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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

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

Reference number
ISO 3146:2000(E)

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 734 10 79
E-mail copyright@iso.ch
Web www.iso.ch

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3146 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This third edition cancels and replaces the second edition (ISO 3146:1985), which has been technically revised.

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.

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”.

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

1 Scope

This International Standard 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 may 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 11357-3:1999, *Plastics — Differential scanning calorimetry (DSC) — Part 3: Determination of temperature and enthalpy of melting and crystallization*.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

3.1

semi-crystalline polymer

polymer containing both crystalline and amorphous phases which may be present in varying proportions

3.2

melting range

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

NOTE 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 may 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 may be used, provided that they give the same results.

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

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

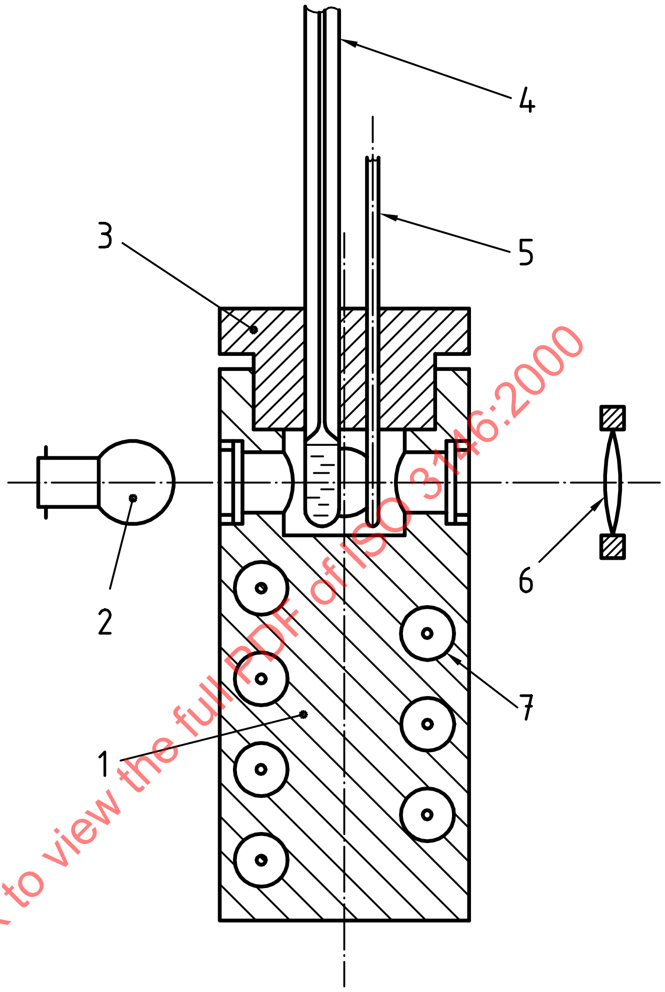
4.2.3 Calibrated thermometer, graduated in divisions of 0,1 °C. The thermometer bulb shall be positioned in such a way that heat dispersion in the apparatus is not impeded.

NOTE Other suitable temperature-measuring devices may be used.

4.3 Test samples

4.3.1 General

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



Key

1	Metal heating block
2	Lamp
3	Metal plug
4	Thermometer
5	Capillary
6	Eyepiece
7	Electrical resistance

Figure 1 — Apparatus for method A

4.3.2 Characteristics

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

4.3.3 Conditioning

If not otherwise specified or agreed between the interested parties, the test sample shall be conditioned at 23 °C ± 2 °C and a relative humidity of (50 ± 5) % for 3 h in accordance with ISO 291 prior to the measurement.

4.4 Procedure

4.4.1 Calibration

Periodically calibrate the thermometer with reagent grade or certified chemicals over the temperature range used for the test.

Chemicals recommended for calibration purposes are listed in Table 1.

Table 1 — Calibration standards

Chemical	Melting temperature[a] (°C)
L-Menthol	42,5
Azobenzene	69,0
8-Hydroxyquinoline	75,5
Naphthalene	80,2
Benzene	96,0
Acetanilide	113,5
Benzoic acid	121,7
Phenacetin (*p*-acetophenetidide)	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(I) chloride	247,0
Phenolphthalein	261,5

[a] The temperatures indicated refer to theoretically pure chemicals; the value of the actual melting point for the standard materials used should be certified by the supplier.

4.4.2 Determination

4.4.2.1 Insert the thermometer (4.2.3) and the capillary tube (4.2.2) containing the test sample into the metal block [4.2.1 a)] and begin heating. Adjust the controller [4.2.1 d)] to heat the specimen gradually (at a rate not higher than 10 °C/min) to a temperature that is about 20 °C lower than the expected melting temperature. When the temperature of the test sample is about 20 °C below the expected melting temperature, adjust the rate of temperature increase to 2 °C/min ± 0,5 °C/min. Record the temperature at which the test sample begins to change shape.

Continue heating at the same rate. Record the temperature at which the last remnants of the crystalline phase disappear.

4.4.2.2 Repeat the operations specified in 4.4.2.1 with a second test sample. If the two results obtained by the same operator on the same sample differ by more than 3 °C, repeat the procedure on two new test samples.

4.5 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) the method used (method A);

c) all details necessary for complete identification of the material tested;

d) the shape and size (or mass) of the test samples;

e) the previous thermal history of the test samples;

f) the conditioning procedure used;

g) the exact heating rate used;

h) the temperatures, in degrees Celsius, of two successive determinations, and their arithmetic mean for the temperature at which the specimen begins to change shape and for the temperature at which the last remnants of crystalline phase disappear;

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.

5 Method B — Polarizing microscope

5.1 Principle

A test sample positioned between the disc polarizer and the cap analyser of a microscope is heated at a controlled rate.

The temperature at which the crystalline phase of the polymer loses its optical anisotropy is measured as detected by the disappearance of birefringence and reported as the melting temperature.

5.2 Apparatus

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

5.2.2 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 the passage of light;

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

c) so 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 the insertion of a temperature-measuring device near the light hole a).

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

5.3 Test samples

5.3.1 Powdered materials

Place a 2 mg to 3 mg portion of the powder sample (particle size < 100 μm) on a clean glass slide and cover with a cover glass.

NOTE Depending on the effective heating rate, the temperature and time at which the test sample is held in the molten state and the effective cooling rate, the results of subsequent melting may differ.

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

5.3.2 Moulded or pelleted materials

Using a microtome, cut a film of approximately 0,02 mm thickness from the sample, place it on a clean slide and cover with a cover glass. Heat and melt it as specified in 5.3.1.