INTERNATIONAL STANDARD

ISO 16790

First edition
2005-06-15

Plastics — Determination of drawing characteristics of thermoplastics in the molten state

Plastiques — Détermination des caractéristiques d'étirage des thermoplastiques à l'état fondu

Reference number
ISO 16790:2005(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 16790 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

Plastics — Determination of drawing characteristics of thermoplastics in the molten state

1 Scope

This International Standard specifies a method for determining the drawing and break characteristics of molten plastics. The method involves the measurement of the force generated in deforming a molten filament under defined extrusion temperature and drawing conditions.

Data is generated under non-isothermal and non-homogeneous deformation conditions. However, it is useful for the interpretation of polymer behaviour in extensional flow.

The method is suitable for thermoplastics moulding and extrusion materials that can be extruded using a capillary extrusion rheometer, or an extruder with capillary rod die or other extrusion devices, and have sufficient melt strength to be handled without difficulty.

Such materials should be chemically stable and produce a uniform extrudate free from heterogeneities, bubbles, unmelted impurities, etc.

This method may provide information on:

— processability for all extrusion techniques;

— the effect of mechanical and thermal history;

— the effect of chemical structure, such as branching, entanglements and molecular mass.

This technique is one of a number of techniques that can be used to measure the extensional flow behaviour of a material. This method of measurement does not necessarily reproduce the drawing conditions to which thermoplastics are subjected to during their processing.

2 Normative references

The following referenced documents are indispensable for the application 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 1133, *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics*

ISO 11443, *Plastics — Determination of the fluidity of plastics using capillary and slit-die rheometers*

3 Terms and definitions

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

3.1
drawing
process of stretching a filament of polymer melt that is being continuously formed by a capillary extrusion rheometer or extruder or other extrusion device

3.2
melt strength
F_b
value of the melt tension at break of the extrudate.

NOTE 1 Also known as the force to break.

NOTE 2 It is expressed in newtons.

3.3
draw ratio at break
DR
ratio of the drawing velocity of the material at break to the mean velocity of the material flowing from the die

3.4
mean velocity
v_m
average velocity of the extrudate at the die exit, determined as the ratio of volume flow rate to die cross-sectional area

NOTE It is expressed in metres per second.

3.5
initial diameter
D_i
initial diameter of the extrudate after swelling on exiting from the die, the extrudate not yet having been subjected to significant drawing

NOTE 1 It is the maximum diameter the extrudate attains.

NOTE 2 It is expressed in metres.

NOTE 3 If no swelling of the extrudate occurs after exiting the die, the initial diameter is taken as the diameter of the die.

NOTE 4 This method may not be suitable for testing materials that do not exhibit swelling of the extrudate after exiting the die as such materials will be difficult to handle in drawing off and will exhibit small drawing forces.

3.6
initial velocity
v_i
velocity of the extrudate near the die exit after swelling, the extrudate not yet having been subjected to significant drawing

NOTE 1 It is determined at the position of the initial diameter of the extrudate.

NOTE 2 It is expressed in metres per second.

3.7
drawing velocity
v_t
velocity imposed on the lower end of the extrudate by the drawing unit

NOTE It is expressed in metres per second.

3.8
drawing force
F_t
force exerted on the extrudate by the drawing unit

NOTE It is expressed in newtons.

3.9
drawing length
l_e
distance between the die exit and the point where the extrudate first contacts the drawing unit's rotating wheels

NOTE It is expressed in metres.

3.10
drawing acceleration
a
rate of increase in the drawing velocity

NOTE It is expressed in metres per square second (m/s^2).

3.11
drawing velocity at break
v_b
velocity recorded at break when a constant drawing acceleration is used

NOTE It is expressed in metres per second.

4 Principle

Molten polymer is extruded from a capillary rheometer, extruder or other extrusion device at a specified temperature. The extrudate is drawn from the die by take-off wheels. Two techniques are used:

a) A series of take-off wheel velocities is used to determine the drawing force as a function of the drawing velocity.

b) A constant rate of acceleration of the take-off wheels is used to determine the melt strength (force to break) of the extrudate.

5 Apparatus

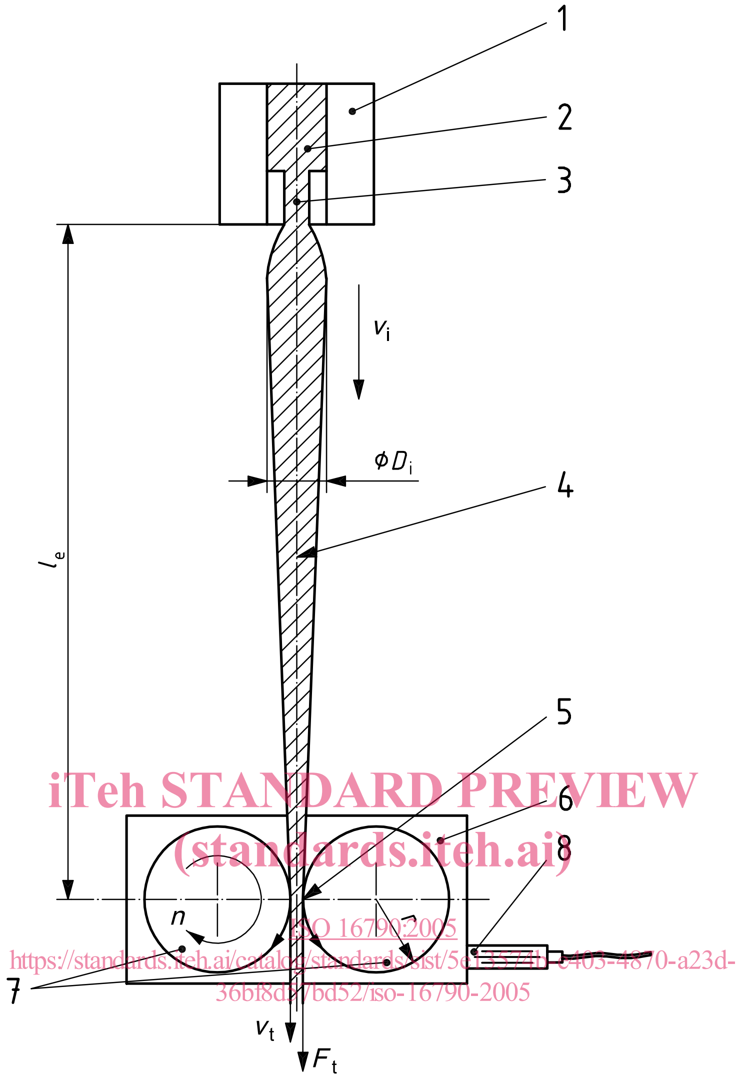
5.1 Apparatus for heating the polymer and forming the extrudate

5.1.1 General

The device to supply the molten polymer at a controlled temperature and flow rate shall consist of a heatable barrel [either a capillary extrusion rheometer (5.1.2) or an extruder (5.1.3)], the bore of which is closed at the bottom end by a die (see Figure 1). The test pressure shall be exerted on the melt contained in the barrel by a piston, a screw or pressurized gas.

5.1.2 Capillary extrusion rheometer

If a capillary extrusion rheometer is used, it shall have capillary dies, a piston, a temperature-measuring device and a pressure-measuring device meeting the requirements of ISO 11443, unless otherwise stated in this International Standard.



Key

1 rheometer or extruder

2 melt (temperature T)

3 capillary (diameter D, length l)

4 axis

5 extrudate/roller contact

6 drawing bench

7 drive rollers (radius r)

8 data acquisition:

rotational speed n

drawing force F_t

l_e drawing length

v_i initial velocity

v_t drawing velocity (= $2\pi rn$)

Figure 1 — Drawing unit — Direct drawing by two take-off rollers

5.1.3 Extruder

5.1.3.1 General

If an extruder is used, it shall be a small extruder with a screw diameter of 25 mm or less. This unit shall be equipped with a rod die and temperature-measuring device.

5.1.3.2 Melt pump

If available, a melt pump may be used to provide a uniform flow of material from the extruder to the die. If a melt pump is used, an extruder with a screw diameter larger than 25 mm may be used.

5.1.3.3 Dies

The die, of known dimensions, shall be angled vertically downwards to allow gravity to act on the extrudate.

For determining the apparent shear rate $\dot{\gamma}_{ap}$ and the apparent shear stress τ_{ap} with one capillary die, in accordance with ISO 11443, the ratio l/D of the length l to the diameter D of the die shall be at least 16 to 1 and its inlet angle shall be 180°

5.1.3.4 Temperature-measuring device

To measure the temperature of the molten polymer (see 6.2), thermocouples or platinum resistance sensors are preferred, but thermometers may be used.

5.2 Apparatus for drawing the polymer extrudate

5.2.1 Drawing unit

5.2.1.1 The drawing unit shall draw the extrudate over a specified length (the drawing length) at a controlled drawing velocity and measure the resulting drawing force.

5.2.1.2 The drawing unit shall have take-off wheels to draw the extrudate.

5.2.1.3 The drawing unit shall have controllers for the speed and acceleration of the take-off wheels.

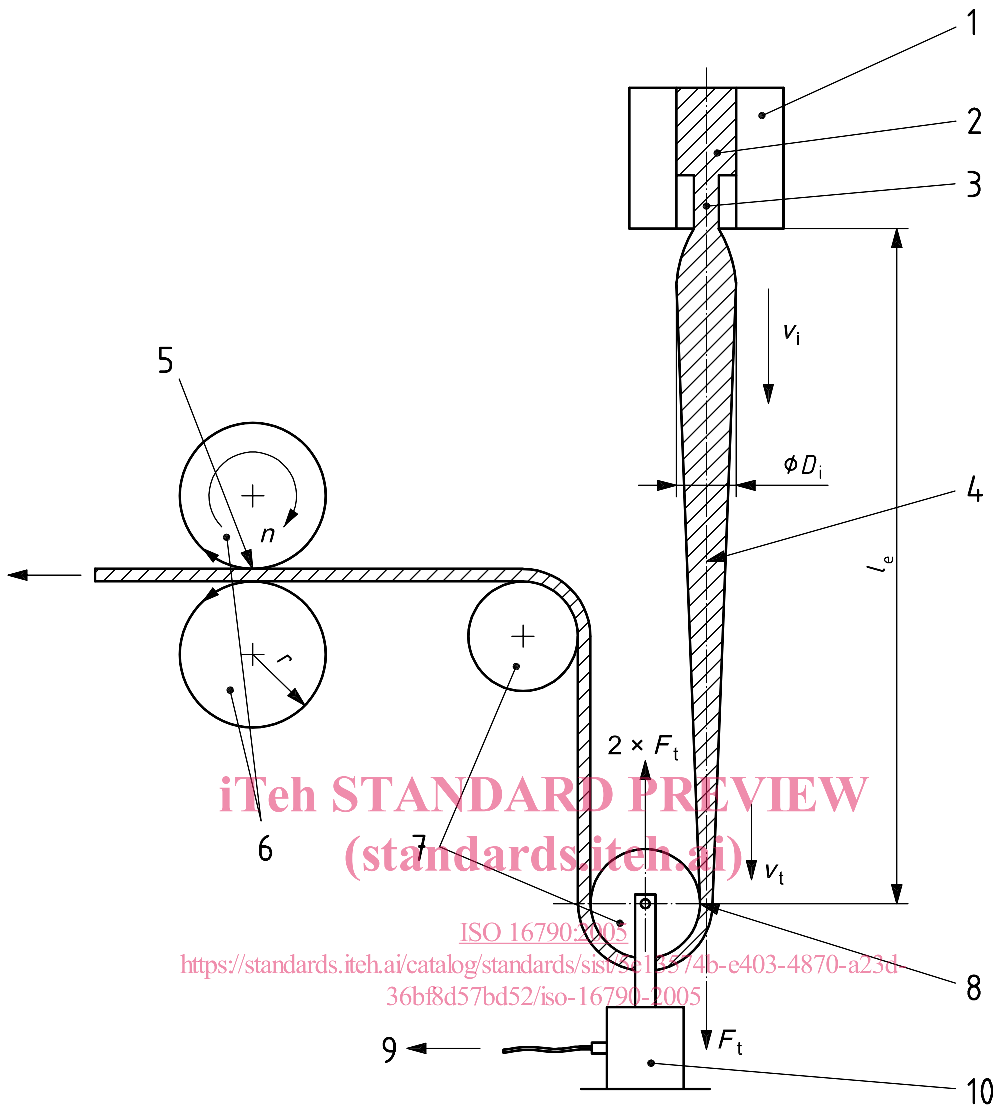
5.2.1.4 The drawing unit shall have a force sensor to measure the drawing force exerted on the extrudate.

5.2.2 Drawing unit design

Drawing may be carried out directly under the die using two rollers to take off the extrudate without excessive slippage or pinching (see Figure 1). Alternatively, it may be carried out, after the extrudate passes around the groove of one or more free-return pulley(s), by a set of two rollers that pinch and take off the extrudate without excessive slippage (Figure 2 shows a possible design: other designs are applicable). Drawing can also be done by winding up the extrudate onto a single wheel. In all cases, the axis of the extrudate in contact with the rollers (see Figure 1), the first return pulley (see Figure 2) or the take-off wheel shall coincide with the capillary die axis.

Where the extrudate passes over a return roller, drawing is considered to occur only in the section between the die exit and the initial point of contact between the extrudate and the pulley groove. In this case, the speed and drawing force of the first return pulley should preferably be measured.

NOTE The return pulley may be cooled in order to prevent any sticking of the molten polymer. The same precaution may be taken for the drawing rollers. In both cases, it is important to ensure that these devices do not have a significant influence on the measurement of the drawing force due to frictional or inertial effects or on the drawing velocity and drawing acceleration due to slippage and pinching.



Key

1 rheometer or extruder
2 melt (temperature T)
3 capillary (diameter D, length l)
4 axis
5 driven, pinched extrudate
6 drive rollers (radius r)
7 return pulleys (free axis of rotation)
8 extrudate contact
9 data acquisition
10 force transducer
l_e drawing length
v_i initial velocity
v_t drawing velocity (= $2\pi rn$)

Figure 2 — Drawing unit — Typical "take-off after return pulley" design

5.3 Data-acquisition system

The data-acquisition system shall be capable of continuously monitoring the drawing force, the drawing velocity, the temperature of the molten material, and the pressure of the melt at the entrance to the die throughout the test.

6 Calibration

6.1 General

The extruder or the rheometer shall be calibrated with respect to the measured variables and parameters, such as temperature, pressure, volume flow rates and capillary dimensions, in compliance with the procedures described in ISO 11443, unless stated otherwise in this International Standard.

6.2 Test temperature

When capillary dies are used, the test temperature shall be either the temperature of the melt in the barrel near the capillary inlet or, if this is not possible, the temperature of the barrel wall near the capillary inlet. This also pertains to the rod die of the extruder. When the barrel wall temperature is measured, thermally conductive fluids may be used in the thermometer well to improve conduction.

The temperature-measuring device used during the test shall have a resolution of 0,1 °C and be calibrated to an accuracy of ± 0,5 °C by a method traceable to certified reference standard(s).

No liquids that may contaminate the die and barrel and influence the ensuing measurements (e.g. silicone oil) shall be used as heat-transfer media during calibration. Woods metal has been found to be a suitable thermal conductor.

6.3 Capillary or rod die

The dimensions of the die shall be measured to an accuracy of ± 0,007 mm for the diameter D and ± 0,025 mm for the length l.

For comparisons between laboratories, a die having an l to D ratio of 16 to 1 and with a 180° inlet angle shall be used.

6.4 Drawing-force transducer

Calibration of the drawing-force transducer shall be carried out in accordance with the manufacturer's recommendations. The accuracy of the force measurement system shall have a maximum permissible error of ± 1 % of full scale.

6.5 Drawing velocity and drawing acceleration

The drawing velocity and drawing acceleration shall both have a maximum permissible error of ± 1 % of full scale. The apparatus may be calibrated with respect to these parameters by measuring the time for a known length of (non-stretchable) material, e.g. paper, to traverse the drawing rollers with and without acceleration, or from measurement of the rotational speed of the drawing rollers.

6.6 Drawing length

This is measured using a device such as a ruler or tape measure. The distance shall be measured to an accuracy of within ± 5 %.

For comparisons between laboratories, the drawing length shall be 100 mm ± 10 mm.