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Plastics — Temperature modulated DSC —

Part 3: Separation of overlapping thermal transitions

Plastiques — DSC à température modulée —

Partie 3: Séparation des transitions thermiques se chevauchant

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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.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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 specifies a thermoanalytical test method which can be used for the separation of overlapping thermal transitions in order to obtain comparable data 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 procedure 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 can be used.

Plastics — Temperature modulated DSC —

Part 3: Separation of overlapping thermal transitions

1 Scope

This document specifies a method for the separation of overlapping thermal transitions of plastics related to reversing and non-reversing heat flow rate, 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 19935-2:2020, *Plastics — Temperature modulated DSC — Part 2: Measurement of specific heat capacity c_p*

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

4.1 Temperature modulation, *T(t)*

According to ISO 19935-1.

4.2 Heating rate

According to ISO 19935-1.

4.3 Heat flow rate, Φ(t)

According to ISO 19935-1.

5 Principles

According to ISO 19935-1.

The total heat flow rate signal is separated into a reversing and a non-reversing component by means of modulation of the heating rate. The reversing heat flow rate corresponds to the heat capacity component while the non-reversing heat flow rate corresponds to the kinetic component of the total heat flow rate.

Separation is limited to overlapping effects related to reversing (heat capacity) component and non-reversing (kinetic) component, respectively, on the time scale of modulation.

6 Apparatus and materials

The apparatus and materials shall be in accordance with ISO 19935-1.

7 Calibration

7.1 General

According to ISO 19935-1.

7.2 Calibration procedure

According to ISO 19935-2.

7.3 Calibration of modulation amplitude

According to ISO 19935-1.

7.4 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.

8.2 Experimental conditions

According to ISO 19935-2.

The determination of reversing and non-reversing specific heat capacity shall be done in accordance with ISO 19935-1.

8.3 Interpretation of results

According to ISO 19935-1.

8.4 Examples of the results — Separation of reversing and non-reversing heat flow rate

Figure 1 shows a typical example of quenched polyethylene terephthalate (PET).

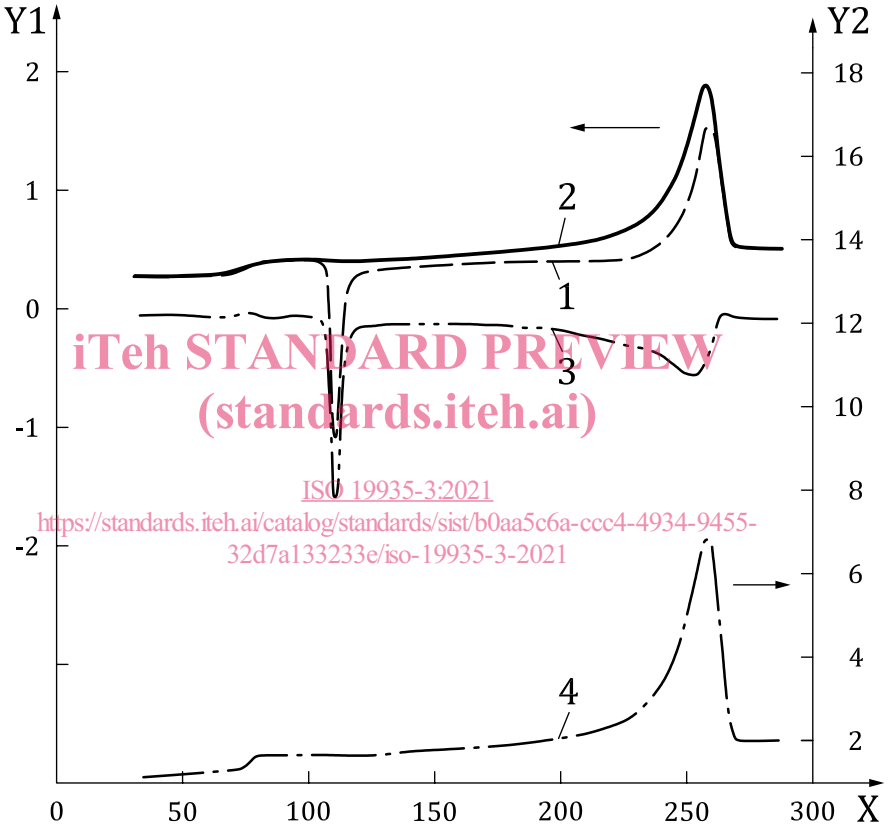
In non-transition regions, such as below glass transition and in the molten state, the reversing and the total flow rate are the same.

In the transition region, non-reversing heat flow rate detects the kinetic process, such as cold crystallization, and melting. It is also available for crystallization, enthalpy relaxation, and chemical reactions.

The specific heat capacity, determined by ISO 19935-2:2020, Formula (2), is shown in Figure 1.

The enthalpies associated with crystallization are not expressed as changes in specific heat capacity in Figure 1 as the result of separation of reversing and non-reserving heat flow rates from the total heat flow rate.

See Annex A for further examples of reversing and non-reversing heat flow rate.



Key

X temperature, *T*, expressed in °C
Y1 heat flow rate (Endo Up), expressed in mW
Y2 specific heat capacity, c_p, expressed in / $Jg^{-1}K^{-1}$
1 total heat flow rate, expressed in mW
2 reversing heat flow rate, expressed in mW
3 non-reversing heat flow rate, expressed in mW
4 Reversing specific heat capacity, expressed in $J\ g^{-1}K^{-1}$

NOTE The measurement conditions are: underlying heating rate of 2,0 K/min, with a modulation frequency 10 mHz and a modulation amplitude ±0,5 K of an input of sinusoidal waveform.

Figure 1 — Reversing and non-reversing heat flow rates separated from the total heat flow rate (Y1) and the specific heat capacity(Y2) of the quenched polyethylene terephthalate (PET) film

9 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 19935-3:2021;

b) type and complete identification of specimen;

c) type (heat flux or power compensated), manufacturer and model of DSC instrument used;

d) material, type and mass of crucible used;

e) type, purity and flow rate of purge gas used;

f) type of calibration procedure, calibration materials used, including source, mass and other properties important for calibration;

g) details of sampling, preparation of specimen and conditioning procedures, if applicable;

h) shape and dimensions of specimen, if applicable;

i) mass of specimen;

j) thermal histories of sample and specimen;

k) temperature program parameters, including time and temperature of isothermal steps and rate of dynamic steps, frequency, amplitude and waveform of the modulation;

l) change of mass of specimen over duration of test, if any;

m) curves of temperature and heat flow rate as well as total, reversing and non-reversing heat flow rate;

n) date of test;

o) any additional information or operating details not specified in this document which might be important for assessment of the results.