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Plastics — Determination of the fluidity of plastics using capillary and slit-die rheometers

Plastiques — Détermination de la fluidité au moyen de rhéomètres équipés d'une filière capillaire ou plate

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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 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 11443 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 11443:1995), of which Clause 4, Subclauses 5.1.2, 5.1.3, 5.1.4, 5.4, 5.5, 7.2 and 8.5, Clause 9 and Annex C have been technically revised.

Plastics — Determination of the fluidity of plastics using capillary and slit-die rheometers

1 Scope

This International Standard specifies methods for determining the fluidity of plastics melts subjected to shear stresses at rates and temperatures approximating to those arising in plastics processing. Testing plastics melts in accordance with these methods is necessary since the fluidity of plastics melts is generally not dependent solely on temperature, but also on other parameters, in particular shear rate and shear stress.

The methods described in this International Standard are useful for determining melt viscosities from 10 Pa·s to 10^7 Pa·s, depending on the measurement range of the pressure and/or force transducer and the mechanical and physical characteristics of the rheometer. The shear rates occurring in extrusion rheometers range from 1 s^{-1} to 10^6 s^{-1}.

Elongational effects at the die entrance cause extrudate swelling at the die exit. Methods for assessing extrudate swelling have also been included.

The rheological techniques described are not limited to the characterization of wall-adhering thermoplastics melts only: for example, thermoplastics exhibiting "slip" effects [1], [2] and thermosetting plastics can be included. However, the methods used for determining the shear rate and shear viscosity are invalid for materials which are not wall-adhering. Nevertheless, the standard can be used to characterize the rheological behaviour of such fluids for a given geometry.

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 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 6507-1, *Metallic materials — Vickers hardness test — Part 1: Test method*

ISO 11403-2, *Plastics — Acquisition and presentation of comparable multipoint data — Part 2: Thermal and processing properties*

ISO 1133, *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics*

3 Terms and definitions

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

3.1
Newtonian fluid
fluid for which the viscosity is independent of the shear rate and of time

3.2
non-Newtonian fluid
fluid for which the viscosity varies with the shear rate and/or with time

NOTE For the purposes of this International Standard, this definition refers to fluids for which the viscosity varies only with the shear rate.

3.3
apparent shear stress
τ_{ap}
fictive shear stress to which the melt in contact with the die wall is subjected, expressed in pascals (Pa)

NOTE It is calculated as the product of test pressure and the ratio of die cross-sectional area to die-wall area.

3.4
apparent shear rate
$\dot{\gamma}_{ap}$
fictive shear rate that the melt at the wall would experience at the observed volume flow rate if its behaviour were Newtonian, expressed in reciprocal seconds (s^{-1}).

3.5
true shear stress
τ
actual shear stress to which the melt in contact with the die wall is subjected, expressed in pascals (Pa)

NOTE 1 It is estimated from the test pressure p by applying corrections for entrance and exit pressure losses, or is directly determined from the melt-pressure gradient in the channel.

NOTE 2 For the purposes of notation, the absence of a subscript is used to denote true values.

3.6
true shear rate
$\dot{\gamma}$
shear rate obtained from the apparent shear rate $\dot{\gamma}_{ap}$ by taking into account the deviations from Newtonian behaviour by appropriate correction algorithms (see Note to 8.2.2), expressed in reciprocal seconds (s^{-1})

NOTE For the purposes of notation, the absence of a subscript is used to denote true values.

3.7
viscosity
η
viscosity in steady shear, defined as the ratio $\tau/\dot{\gamma}$ of true shear stress τ to true shear rate $\dot{\gamma}$, expressed in pascal seconds (Pa·s)

3.8
apparent viscosity
η_{ap}
ratio $\tau_{ap}/\dot{\gamma}_{ap}$ of apparent shear stress τ_{ap} to apparent shear rate $\dot{\gamma}_{ap}$, expressed in pascal seconds (Pa·s)

3.9
Bagley corrected apparent viscosity
η_{apB}
ratio $\tau/\dot{\gamma}_{ap}$ of true shear stress τ to apparent shear rate $\dot{\gamma}_{ap}$, expressed in pascal seconds (Pa·s)

3.10
Rabinowitsch corrected apparent viscosity
η_{apR}
ratio $\tau_{ap}/\dot{\gamma}$ of apparent shear stress τ_{ap} to true shear rate $\dot{\gamma}$, expressed in pascal seconds (Pa·s)

NOTE This term is appropriate for use when testing with a single die of large length to diameter aspect ratio for which entrance effects are negligible.

3.11
volume flow rate
Q
volume of melt flowing through the die per unit time, expressed in cubic millimetres per second (mm^3/s)

3.12
swell ratio at room temperature
S_a
ratio of the diameter of the extrudate to the diameter of the capillary die, both measured at room temperature

3.13
swell ratio at the test temperature
S_T
ratio of the diameter of the extrudate to the diameter of the capillary die, both measured at the test temperature

3.14
percent swell at room temperature
s_a
difference between the diameter of the extruded strand and the diameter of the capillary die, expressed as a percentage of the diameter of the capillary die, both measured at room temperature

3.15
percent swell at the test temperature

s_T
difference between the diameter of the extruded strand and the diameter of the capillary die, expressed as a percentage of the diameter of the capillary die, both measured at the test temperature

NOTE Equivalent slit-die extrudate swell terms can be derived, based on the thickness of slit-die extrudate with reference to the slit-die thickness.

3.16
preheating time
time interval between completion of charging of the barrel and the beginning of measurement

3.17
dwell time
time interval between the completion of charging of the barrel and the end of measurements

NOTE In certain special cases, it may be necessary to note the dwell time at the end of each measurement where more than one measurement per barrel filling is made.

3.18
extrusion time
time corresponding to the period of measurement for a given shear rate

3.19
critical shear stress
value of the shear stresses at the die wall at which any of the following occur:

— a discontinuity in the curve plotting shear stress against flow rate or shear rate;

— roughness (or waving) of the extrudate as it leaves the die

NOTE It is expressed in pascals (Pa).

3.20
critical shear rate
shear rate corresponding to the critical shear stress, expressed in reciprocal seconds (s^{-1})

4 General principles

The plastics melt is forced through a capillary or slit die of known dimensions. Two principal methods can be used: for a specified constant test pressure p the volume flow rate Q is measured (method 1), or for a specified constant volume flow rate Q the test pressure p is measured (method 2). These methods can be used with capillary dies (method A) and slit dies (method B). For full designation of the test-method options, see Table 1.

Table 1 — Designation of test methods

Die cross-section	Preset parameter	
	Test pressure, p	Volume flow rate, Q
Circular (capillary die)	A1	A2
Rectangular (slit die)	B1	B2

Measurements can be made using a range of values of the preset parameter (either applied test pressure in method 1, or volume flow rate in method 2).

If a slit die with pressure transducers positioned along its length and also upstream of the die entry is used, then entrance and exit pressure-drop values can be determined. If capillary dies of the same radius but of varying length are used, then the sum of the entrance and exit pressure drops can be determined.

A slit die with pressure transducers positioned along its length is particularly suited for automated measurements using on-line computer evaluation.

Recommended values for capillary die dimensions and for flow rates and temperatures to be used in testing are presented either in the relevant clauses below or in ISO 11403-2.

NOTE In using a slit die, either the aspect ratio H/B between the thickness H and the width B of the slit is small or else a correction for H/B (see Annex A) is necessary. In the latter case, the calculated quantities are dependent on assumptions made in deriving the correction equations used, notably that elastic effects are irrelevant.

5 Apparatus

5.1 Test device

5.1.1 General

The test device shall consist of a heatable barrel, the bore of which is closed at the bottom end by an interchangeable capillary or slit die. The test pressure shall be exerted on the melt contained in this barrel by a piston, screw or by the use of gas pressure. Figures 1 and 2 show typical examples; other dimensions are permitted.

5.1.2 Rheometer barrel

The barrel shall consist of a material resistant to wear and corrosion up to the maximum temperature of the heating system.

The barrel may have a lateral bore for the insertion of a melt-pressure transducer close to the die entrance.

The permissible deviations in the mean bore diameter throughout the length of the barrel shall be less than ± 0,007 mm.

The barrel shall be manufactured using techniques and materials that produce a Vickers hardness preferably of at least 800 HV 30 (see ISO 6507-1 and Note 1) and a surface roughness of less than R_a = 0,25 µm (average arithmetic discrepancy, see ISO 4287).

NOTE 1 For temperatures up to 400 °C, nitrided steel has been found suitable. Materials of hardness values lower than that specified but of sufficient corrosion and abrasion resistance have been found to be acceptable for construction of the barrel and dies.

NOTE 2 An increase in barrel-bore diameter increases the number of measurements that can be made with a single barrel filling and increases the shear-rate range of the instrument. Disadvantages of using a larger barrel-bore diameter are that larger sample masses are required, and that the time necessary to reach temperature equilibrium throughout the sample is greater. The barrel-bore diameters of commercially available rheometers lie in the range between 6,35 mm and 25 mm.

5.1.3 Capillary dies (method A)

5.1.3.1 The entire length of the capillary die wall shall be machined to an accuracy of ± 0,007 mm for the diameter (D) and ± 0,025 mm for the length (L) (see Figure 1).

The capillary shall be manufactured using techniques and materials that produce a Vickers hardness preferably of at least 800 HV 30 (see ISO 6507-1 and Note 1 to 5.1.2) and a surface roughness of less than R_a = 0,25 µm (average arithmetic discrepancy, see ISO 4287).

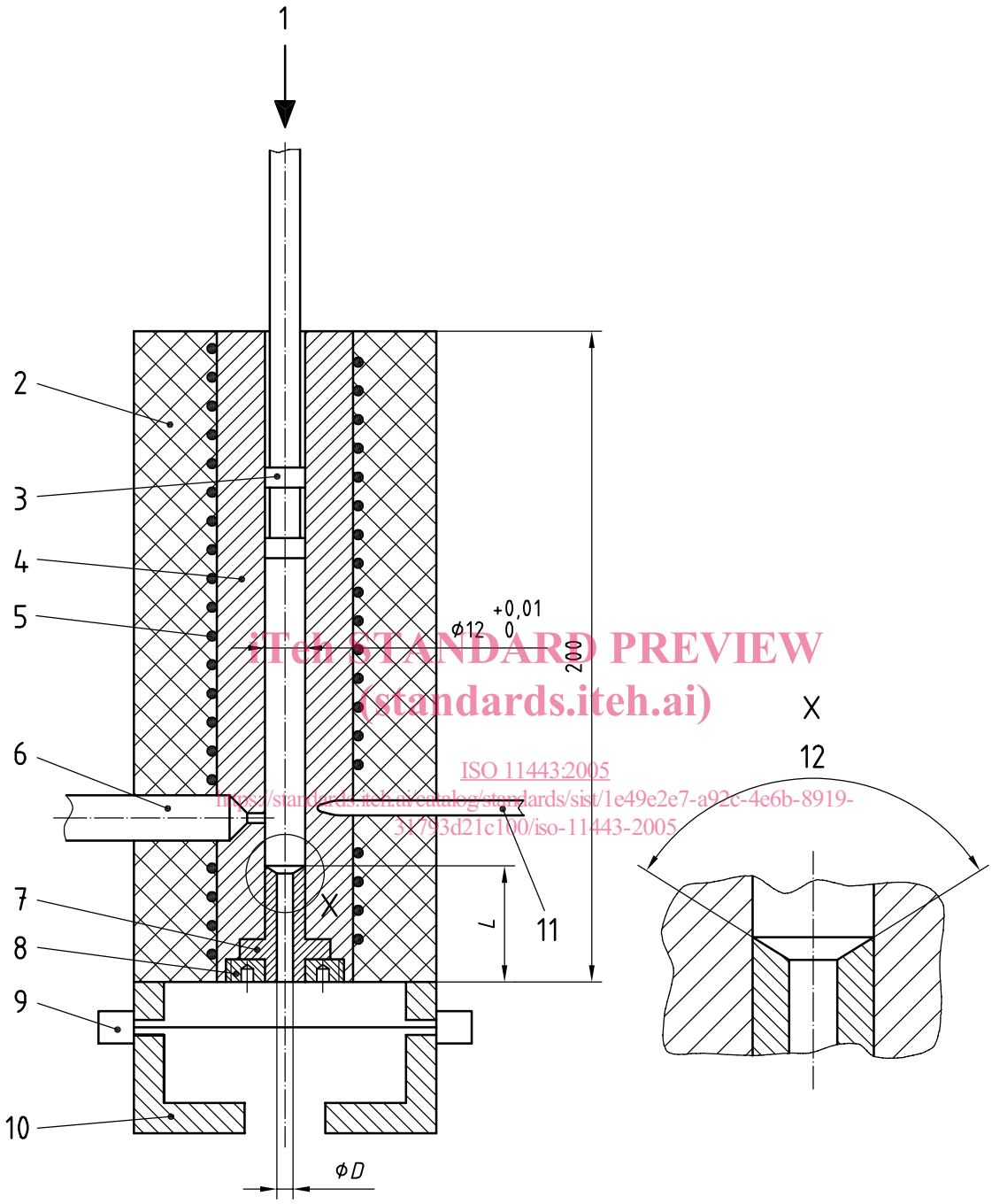
The capillary opening shall show no visible machining marks nor perceptible eccentricity.

NOTE 1 Diameters of capillary dies typically used lie in the range between 0,5 mm and 2 mm, with various lengths to obtain the desired L/D ratios. For testing of filled materials, larger diameters might be required.

NOTE 2 Hardened steel, tungsten carbide, stellite and hardened stainless steel are the most common die materials.

NOTE 3 The precision with which capillary dimensions can be measured is dependent upon both the capillary radius and the capillary length. With capillaries of diameter smaller than 1,25 mm, the specified precision (± 0,007 mm) is difficult to obtain. Due to the extreme sensitivity of flow data to capillary dimensions, it is important that the capillary dimensions, and the precision with which the dimensions are measured, are known and reported. This also applies to the dimensions (thickness, width and length) of slit dies (see 5.1.4).

Dimensions in millimetres

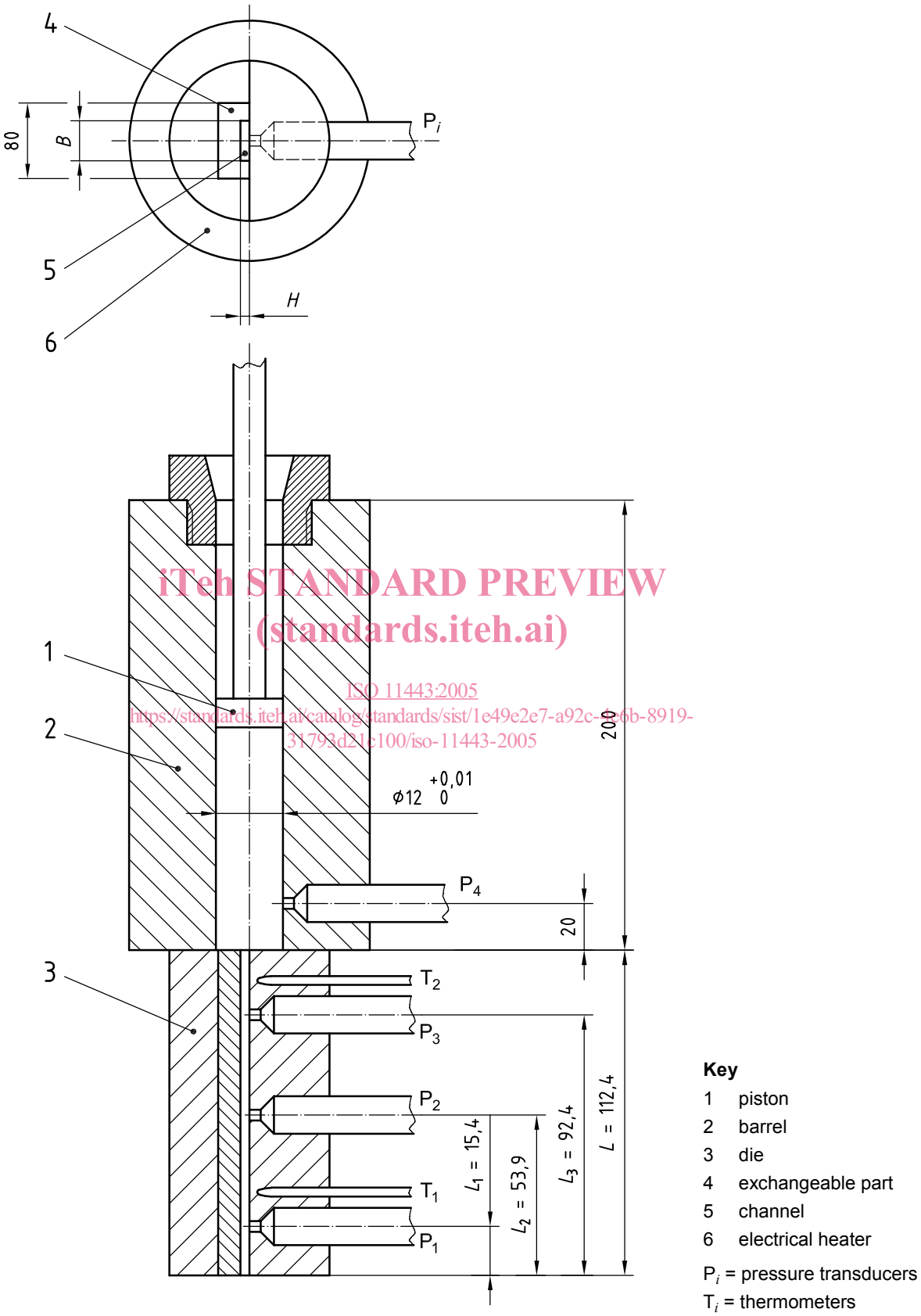


Key

1	applied force or constant velocity	5	heating coil	9	optical sensor
2	thermal insulation	6	pressure transducer	10	temperature-controlled air chamber
3	piston	7	capillary die	11	thermometer
4	barrel	8	die-retaining nut	12	inlet angle

Figure 1 — Typical example of an extrusion rheometer used with a capillary die

Dimensions in millimetres



Key

1 piston
2 barrel
3 die
4 exchangeable part
5 channel
6 electrical heater

P_i = pressure transducers
T_i = thermometers

Figure 2 — Typical example of an extrusion rheometer used with a slit die

5.1.3.2 To determine the apparent shear rate $\dot{\gamma}_{ap}$ and the apparent shear stress τ_{ap} with one capillary die, the ratio L/D of the length L to the diameter D of the capillary die shall be at least 16 and its inlet angle shall be 180°, unless otherwise specified by the referring standard. Only data obtained with capillaries of the same inlet angle (± 1°), length (± 0,025 mm) and diameter (± 0,007 mm) shall be compared. The inlet angle is defined in Figure 1.

It is recommended that a die of length either 16 mm or 20 mm, diameter of 1 mm and entry angle of 180° be used (see Note 1). Options for the die diameter of 0,5 mm, 2 mm or 4 mm are permitted when the recommended value is not appropriate, for example for heavily filled materials. For dies of diameter other than 1 mm, the recommended ratio of length to diameter (L/D) shall be the same, where possible, as that of the 1 mm diameter die used in that instrument.

NOTE 1 Die lengths of 16 mm and 20 mm are most commonly used, the choice often being dependent on, and limited by, the design of the instrument.

NOTE 2 For a given value of the apparent shear rate, the effect of shear heating of the melt is reduced by use of smaller-diameter capillary dies.

5.1.3.3 To determine the true shear rate $\dot{\gamma}$ and the true shear stress τ, capillary dies of the same diameter (± 0,007 mm) and inlet angle (± 1°) and having at least two different L/D ratios selected from the recommended series L/D = 0,25 to 1, 5, 10, 16, 20, 30, 40 (see also 8.4.2) are required, provided the following conditions are met.

The use of only two dies, of the same diameter (± 0,007 mm) and inlet angle (± 1°), of $L/D \leqslant 5$ and $L/D \geqslant 16$ is permitted where the test conditions are such that the resultant Bagley plot is not significantly non-linear, i.e. these conditions having been established in advance for each class of sample by using additional dies (see 8.4). When using only two dies the difference in the L/D ratios of the two dies shall be at least 15.

It is recommended that, when using only two dies to determine shear viscosity corrected for entrance pressure drop effects, a short die of length to diameter (L/D) ratio in the range 0,25 to 1, and a long die of length to diameter (L/D) ratio in the range 16 to 20, both dies having a diameter of 1 mm and an entry angle of 180°, be used. Options for the die diameter, of 0,5 mm, 2 mm or 4 mm, shall be permitted when the recommended value of 1 mm is not appropriate, for example for heavily filled materials. For dies of diameter other than 1 mm the recommended ratios of length to diameter (L/D) shall be the same as that specified for the 1 mm diameter dies.

NOTE The procedure for correction for entrance pressure drop effects (see 8.4) is based on an extrapolation of data to a die length of zero, rather than by making the approximation that the short die yields the entrance pressure drop value.

5.1.4 Slit dies (method B)

5.1.4.1 The entire length of the slit die shall be machined to an accuracy of ± 0,007 mm for the thickness, ± 0,01 mm for the width and ± 0,025 mm for the length. As applicable, the distance between the centres of the pressure transducers and the exit plane shall be determined to ± 0,05 mm. (See Note 3 to 5.1.3.1.)

The die shall be manufactured using techniques and materials that produce a Vickers hardness preferably of at least 800 HV 30 (see ISO 6507-1 and Note 1 to 5.1.2) and a surface roughness of less than R_a = 0,25 µm (average arithmetic discrepancy, see ISO 4287.)

NOTE For slit-die materials, see Note 1 to 5.1.2 and Note 2 to 5.1.3.1.

5.1.4.2 To determine the apparent shear rate $\dot{\gamma}_{ap}$ and the apparent shear stress τ_{ap}, unless otherwise specified by the referring standard, the slit die shall have a ratio H/B of the thickness H to the width B of at most 0,1 and shall have an inlet angle of 180°. Only data obtained with slit dies of the same inlet angle (± 1°), thickness (± 0,007 mm), width (± 0,01 mm) and length (± 0,025 mm) shall be compared.

5.1.4.3 To determine the true values of shear rate $\dot{\gamma}$ and shear stress τ, slit dies conforming to the specification given in 5.1.4.1 and 5.1.4.2 may be used in exactly the same way as capillary dies, i.e. using the Bagley correction method modified accordingly (see 8.4). Alternatively, a slit die with pressure transducers positioned along the length of its channel can be used to determine true shear stress values.

5.1.5 Piston

If a piston is used, its diameter shall be 0,040 mm ± 0,005 mm smaller than the barrel-bore diameter. It may be equipped with split or whole sealing rings in order to reduce melt backflow past the land of the piston. The hardness of the piston shall be less than that of the barrel, but not less than 375 HV 30 (see ISO 6507-1).

5.2 Temperature control

For all temperatures that can be set, the barrel-temperature control shall be designed such that, within the range of the capillary die or slit die, as applicable, and the permissible filling height of the barrel, the temperature differences and variations measured at the wall do not exceed those given in Table 2 for the duration of the test.

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

Test temperature, θ °C	**Temperature difference from the set temperature as a function of distance** [a] °C	**Temperature variation as a function of time** [a] °C
$\leqslant 200$	± 1,0	± 0,5
$200 < \theta \leqslant 300$	± 1,5	± 1,0
> 300	± 2,0	± 1,5

[a] For all positions within the range of the capillary die or slit die, as applicable, and the permissible filling height of the barrel, for the duration of the test.

The test device shall be designed so that the test temperature can be set in steps of 1 °C or less.

5.3 Measurement of temperature and calibration

5.3.1 Test temperature

5.3.1.1 Method A: Capillary dies

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. It is preferable that the test temperature is measured at a position not more than 10 mm above the capillary inlet. (See also 5.3.2.)

5.3.1.2 Method B: Slit dies

When slit dies are used, the die-wall temperature shall be measured and taken as the test temperature. This temperature shall be equal to the test temperature measured in the barrel to within the distance-related and time-related temperature tolerances given in Table 2. (See also 5.3.1.1 and 5.3.2.)

5.3.2 Measurement of test temperature

The tip of the temperature-measuring device shall be either in contact with the melt or, if this is not possible, in contact with the metal of the barrel or die wall not more than 1,5 mm from the melt channel. Thermally conductive fluids may be used in the thermometer well to improve conduction. Thermometers, preferably thermocouples or platinum resistance sensors, may be placed as shown in Figure 1 and Figure 2.