
Polimerni materiali - Ugotavljanje temperature ali območja taljenja kristaliničnih polimerov s kapilarno cevko in polarizacijskim mikroskopom (ISO/DIS 3146:2021)

Plastics - Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers by capillary tube and polarizing-microscope methods (ISO/DIS 3146:2021)

Kunststoffe - Bestimmung des Schmelzverhaltens (Schmelztemperatur oder Schmelzbereich) von teilkristallinen Polymeren im Kapillarrohr- und Polarisationsmikroskop-Verfahren (ISO/DIS 3146:2021)

Plastiques - Détermination du comportement à la fusion (température de fusion ou plage de températures de fusion) des polymères semi-cristallins par méthodes du tube capillaire et du microscope polarisant (ISO/DIS 3146:2021)

Ta slovenski standard je istoveten z: prEN ISO 3146

ICS:

83.080.01	Polimerni materiali na splošno	Plastics in general
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Plastiques — Détermination du comportement à la fusion (température de fusion ou plage de températures de fusion) des polymères semi-cristallins par méthodes du tube capillaire et du microscope polarisant

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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).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 3146:2000), which has been technically revised.

The main changes compared to the previous edition are as follows:

- a) the specifications of the apparatus and measurement procedure have been revised;
- b) the technical corrigendum 1 dated 2002 has been incorporated;
- c) the table of calibration standards has been revised by adding further entries, synonyms and CAS numbers of organic chemicals, correcting errors and modifying melting temperatures according to added references.

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

The melting behaviour of a crystalline or semi-crystalline polymer is a structure-sensitive property.

In polymers, a sharp melting point, such as is observed for low molecular mass substances, usually does not occur; instead a melting temperature range is observed on heating, from the first change of shape of the solid particles to the transformation into a highly viscous or viscoelastic liquid, with accompanying disappearance of the crystalline phase. The melting range depends upon a number of parameters, such as molecular mass, molecular mass distribution, per cent crystallinity, and thermodynamic properties.

The melting range can also depend on experimental parameters such as previous thermal history of the specimen, heating or cooling rate, etc. The lower or upper limit of the melting range, or its average value, is sometimes conventionally referred to as the “melting temperature”.

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Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers by capillary tube and polarizing-microscope methods

1 Scope

This document specifies two methods for evaluating the melting behaviour of semi-crystalline polymers.

Melting temperatures determined by the different methods usually differ by several kelvins for the reasons explained in the introduction.

Method A: Capillary tube

This method is based on the changes in shape of the polymer. It is applicable to all semi-crystalline polymers and their compounds.

NOTE 1 Method A can also be useful for the evaluation of the softening of non-crystalline solids.

Method B: Polarizing microscope

This method is based on changes in the optical properties of the polymer. It is applicable to polymers containing a birefringent crystalline phase. It may not be suitable for plastics compounds containing pigments and/or other additives which could interfere with the birefringence of the polymeric crystalline zone.

NOTE 2 Another method applicable to semi-crystalline polymers is described in ISO 11357-3, *Plastics — Differential scanning calorimetry (DSC) — Part 3: Determination of temperature and enthalpy of melting and crystallization*.

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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following 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/>

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3.1 melting range
the temperature range over which crystalline or semi-crystalline polymers lose their crystalline characteristics or particulate shape when heated

Note 1 to entry: The “melting temperatures” determined by methods A and B are defined in 4.1 and 5.1.

4 Method A — Capillary tube

4.1 Principle

A test sample is heated at a controlled rate, and the temperature of the sample measured at the first visible change in shape and again at the disappearance of the last remnants of the crystalline phase.

The first temperature is taken as the melting temperature of the sample, while the range between the two temperatures describes its melting range.

NOTE This method can be used for non-crystalline materials where required by the relevant specifications or by agreement between the interested parties.

4.2 Apparatus (see Figure 1)

4.2.1 Melting apparatus, consisting of the following items:

- a) Cylindrical metal block, the upper part of which is hollow and forms a chamber.
- b) Metal plug, with two or more holes, allowing a thermometer and one or more capillary tubes to be mounted in the metal block.
- c) Heating system for the metal block, for example an electrical resistance enclosed in the block.
- d) Rheostat for regulation of the power input, if electrical heating is used.
- e) Four windows of heat-resistant glass on the lateral walls of the chamber, diametrically disposed at right angles to each other. In front of one of these windows an eyepiece is mounted for observing the capillary tube. The other three windows are used for illuminating the inside of the enclosure by means of lamps.

NOTE Other suitable types of melting apparatus can be used, provided that they give the same results.

4.2.2 Capillary tube, of heat-resistant glass, closed at one end.

The maximum external diameter should preferably be 1,8 mm.

4.2.3 Calibrated thermometer, graduated in divisions of 0,1 °C. The thermometer bulb shall be positioned in such a way that heat dispersion in the apparatus is not impeded.

NOTE Other suitable temperature-measuring devices can be used.

4.3 Test samples

4.3.1 General

The test samples used shall be representative of the material to be tested.