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## Plastics — Temperature modulated DSC —

### Part 2: Measurement of accurate specific heat $c_p$

*Plastiques — DSC à température modulée —*

*Partie 2: Mesurage précis de la chaleur spécifique  $c_p$*

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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](http://www.iso.org/directives)).

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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](http://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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# Plastics — Temperature modulated DSC —

## Part 2: Measurement of accurate specific heat $c_p$

### 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, $\Phi(t)$

According to ISO 19935-1.

## 5 Principles of determination of specific heat capacity with temperature modulated DSC

### 5.1 General

The use of temperature modulated DSC is advantageous for the measurement of specific heat capacity not only outside the region of a transition, because it does not require a stable baseline to achieve high accuracy. The measurement of specific heat capacity is only based on the amplitude of the modulated heat flow rate signal.

### 5.2 Specific heat capacity with no processes

According to ISO 19935-1.

### 5.3 Reversing and non-reversing specific heat capacity

According to ISO 19935-1.

### 5.4 Step scan method

In the step scan method, the isothermal line is called as iso-kinetic (iso-K) base line. Specific heat capacity,  $c_p$ , can be determined by integrating the heat flow rate  $\Phi(t)$  in one cycle as [Formula \(1\)](#):

$$c_p = \frac{1}{m\Delta T} \int_{t_0}^{t_e} \Phi(t) dt \quad (1)$$

where

- $m$  is the mass of the sample;
- $\Delta T$  is a temperature step;
- $t_e, t_0$  is a time interval in one period;

### 5.5 Multiple frequencies

Assuming the linear response theory, the input of multiple frequencies or random pulses as a modulated heat source are analysed with the procedures in the ISO 19935-1. An example is shown in [Annex D](#).

## 6 Apparatus and materials

### 6.1 General

Use the apparatus according to ISO 19935-1.

### 6.2 Temperature control of modulated differential scanning calorimeter

Specifications of temperature control required to temperature modulated DSC are added as follows.

- a) Capability to vary temperature periodically, or by a step, or by an input of pulse sequence with an amplitude, typically in the range of ( $\pm 0,1$  to  $2,0$ ) K, and a frequency, typically down to 10 mHz, superimposed on the underlying rate, typically less than 3 K/min.



- b) Depending on the relation of the underlying scanning rate  $\beta_0$  to the amplitude of sample temperature profile  $T_A\omega$ , where  $\omega$  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 to 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 depending on the magnitude of the specific heat capacity to be determined.

### 8.2 Calculation of the specific heat capacity

Specific heat capacity is calculated as [Formula \(2\)](#):

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

where

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

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

$\Phi_A$  is the amplitude of the periodic part of the modulated heat flow rate, in mW;

$m$  is the mass of the specimen, in mg;

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

$K(\omega)$  is the calibration constant calculated with the definition in ISO 19953-1:2018, 7.2.  
For precise measurements,  $\omega$  shall be low (typically  $\omega < 60$  mrad/s), so that  $K(\omega)$  becomes independent on  $\omega$ .

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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