# SLOVENSKI STANDARD
# SIST ISO 3146:1996

**01-junij-1996**

---

**Polimerni materiali - Določanje temperature ali območja taljenja kristaliničnih polimerov**

Plastics -- Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers



Plastiques -- Détermination du comportement à la fusion (température de fusion ou plage de température de fusion) des polymères semi-cristallins



**Ta slovenski standard je istoveten z:** **ISO 3146:1985**

---

**ICS:**

| | | |
|---|---|---|
| 83.080.01 | Polimerni materiali na splošno | Plastics in general |

**SIST ISO 3146:1996** **en**

# International Standard 3146

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

# Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers

*Plastiques — Détermination du comportement à la fusion (température de fusion ou plage de température de fusion) des polymères semi-cristallins*



**Second edition — 1985-12-15**



**UDC 678.7 : 620.1 : 536.421.1** **Ref. No. ISO 3146-1985 (E)**

**Descriptors :** plastics, polymers, tests, determination, melting points, test equipment.



Price based on 10 pages

## 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.



International Standard ISO 3146 was prepared by Technical Committee ISO/TC 61, *Plastics*.



This second edition cancels and replaces the first edition (ISO 3146-1974), of which it constitutes a minor revision.



Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.



Printed in Switzerland

# Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers



## 0 Introduction

The melting behaviour of a crystalline or partly 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, if present. The melting range depends upon a number of parameters, such as molecular mass, molecular mass distribution, per cent crystallinity, and thermodynamic properties.

It may also depend on the previous thermal history of the specimens. The lower or upper limit of the melting range, or its average value, is sometimes conventionally referred to as the "melting temperature".

## 1 Scope and field of application

This International Standard specifies three methods for evaluating the melting behaviour of semi-crystalline polymers.

**Section one** specifies a capillary tube method (method A), which is based on the changes in shape of the polymer. This method is applicable to all polymers and their compounds, even if there is no crystalline phase.

**Section two** specifies a polarizing microscope method (method B), which is based on changes in the optical properties of the polymer. This method 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.

**Section three** specifies a thermal analytical method (method C), having two variants :

— method C1, which uses Differential Thermal Analysis (DTA);

— method C2, which uses Differential Scanning Calorimetry (DSC).

Both are applicable to all polymers containing a crystalline phase and their compounds.

The melting temperatures determined by the different methods usually differ by several kelvins for the reasons explained in the Introduction.

Of the methods given above, experiments have indicated DSC (Differential Scanning Calorimetry) to be the method of choice as having the best reproducibility of results.

## 2 Definitions

**2.1 semi-crystalline polymers** : Polymers containing a crystalline phase surrounded by amorphous materials.

**2.2 melting range** : The temperature range over which crystalline polymers lose their crystallinity when heated.

NOTE — The conventional "melting temperatures" determined by methods A and B are defined in clauses 3 and 8.

# Section one : Method A — Capillary tube

## 3 Principle

Heating of a specimen, at a controlled rate, and observation for change in shape.

Reporting of the temperature of the specimen at the first visible deformation as the melting temperature.

NOTE — This method may also be used for non-crystalline materials according to the relevant specifications or by agreement between the interested parties.

## 4 Apparatus (see figure 1)

**4.1 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 into the metal block a);

c) heating system for the metal block a) provided, for example, by 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 is mounted an eyepiece 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 melting apparatuses may be used, provided that they give the same results.

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

NOTE — The maximum external diameter should preferably be 1,5 mm.

**4.3 Calibrated thermometer**, graduated in divisions of 1 K. The thermometer probe shall be positioned in such a way that heat dispersion in the apparatus is not impeded.



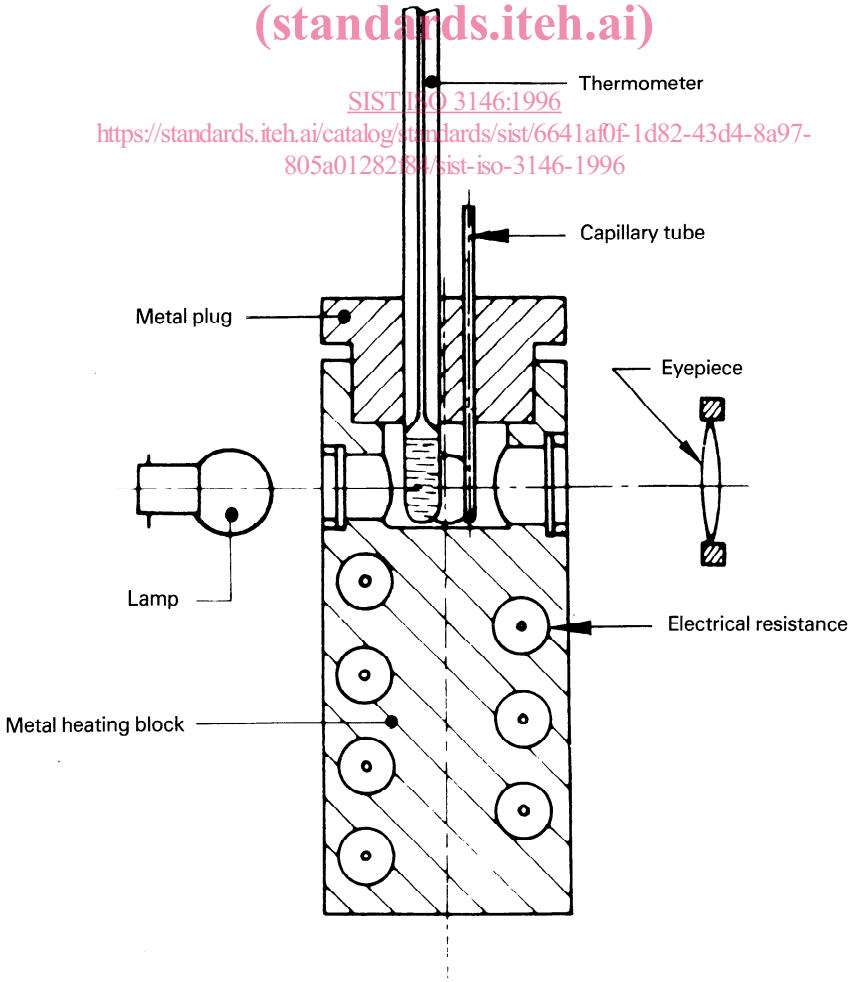


**Figure 1 — Apparatus for method A**

NOTE — Other suitable temperature-measuring devices may be used.

## 5 Test specimens

The specimens used shall be representative of the sample of material to be tested.

### 5.1 Characteristics

Powder of particle size up to 100 µm or cut pieces of films of thickness 10 to 20 µm should preferably be used. Comparison tests shall be carried out on specimens of the same or similar particle size, or similar thickness in the case of layers or films.

### 5.2 Conditioning

If not otherwise specified or agreed to by the interested parties, the sample shall be conditioned at 23 ± 2 °C and relative humidity of 50 ± 5 % for 3 h prior to the measurement.

## 6 Procedure

### 6.1 Calibration

Calibrate the temperature-measuring system periodically over the temperature range used for the test, with reagent grade or certified chemicals.

Chemicals recommended for calibration are listed in table 1.

**Table 1 — Calibration standards**

| Chemical | Melting temperature[1] (°C) |
|---|---|
| L-Menthol-1 | 42,5 |
| Azobenzene | 69,0 |
| 8-Hydroxyquinoline | 75,5 |
| Naphthalene | 80,2 |
| Benzyl | 96,0 |
| Acetanilide | 113,5 |
| Benzoic acid | 121,7 |
| Phenacetin [*N* (4-ethoxyphenyl) acetamide] | 136,0 |
| Adipic acid | 151,5 |
| Indium | 156,4 |
| Sulfanilamide | 165,7 |
| Hydroquinone | 170,3 |
| Succinic acid | 189,5 |
| 2-Chloroanthraquinone | 208,0 |
| Anthracene | 217,0 |
| Saccharin | 229,4 |
| Tin | 231,9 |
| Tin(II) chloride | 247,0 |
| Phenolphthalein | 261,5 |

1) The temperatures indicated refer to theoretically pure chemicals; the values of the actual melting point for the standard materials used should be certified by the supplier.



### 6.2 Determination

**6.2.1** Insert the thermometer (4.3) and the capillary tube (4.2) containing the specimen into the heating chamber [4.1a)] and start the heating. When the temperature of the specimen is about 20 K below the expected melting temperature, regulate the rate of temperature increase to 2 ± 0,5 K/min. Record the temperature at which the specimen begins to change shape.

**6.2.2** Repeat the operations specified in 6.2.1 with a second specimen. If the two results obtained by the same operator on the same sample differ by more than 3 K, repeat the procedure on two new specimens.

Insufficient data are available for establishing reproducibility.

## 7 Test report

The test report shall include the following information :

a) reference to this International Standard;

b) reference of the method used (method A);

c) complete identification and description of the material tested;

d) shape and size (or mass) of the specimens;

e) previous thermal history of the specimens;

f) conditioning;

g) heating rate;

h) temperatures, in degrees Celsius or in kelvins, of two successive individual measurements, and their arithmetic mean;

i) any operational details not specified in this International Standard or regarded as optional, as well as any incidents liable to have affected the results.

# Section two : Method B — Polarizing microscope

## 8 Principle

Heating of a specimen, positioned between the polarizer and analyser of a microscope, at a controlled rate.

Measurement of the temperature at which the crystalline polymer loses its optical anisotropy, as detected by the disappearance of birefringence, as the melting temperature.

## 9 Apparatus

Ordinary laboratory apparatus and

**9.1 Microscope**, with a disk polarizer and a cap analyser, or a polarizing microscope with built-in analyser, with magnification from X 50 to X 100.

**9.2 Micro hot-stage**, consisting of an insulated metal block that can be mounted slightly above the microscope stage. This block shall be

a) provided with a hole for light passage;

b) electrically heated, with adequate controls for adjustment of heating and cooling rates;

c) constructed to provide a chamber with a heat baffle and a glass cover, for carrying out measurements in an inert atmosphere;

d) provided with a hole for insertion of a temperature-measuring device near the light hole.

**9.3 Thermometers**, calibrated, or equivalent **temperature-measuring devices**, for the test temperature ranges.

## 10 Test specimens

### 10.1 Powdered materials

Place a 2 to 3 mg portion of the powder (particle size not more than 100 µm) on a clean slide and cover with a cover glass.

Heat the specimen, the slide and the cover on a hot-plate slightly above the melting temperature of the polymer. By a slight pressure on the cover glass, form a thin film of thickness 0,01 to 0,04 mm and allow it to cool slowly by switching off the hot-plate.

### 10.2 Moulded or pelleted materials

Cut from the sample, with a microtome, a film of thickness approximately 0,02 mm, place it on a clean slide and cover with a cover glass. Heat and melt it as specified in 10.1.

### 10.3 Film or sheet materials

Cut a 2 to 3 mg portion of the film or sheet, place it on a clean slide, cover with a cover glass and proceed as specified in 10.1.

NOTE — The preliminary melting of the specimens between slide and cover presents the advantage of destroying any birefringence due to orientation or internal stresses, and also of reducing the danger of oxidation during the test. The need for an inert gas stream — as described in 11.2 — is thus limited to very special cases. The reproducibility of the measurements is also increased. However, by agreement between the interested parties, the determination may be carried out directly on the powder or cut film piece without preliminary melting. This deviation should be stated in the test report.

### 10.4 Conditioning

See 5.2.

## 11 Procedure

### 11.1 Calibration

See 6.1.



### 11.2 Determination



Place the glass microscope slide with the specimen on the micro hot-stage (9.2). Adjust the light source to maximum light intensity and focus the microscope (9.1).

For specimens that are degradable by air, adjust the gas inlet to the stage so that a slight stream of inert gas blankets the stage, keeping it under slight positive pressure to prevent ingress of air. Rotate the analyser to obtain a dark field; the crystalline material will appear bright on a dark field. Adjust the controller to heat the stage gradually (at a rate not higher than 10 K/min) to a temperature that is lower than the melting temperature, $\theta_m$, as determined approximately by previous test, by the following amounts :

10 K for $\theta_m \leqslant 150$ °C

15 K for 150 °C $< \theta_m \leqslant 200$ °C

20 K for $\theta_m > 200$ °C

Then adjust the controller so that the temperature rises at a rate of 1 to 2 K/min.

Observe the temperature at which birefringence disappears, leaving a totally dark field. Record this temperature as the melting temperature of the sample.

Turn off the heating and remove the glass cover, heat baffle and specimen slide.

Repeat the procedure with another specimen. If the two results obtained by the same operator on the same sample differ by more than 1 K, repeat the procedure on two new specimens.

According to the results of round robins, the repeatability was 2 K. Insufficient data are available for establishing reproducibility.

## 12 Test report

The test report shall include the following information :

a) reference to this International Standard;

b) reference of the method used (method B);

c) complete identification and description of the material tested;

d) shape and size (or mass) of the specimens;

e) previous thermal history of the specimens;

f) conditioning;

g) description of preliminary heating on the slide, if applicable;

h) presence and type of inert gas, if applicable;

i) heating rate;

j) temperatures, in degrees Celsius or kelvins, of two successive individual measurements and their arithmetic mean;

k) any operational details not specified in this International Standard or regarded as optional, as well as any incidents liable to have affected the results.