establishes a method for measurement of specific heat capacity, c_p, using temperature modulated differential scanning calorimetry.

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*

ISO 19935-1, *Plastics — Temperature modulated DSC — Part 1: General principles*

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, ISO 11357-1, ISO 19935-1 and ISO 80000-5 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4   Symbols and abbreviated terms

4.1   Temperature modulation, *T(t)*

According to ISO 19935-1.

4.2   Scanning rate

According to ISO 19935-1.

4.3   Heat flow rate, Φ(t)

According to ISO 19935-1.

b) Depending on the relation of the underlying scanning rate β_0 to the amplitude of sample temperature profile $T_A\omega$ (where ω the angular frequency) four cases of scanning rates are distinguished.

1) Quasi–isothermal mode, $\beta_0 = 0$. If $\beta_0 = 0$, heat capacity can be measured as function of time, e.g. during chemical reactions like curing or during crystallization. All other calorimetric methods measure heat capacity as function of temperature only.

2) For other modes, such as $T_A\omega > \beta_0$, $T_A\omega < \beta_0$, and $T_A\omega = \beta_0$, refer to ISO 19935-1.

c) Any mode of temperature modulation specified in ISO 19935-1 can be used.

7 Calibration

7.1 General

According to ISO 19935-1.

7.2 Calibration of modulation amplitude

According to ISO 19935-1.

7.3 Calibration of phase

According to ISO 19935-1.

8 Procedure

8.1 General

The procedures of temperature modulated DSC (setting up the apparatus, loading the specimen into the crucibles, insertion of crucibles into the instrument, performing measurements, and removal of crucibles) shall be in accordance with ISO 11357-1.

The experimental conditions specified for temperature modulated DSC (for example, amplitude of the modulated heat flow rate, amplitude of modulated temperature, and the frequency of the modulation superimposed on the underlying rate) to be used depend on the magnitude of the specific heat capacity to be determined.

3

8.2   Calculation of the specific heat capacity

Specific heat capacity is calculated as Formula (2):

$$c_p = \frac{\Phi_A}{m \cdot T_A \cdot \omega} \cdot K(\omega) \qquad (2)$$

where

c_p is the specific heat capacity of the specimen, in $Jg^{-1}K^{-1}$;

T_A is the amplitude of sample temperature profile, in K;

Φ_A is the amplitude of the periodic part of the modulated heat flow rate, in mW;

m is the mass of the specimen, in mg;

ω is the angular frequency of the modulated temperature, in rad/s; $\omega = 2\pi f$, where f is the frequency in Hz;

$K(\omega)$ is the calibration constant calculated according to the definition in ISO 19953-1. For precise measurements, ω shall be low (typically $\omega < 60$ mrad/s), so that $K(\omega)$ becomes independent on ω.

For the case of step scan mode, Formula (1) shall be used to calculate c_p.

8.3   Examples of the results

8.3.1   Modulated heat flow rate and scanning rate of modulation

Figure 1 shows a typical example of modulated temperature and scanning rate (of modulation) derived by a derivative of modulated temperature of a sinusoidal waveform for the heat flux DSC plotted vs time. The scanning rate of modulation is measured as a sinusoidal wave.

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$$c_\mathrm{p} = \frac{\Phi_A}{m \cdot T_\mathrm{A} \cdot \omega} \cdot \mathrm{K}(\omega)$$

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