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

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

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Foreword

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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

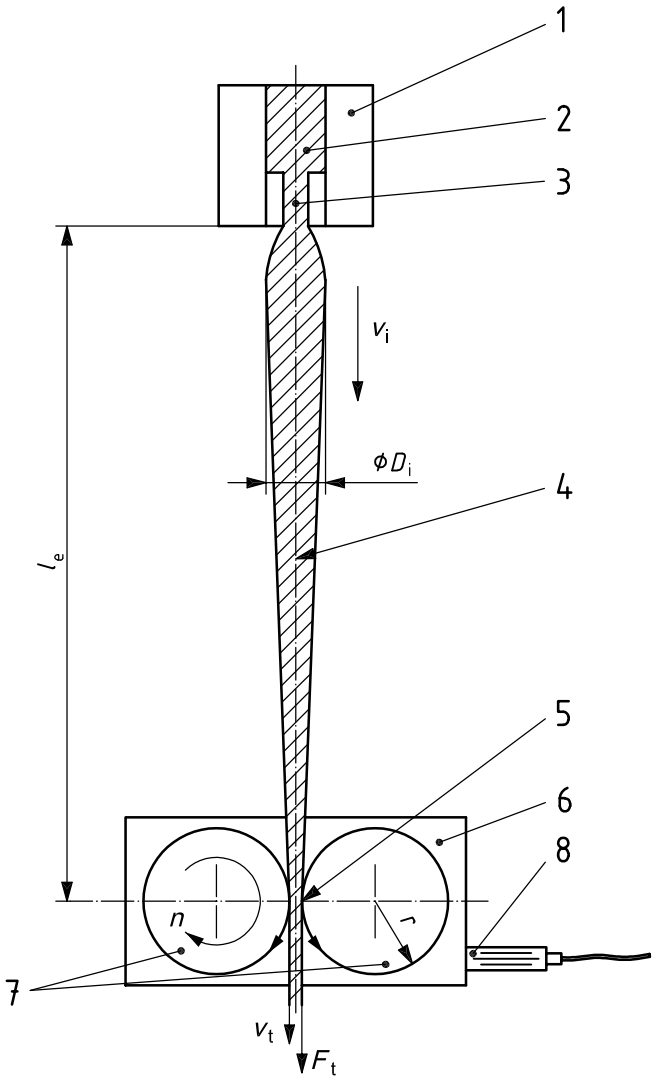
5.1 Apparatus for heating the polymer and forming the extrudate

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.

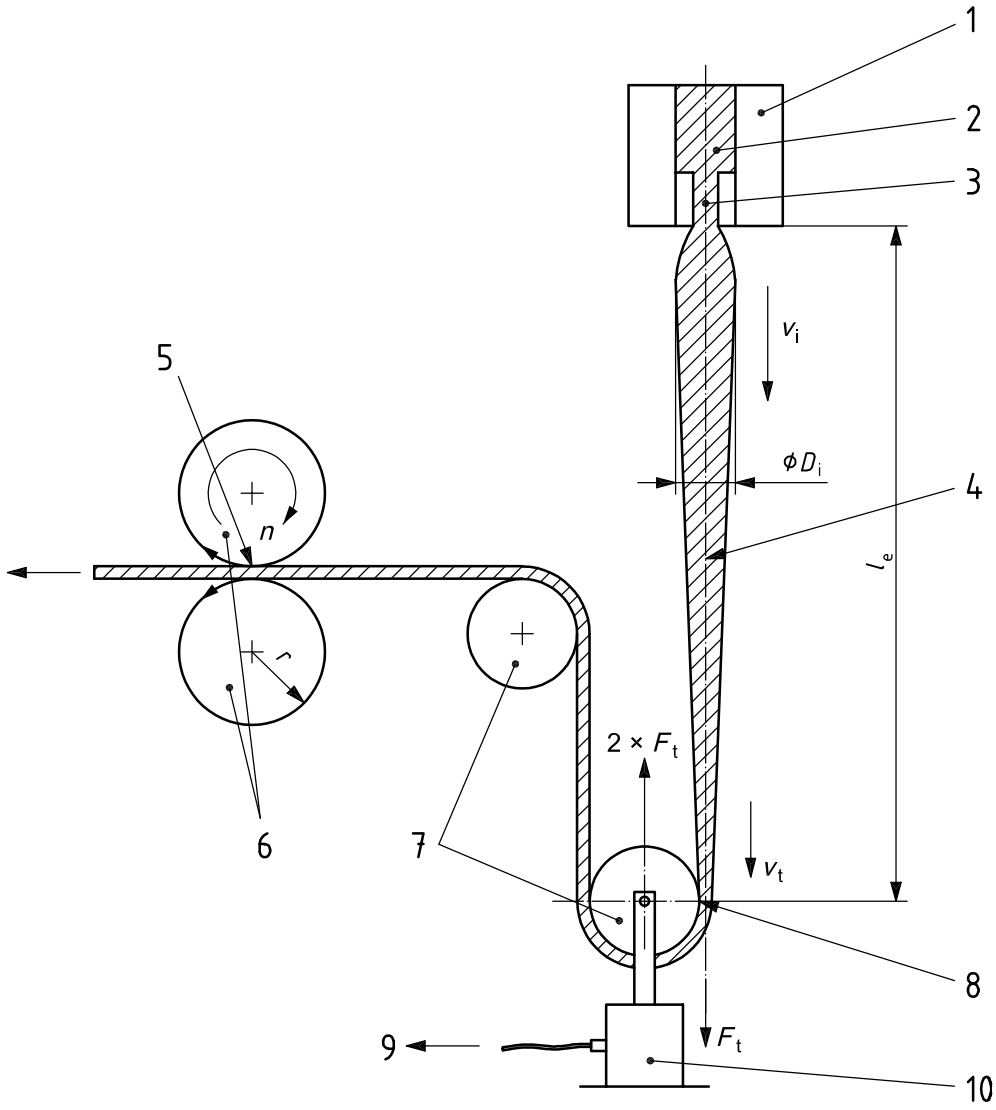
5.2.1.4 The drawing unit shall have a force sensor to measure the drawing force exerted on the extrudate.

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.

NOTE This short drawing length minimizes the cooling of the extrudate and thus maintains a more uniform temperature range over the entire length of the extrudate.

7 Sampling

A representative sample shall be taken from the product for use as the test sample. Any conditioning of the sample shall be as described in the material specification standard, if available, or by agreement between the interested parties, or in accordance with ISO 291.

8 Procedure

8.1 Cleaning the apparatus

Before each measurement, ensure that the barrel, pressure transducer bores (where applicable), piston and capillary die are free of foreign matter. Make a visual examination to check for cleanliness.

If solvents are used for cleaning, ensure that no contamination of the barrel, piston or capillary die has occurred that might influence the test results.

NOTE 1 For the purpose of cleaning, circular brushes made of copper/zinc alloy (brass) and linen cloths have proven satisfactory. However, the use of copper-containing materials may accelerate degradation of the polymer when testing polyethylene and polypropylene. Cleaning can also be performed by cautious burning out.

NOTE 2 Using graphite on threads facilitates their unlocking after testing.

WARNING — The operating conditions chosen may entail partial decomposition of the material under test and any materials used for cleaning purposes, or cause them to release dangerous volatile substances. Also, both the instrument and extrudate are likely to be very hot and present the risk of contact burns. The user of this International Standard is therefore responsible for keeping him- or herself informed of possible risks of accident and for providing the appropriate means of protection.

8.2 Rheometer or extruder preparation

Assemble the capillary rheometer or the extruder with the appropriate capillary or rod die. Allow the assembled apparatus to reach thermal equilibrium at the test temperature before applying the final torque on the die (where applicable).

It is recommended that a 2 mm diameter die be used for high-viscosity materials and a 1 mm diameter die for low-viscosity materials, although selection of the die diameter should depend on the extrudability of the material.

When using an extruder, adjust the temperature of the equipment and the rotational speed of the extruder to obtain the desired melt temperature and material flow rate from the die.

Conditions for the test should be set based on the ability to handle the extrudate during take-off.

Typical test temperature ranges for several materials are given in Table 1 (see Note). Table 1 also includes a temperature that should be used to allow comparisons between laboratories as a quality control check.

NOTE The most useful data are generally obtained at the temperatures used in processing of the material. The shear stress and shear rate applied in testing should also closely approximate those observed in actual processing.

Table 1 — Typical test temperatures

Material	Temperature range °C	Temperature for comparison °C
Polyacetal	190 to 220	190
Polyacrylate	140 to 300	230
Acrylonitrile/butadiene/styrene (ABS)	200 to 280	220
Cellulose esters	190 to 210	190
Polyamide	190 to 300	235
Polybutylene	160 to 250	190
Poly(chlorotrifluoroethylene)	185 to 310	265
Polyethylene and ethylene copolymers and terpolymers	150 to 250	190
Polycarbonate	260 to 300	300
Polypropylene	180 to 270	230
Polystyrene and styrene copolymers	180 to 280	200
Poly(vinyl chloride)	170 to 210	175
Poly(butylene terephthalate)	245 to 270	250
Poly(ethylene terephthalate)	275 to 300	285
PMMA and copolymers	180 to 300	230
Poly(vinylidene fluoride)	195 to 240	230
Poly(vinylidene chloride)	150 to 170	150
Ethylene/vinyl alcohol copolymer	190 to 230	190
Polyetheretherketone	340 to 380	372
Polyethersulfone	330 to 380	372

8.3 Loading the capillary rheometer barrel

To avoid air inclusions in the capillary rheometer, introduce the sample into the barrel in separate small quantities, performing intermediate compactions by means of a piston. Fill the barrel to within approximately 12,5 mm of the top. Accomplish charging in a maximum of 2 min.

8.4 Preheating

For the capillary rheometer, immediately after charging the barrel, start the preheat timer. Extrude a small portion of the charge out of the barrel. Stop the extrusion and wait until a preheat time of at least 5 min is completed, unless otherwise specified by the referring standard.

Check that the preheat time used is sufficient to obtain thermal equilibrium of the test sample throughout the volume of the barrel for each material to be tested. This can be done by ensuring that on increasing the preheat time the measured quantity (e.g. pressure) at constant test conditions does not change by more than $\pm$ 5 %, or by inserting a thermometer into the sample in the barrel and ensuring that the temperature is equal to the specified test temperature within the tolerance for the distance- and time-related temperature differences given in Table 2.

Then extrude a small quantity of the substance under test, stop the piston, wait for 1 min and perform the measurement.

Table 2 — Maximum allowable temperature differences as a function of distance and as a function of time

Test temperature, T °C	Temperature differences °C	
	As a function of distance[a]	As a function of time[b]
$\leqslant 200$	$\pm 1{,}0$	$\pm 0{,}5$
$200 < T \leqslant 300$	$\pm 1{,}5$	$\pm 1{,}0$
> 300	$\pm 2{,}0$	$\pm 1{,}5$

[a] Over the distance of 10 mm to 75 mm above the die.

[b] Measured at 10 mm above the die.

8.5 Running the extruder

Progressively bring the screw rotational speed up to the speed at which the test will be run. After approximately 15 min, check that the extrusion is stable by monitoring the variation in the melt pressure and the melt temperature for a minimum of 30 s. The melt pressure shall vary by no more than $\pm$ 5 % and the temperature by no more than $\pm$ 1 °C. If the melt pressure shows higher than normal variation, a melt pump shall be used to reduce the variation, or an alternative acceptable extruder shall be used.

8.6 Preliminary checks of the extrudates

8.6.1 Absence of defects

8.6.1.1 If there is a homogeneity defect (particles, bubbles, non-melting impurities, etc.), investigate the potential external causes (e.g. contamination of the material), modify the material preconditioning (e.g. drying) or adjust the extrusion conditions to remove the defect.

8.6.1.2 If there are surface finish defects (melt fracture, etc.), modify the test conditions (material temperature, flow rate, capillary diameter, etc.) that influence the flow of the extrudate to remove the defect.

8.6.2 Check on drawing down under gravity

8.6.2.1 Check that the extrudate flowing freely from the die does not stretch significantly under its own weight, nor shrink significantly due to material relaxation. Shrinkage can be detected by shortening of the extrudate upon stopping the flow. A tendency to stretch is demonstrated by a reduction in the diameter of the extrudate just after exiting from the capillary. The degree of diameter reduction can be determined as described in 8.6.2.2 to 8.6.2.6.

8.6.2.2 Allow the extrudate to flow onto a plate positioned below the capillary at a vertical distance equal to the drawing length l_e.

8.6.2.3 Then cut the extrudate for the first time roughly 30 mm from the capillary exit. Allow the extrudate to flow until its length reaches 60 mm and cut it a second time at the capillary exit (see Figure 3).

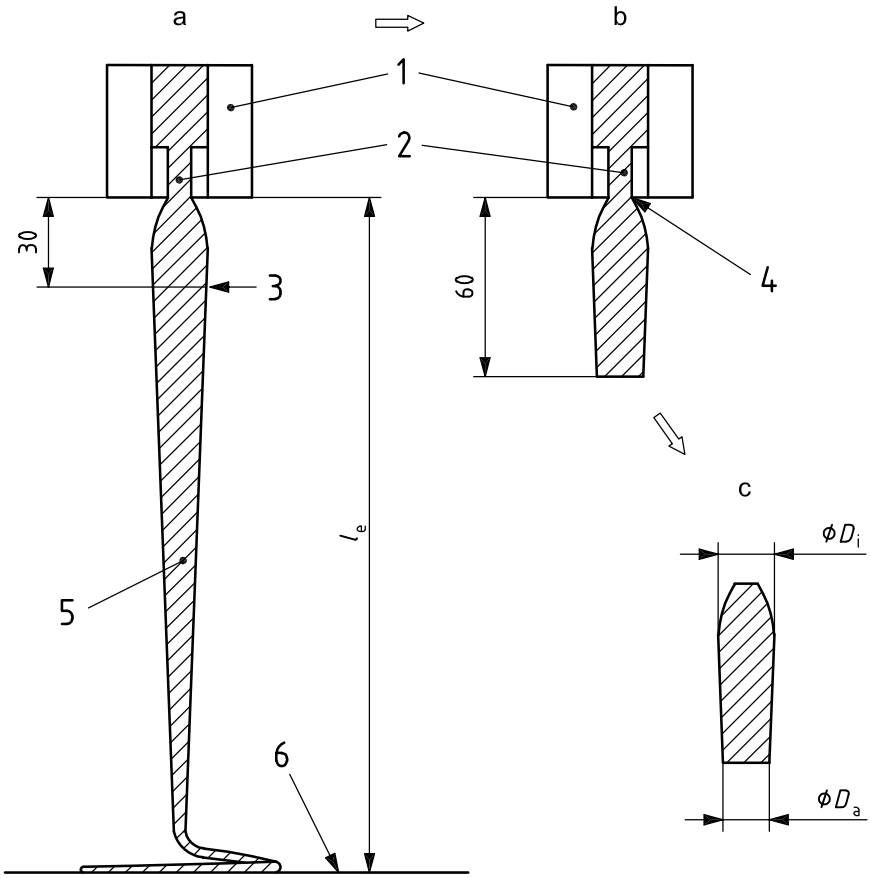
8.6.2.4 Carefully seize this extrudate with tongs, taking care not to damage the cut ends, and allow it to cool down to room temperature.

8.6.2.5 Compare the diameter D_a at a point just above the first cut to the maximum diameter D_i just below the end of the extrudate where the second cut was made (see Figure 3). The smaller diameter D_a is a result of extrudate draw-down.

8.6.2.6 If the ratio of the diameters D_a/D_i is less than 0,9, the extrudate is considered to be susceptible to stretching under the sole effect of its weight. If the ratio is less than 0,9, modify the extrusion conditions by reducing the melt temperature or increasing the extrusion rate until a ratio of at least 0,9 is obtained. If this is not possible, set the extrusion conditions to get the ratio as close as possible to 0,9. If the ratio remains less than 0,9, record this in the test report.

NOTE The factor of 0,9 is significant in that if the ratio is below this value it means that gravity is playing a significant role in the measurement and the true force of the drawing is not being measured.

Dimensions in millimetres



Key

1 rheometer or extruder
2 capillary (diameter D, length l)
3 first cut
4 second cut
5 strand
6 plate
l_e drawing length

Figure 3 — Extrusion of an extrudate without drawing — Verification of non-elongation of the extrudate

8.7 Determination of the extrudate behaviour

8.7.1 Determination of the mean velocity

When using a screw extruder, with the extrusion parameters set and stable conditions attained, measure the mass flow rate Q by weighing the quantity of material extruded from the die in 1 min. Weigh the extrudate to an accuracy of $\pm$ 1 g.

NOTE 1 The mass flow rate Q corresponds to the mean velocity v_m of the extrudate flow exiting from the die. This mean velocity is normally slightly higher than the initial velocity v_i, which takes into account the extrudate swelling near the die exit.

NOTE 2 The mass flow rate Q must not be confused with the melt mass-flow rate (MFR) obtained using ISO 1133.

The mean velocity is given by

$$v_m = Q/(\rho_T \times S_T \times 60) \quad (1)$$

where

v_m is the mean velocity, expressed in metres per second;

Q is the extrudate mass flow rate, expressed in kilograms per minute;

ρ_T is the melt density at temperature T, expressed in kilograms per cubic metre;

S_T is the capillary cross-sectional area at temperature T, expressed in square metres.

When using a piston-driven extruder, determine the mass flow rate Q from the piston speed and diameter and the density of the melt.

NOTE 3 When using a piston-driven extruder, the mean velocity can be determined from the piston speed and the diameters of the piston and die, without recourse to the melt density.

8.7.2 Determination of the drawing force as a function of the drawing velocity

8.7.2.1 Position the drawing unit underneath the capillary exit at the prescribed drawing length and engage the extrudate. A suitable drawing velocity for start-up is 0,012 5 m/s, although this will vary from material to material and from temperature to temperature.

8.7.2.2 Start recording the drawing force and drawing velocity.

8.7.2.3 Increase the drawing velocity until it is equal to the initial velocity of the extrudate. For an extrudate which is drawing down under its own weight, the velocity at the drawing unit will be higher than the initial velocity and thus material will build up at the drawing unit. Increase the speed until the wheel speed matches the extrudate speed at the wheel. This is referred to as the minimum drawing velocity. If there is material shrinkage, the drawing velocity shall be set to the initial velocity.

8.7.2.4 After the extrudate has stabilized and baseline signals have been recorded for these conditions, increase the drawing velocity so as to show a measurable force on the drawn extrudate, by observing the indications of the force transducer.

8.7.2.5 In systems where the take-off system has two take-off wheels (see Figure 1), it is necessary to keep the amount of material below the take-off to a minimum to eliminate post-tensioning effects.

8.7.2.6 Wait for a minimum of 30 s for the applied drawing force to stabilize. Record the drawing force F_t and the drawing velocity v_t for this first stage of measurement.

8.7.2.7 Continue to make additional measurements by increasing the drawing velocity in successive increments, waiting a minimum of 30 s for the resultant load to stabilize for each measurement.

8.7.2.8 Continue to increase the drawing velocity until extrudate break occurs. Velocity increases shall be made such that at least six force measurements are made at different velocities before break occurs.

8.7.2.9 Repeat the procedure a minimum of two additional times to determine the repeatability of the measurement.

8.7.3 Measurement of melt strength

8.7.3.1 Set the drawing velocity of the take-off wheels to a value slightly below the minimum drawing velocity observed in the initial study of drawing force versus drawing velocity (see 8.7.2.3). Allow the melt strand to fall between the wheels, and then close the wheels to firmly grip the melt strand. A suitable drawing velocity for start-up is 0,012 5 m/s, although this will vary from material to material and from temperature to temperature.

8.7.3.2 Start recording the drawing force and drawing velocity.

8.7.3.3 Increase the drawing velocity until it is equal to the minimum drawing velocity.

8.7.3.4 After the extrudate has stabilized and baseline signals have been recorded for these conditions, start the constant acceleration. Note when acceleration is applied.

A suitable starting value for the acceleration is 0,000 6 m/s^2, although this setting is material- and temperature-dependent. Recommended acceleration values for testing of polyolefins are 0,000 6 m/s^2, 0,000 8 m/s^2, 0,001 0 m/s^2 and 0,001 2 m/s^2.

The initial velocity and acceleration values used are dependent on the elasticity of the material being tested. If the material exhibits very low elasticity, as indicated by breakage of the extrudate early on in the test, then reduce the acceleration.

8.7.3.5 Once the melt strand has broken and the force has returned to zero, disengage the take-off wheels.

8.7.3.6 Record the time to break, the melt strength and the draw ratio at break.

8.7.3.7 A minimum of three determinations are recommended to determine the data repeatability.

9 Calculation and expression of results

9.1 Initial cross-sectional area of the extrudate

The initial cross-sectional area at room temperature $(S_i)_{T_0}$ is determined from the measurement of the initial diameter $(D_i)_{T_0}$ of the extrudate cooled down to room temperature T_0:

$$\left(S_i\right)_{T_0} = \pi \times \left(D_i\right)^2_{T_0} \Big/ 4 \qquad (2)$$

where $(D_i)_{T_0}$ is the initial diameter at room temperature T_0, expressed in metres.

From this, the initial cross-sectional area at the test temperature T is deduced, taking into account the ratio of the extrudate densities at T and T_0 and assuming equal shrinkage rates in all directions, in accordance with the following equation:

$$(S_i)_T = (S_i)_{T_0} \times (\rho_{T_0} / \rho_T)^{2/3} \quad (3)$$

where

$(S_i)_T$ is the initial cross-sectional area at temperature T, expressed in square metres;

ρ_{T0} is the density at room temperature T_0, expressed in kilograms per cubic metre;

ρ_T is the density at the test temperature T, expressed in kilograms per cubic metre.

Alternatively, the initial diameter at the test temperature may be measured by non-contacting optical means and used to calculate the initial cross-sectional area at the test temperature.

9.2 Initial velocity

From the mass flow rate Q, calculate the initial velocity of the extrudate at temperature T:

$$(v_i)_T = Q/[\rho_T \times (S_i)_T] \quad (4)$$

where

$(v_i)_T$ is the initial velocity at temperature T, expressed in metres per second;

Q is the mass flow rate, expressed in kilograms per second.

NOTE This initial velocity allows the predetermination of the minimum drawing velocity of the extrudate which has not been subjected to drawing. This initial velocity is equal to the minimum drawing velocity when the extrudate is not subjected to drawing by the take-off wheels or drawing under its own weight.

9.3 Drawing velocities

Drawing velocities are calculated in accordance with the following equation:

$$v_t = 2\pi r n \quad (5)$$

where

r is the radius of the rollers, expressed in metres;

n is the rotational speed of the drawing rollers, expressed in revolutions per second.

9.4 Drawing forces

The drawing force is measured during the two drawability determinations as

a) the measured forces F_t corresponding to the different velocities v_t when the drawing velocity is increased in a step-wise manner;

b) the force F_b corresponding to the velocity v_b recorded at break when a constant drawing acceleration is used.

When the capability exists, develop a plot of the force versus drawing velocity during acceleration.

9.5 Draw ratio

Calculate the draw ratio at break DR using the following equation:

$$DR = v_b/v_m \quad (6)$$

where

v_b is the drawing velocity at break, expressed in metres per second;

v_m is the mean velocity at the die, expressed in metres per second.

10 Precision

The precision of this test method is unknown because intralaboratory and interlaboratory data are not available.

11 Test report

The test report shall include the following information, as applicable:

General:

a) A reference to this International Standard;

b) the date of the test.

Material and test conditions:

c) A description of the material under test and details of any conditioning carried out on the material;

d) a description of the equipment used, i.e. capillary rheometer or extruder details;

e) the geometry and dimensions of the capillary or rod die;

f) the sample preheat time, for capillary rheometer based measurements;

g) the temperature profile, screw rotation speed and all other settings necessary to repeat the measurement, for extruder-based measurements;

h) a description of drawing unit;

i) the velocity and acceleration settings used;

j) the drawing length l_e;

k) the mass flow rate Q;

l) the extrusion pressure at the die entrance;

m) the melt extrusion temperature;

n) the melt density at the test temperature;

o) the melt density at room temperature.

Test results:

p) The initial diameter of the extrudate D_i;

q) the ratio of D_a to D_i;

r) the initial velocity v_i;

s) the drawing velocities v_t;

t) the drawing forces F_t;

u) the melt strength F_b;

v) the draw ratio at break DR;

w) a graphical representation of

- drawing force versus drawing velocity, when the velocity is increased in increments,
- drawing force versus time from minimum drawing velocity, on application of acceleration to break,
- drawing force versus drawing velocity from minimum drawing velocity, on application of acceleration to break;

x) a description of the sample failure mechanism (break or cut);

y) a description of any defects in the extrudate;

z) a description of any problems encountered during the test.

IMPORTANT — The melt strength shall never be reported alone. These results shall always be reported with the actual measurement conditions.

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