BS ISO 11443:2014

BSI Standards Publication

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

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National foreword

This British Standard is the UK implementation of ISO 11443:2014. It supersedes BS ISO 11443:2005 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/21, Testing of plastics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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INTERNATIONAL STANDARD

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Third edition
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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

Reference number
ISO 11443:2014(E)

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This third edition cancels and replaces the second edition (ISO 11443:2005), of which it constitutes a minor revision with the following changes:

— polybutene-1 (PB-1) with typical temperature range of 150 °C to 230 °C has been added in Table 3;

— normative references have been updated.

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, this International Standard can be used to characterize the rheological behaviour of such fluids for a given geometry.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1133-1, *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 1: Standard method*

ISO 1133-2, *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 2: Method for materials sensitive to time-temperature history and/or moisture*

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*

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 1 to entry: 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 1 to entry: 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 to entry: 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 to entry: 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 1 to entry: 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 $\tau / \dot{\gamma}_{ap}$ 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 $\tau_{\text{ap}} / \dot{\gamma}$ of apparent shear stress τ_{ap} to true shear rate $\dot{\gamma}$, expressed in pascal seconds (Pa·s)

Note 1 to entry: 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 1 to entry: 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 1 to entry: In certain special cases, it can 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 1 to entry: 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 lengths 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 online 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 formulae 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 can 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).

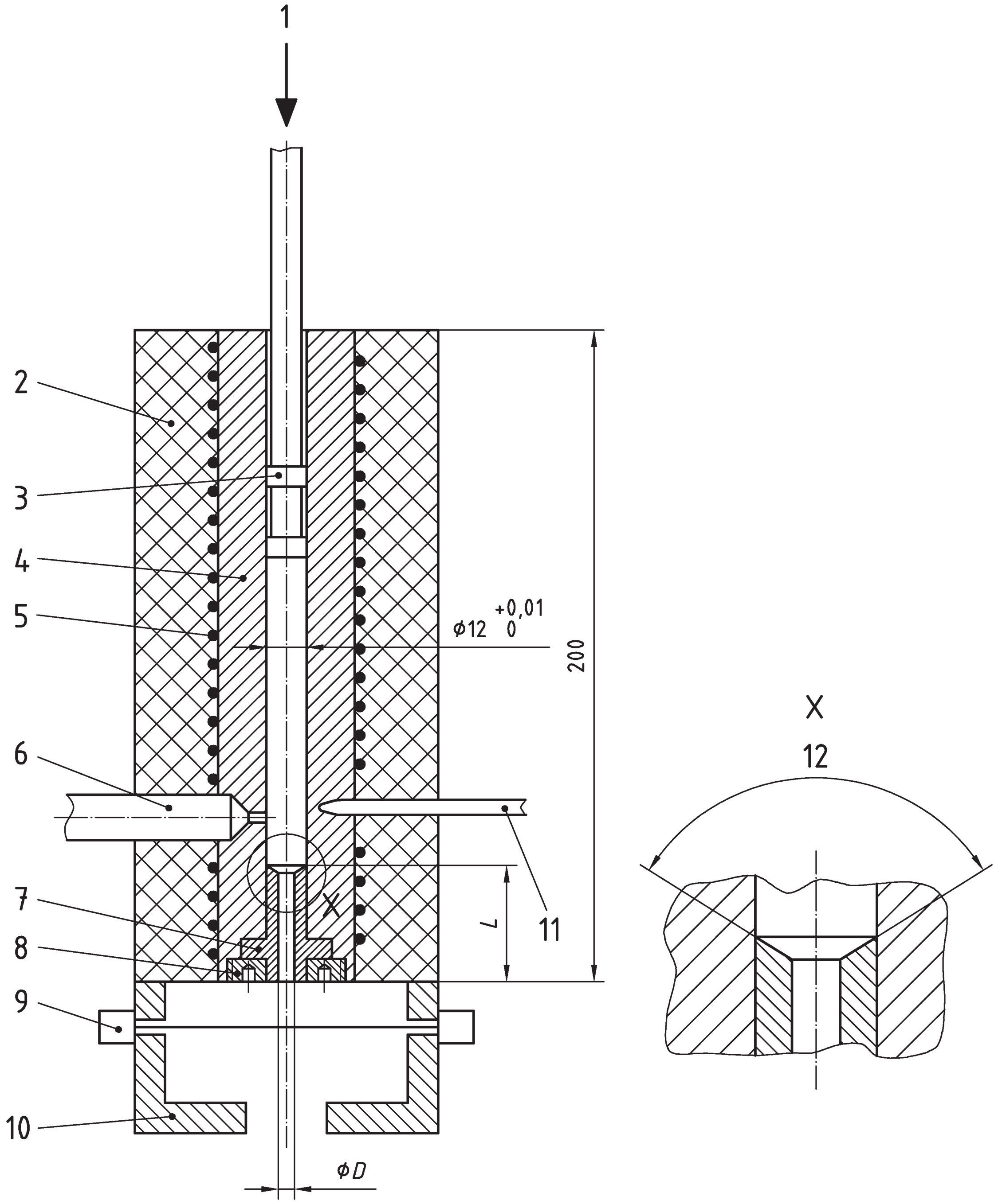
The capillary opening shall show no visible machining marks nor perceptible eccentricity.

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

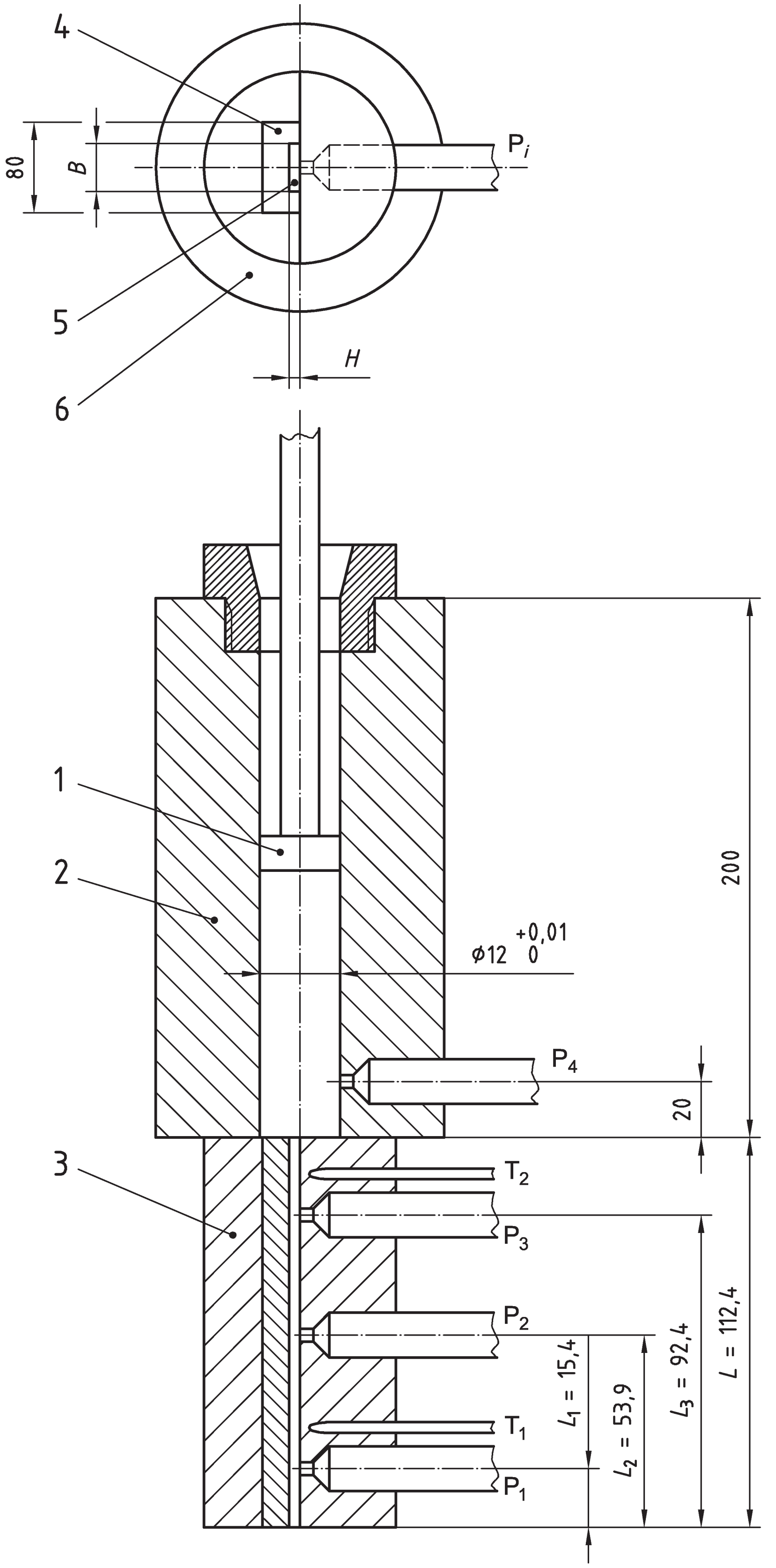


Key

1	applied force or constant velocity	7	capillary die
2	thermal insulation	8	die-retaining nut
3	piston	9	optical sensor
4	barrel	10	temperature-controlled air chamber
5	heating coil	11	thermometer
6	pressure transducer	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 $\dot{\gamma}_{\mathrm{ap}}$ 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 International 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, and 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 $L/D \leq 5$ and $L/D \geq 16$ is permitted where the test conditions are such that the resultant Bagley plot is not significantly nonlinear, 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 $\dot{\gamma}_{\mathrm{ap}}$ and the apparent shear stress τ_{ap}, unless otherwise specified by the referring International 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 can 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 can 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
≤200	±1,0	±0,5
$200 < \theta \leq 300$	±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 can be used in the thermometer well to improve conduction. Thermometers, preferably thermocouples or platinum resistance sensors, can be placed as shown in Figure 1 and Figure 2.

5.3.3 Temperature calibration

The temperature-measuring device used during the test shall read to within 0,1 °C and be calibrated by means of a standard thermometer, with error limits of ±0,1 °C, while complying with the depth of immersion prescribed for the thermometer concerned. For this purpose, the barrel can be filled to the top with a low-viscosity melt.

No liquids that can contaminate the die or barrel or influence the ensuing measurements, e.g. silicone oil, shall be used as heat transfer media during calibration.

5.4 Measurement of pressure and calibration

5.4.1 Test pressure

The test pressure shall be the pressure drop in the melt, measured as the difference between the pressure in the melt before the capillary-die or slit-die inlet and the pressure at the die exit, as applicable. If possible, the test pressure shall be measured by a melt-pressure transducer located near the entrance of the die, in which case the distance from the pressure transducer to the die entry face shall be kept constant for all tests and should preferably be not more than 20 mm (see Note). Otherwise, the test pressure shall be determined by the force exerted on the melt, e.g. by the piston, that force being measured by a load cell above the piston (see B.1).

NOTE It is important that the distance from the die entry face to the pressure transducer is kept constant for all tests as this will otherwise affect the pressure drop measured. The use of pressure transducers at a distance equivalent to that of the barrel diameter from the die entry face can reduce fluctuations in the pressure being measured that can arise due to recirculating flow above the die entry.

If testing is to be carried out by extruding to a channel or vessel pressurized to a pressure above atmospheric pressure, then the pressure at the die exit shall also be measured, preferably using a pressure transducer located immediately below the exit of the die.

The force- or pressure-measuring devices shall be operated in the range between 1 % and 95 % of their nominal capacity.

5.4.2 Pressure drop along the length of the slit die

When using slit dies, the pressure profile along the length of the die shall be measured by flush-mounted melt-pressure transducers positioned along the die wall.

Alternatively, when slit dies not equipped with melt-pressure transducers are used, the sum of entrance and exit pressure losses can be taken into account by employing the Bagley correction modified for slit dies (see 8.4.3).

5.4.3 Calibration

External hydraulic test equipment can be used for the calibration of melt-pressure transducers. Load cells shall be calibrated in accordance with manufacturer’s specifications. The maximum permissible error in the reading of the melt-pressure transducers or load cells shall be both less than or equal to 1 % of the full scale value and less than or equal to 5 % of the absolute value. The calibration of melt-pressure transducers should preferably be performed at the test temperature.

5.5 Measurement of the volume flow rate of the sample

The volume flow rate shall be determined either from the feed rate of the piston or by weighing the mass of the sample extruded during a measured period of time.

If weighing is performed, the conversion to the volume flow rate shall be made by using the density of the melt at the prevailing test temperature, the influence of the hydrostatic pressure on the density being ignored.

The volume flow rate shall be determined to within 1 %.

It is recommended that, for purposes of providing comparable data, the apparent shear rates and hence flow rates used for testing are such that data at the true shear rates specified in ISO 11403-2 can be determined by interpolation. The apparent shear rates should be set at equispaced intervals, when plotted logarithmically, and there should be at least two data points per decade of apparent shear rate.

NOTE The specified maximum permissible error for determining the volume flow rate via the feed rate of the piston can only be conformed to if, *inter alia*, the leakage rate between the piston and barrel is sufficiently small. Experience indicates that this can be achieved if the clearance between piston and barrel does not exceed 0,045 mm (see 5.1.5).

6 Sampling

From the material to be tested, a representative sample shall be taken for use as the test sample. The number of determinations per single barrel filling depends on the moulding material under test and shall therefore be agreed upon between the interested parties. The temperature during test sample preparation shall be less than that during the subsequent test.

7 Procedure

7.1 Cleaning the test device

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

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

NOTE For the purpose of cleaning, circular brushes made of a copper/zinc alloy (brass) and linen cloths have proved satisfactory. However, the use of copper-containing materials can accelerate degradation of the polymer when testing polyethylene and polypropylene. Cleaning can also be performed by cautious burning out. Using graphite on threads facilitates unlocking after the test.

WARNING — The operating conditions chosen can entail partial decomposition of the material under test, or cause it to release dangerous volatile substances. The users of this International Standard shall make themselves aware of possible risks, shall prevent or minimize such risks as appropriate, and shall provide appropriate means of protection.

7.2 Selection of test temperatures

It is recommended that, for the purpose of providing data for comparison or for modelling, data at three temperatures be obtained (see ISO 11403-2). For any given material type, one of the temperatures used should preferably be the same as that specified in the appropriate material designation or specification standard for use in melt flow rate testing (see ISO 1133-1 and ISO 1133-2). For the other two temperatures, it is recommended that a temperature interval of 20 °C be used (see Notes 1 and 2). Both of the additional temperatures can be either higher or lower than the recommended temperature as used for the melt flow rate test (see ISO 1133-1 and ISO 1133-2), or one higher and one lower. However, other values can be used and can be preferable to use, depending on the specific grade of material and the application for which the data are required.

NOTE 1 From an analysis of CAMPUS databases, the average interval in temperature used to measure shear viscosity ranged from 10 °C to 30 °C and was dependent on the material grade.

NOTE 2 Typical test temperatures for several materials are given in Table 3. These are listed for information only. The most useful data are generally obtained at the temperatures used for processing the material. The shear stresses and shear rates applied are also intended to closely approximate those observed in the actual processing.

Table 3 — Typical test temperatures

Material	Temperature °C
Polyacetal	190 to 220
Polyacrylate	140 to 300
Polybutene-1 (PB-1)	150 to 230
Acrylonitrile-butadiene-styrene (ABS)	200 to 280
Cellulose esters	190
Polyamide PA66	250 to 300
Polyamide (not PA66)	190 to 300
Poly(chlorotrifluoroethylene)	265
Polyethylene and ethylene copolymers and terpolymers	150 to 250
Polycarbonate	260 to 300
Polypropylene	190 to 260
Polystyrene and styrene copolymers	180 to 280
Poly(vinyl chloride)	170 to 210
Poly(butylene terephthalate)	245 to 270
Poly(ethylene terephthalate)	275 to 300
PMMA and copolymers	180 to 300
Poly(vinylidene fluoride)	195 to 240
Poly(vinylidene chloride)	150 to 170
Ethylene-vinyl alcohol copolymer	190 to 230
Polyetheretherketone	340 to 380
Polyethersulfone	360

7.3 Preparation of samples

In cases where the fluidity of the melt depends on one or more factors, such as the residual monomer content, gas inclusions, and/or moisture, apply pretreatment or conditioning procedures in accordance with the referring International Standard and/or the relevant material standard, as applicable.

NOTE Examples of materials that can require special preparation regimes include poly(ethylene terephthalate), poly(butylene terephthalate), and polycarbonate.

Allow the assembled apparatus to reach thermal equilibrium at the test temperature before applying the final torque on the die (where applicable), then start charging (see Warning in 7.1).

To avoid air inclusions, 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 not more than 2 min.

7.4 Preheating

Immediately after charging the barrel, start the preheat timer. Either extrude a small portion of the barrel charge at a constant pressure (method 1) or apply a constant volume flow rate until a positive load or pressure is obtained (method 2). Then stop the extrusion or volume flow until a preheat time of at least 5 min, unless otherwise specified by the referring International Standard, is completed. 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, either by ensuring that on increasing the preheat time, the measured quantity (volume flow rate or test pressure, as applicable) at constant test conditions does not change by more than ±5 %, or by inserting a thermometer into the sample in the barrel and ensuring that, within the specified preheat time, the sample temperature is equal to the specified test temperature within the tolerance for the distance-related temperature difference 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.

7.5 Determination of the maximum permissible test duration

To check that degradation or other processes are not affecting measurements, carry out a repeat measurement towards the end of the test on the same barrel charge, using the same conditions as were used at the beginning of the test. Compare values obtained at the start and end of the test. A difference in values is indicative of degradation or other processes affecting measurements.

Alternatively, for each sample and each test temperature, determine by testing, employing several different preheating times prior to the actual tests, the maximum permissible test duration which corresponds to the time span, from the end of charging of the barrel, within which the measured quantity (volume flow rate or test pressure, as applicable) at constant test conditions does not change by more than ±5 % (see also 7.4.)

If determination at all of the required values of test pressure or volume flow rate is not possible within the maximum permissible test duration of a single test, then make measurements stage by stage, using several barrel fillings of the same sample (see Note 1 to 7.8).

NOTE For materials that are unstable, in order to minimize the effect of changes on the measurements, it is recommended that testing be carried out using a decreasing speed profile. The degree of compaction of the sample can also influence its stability.

7.6 Determination of test pressure at constant volume flow rate: Method 2

If the test pressure necessary to maintain a given volume flow rate is to be determined (see also 5.4.1 and 7.8), use either of the following methods (see Table 1):

- method A2, using capillary dies;
- method B2, using slit dies.

7.7 Determination of volume flow rate at constant test pressure: Method 1

If, as an alternative to 7.6, the volume flow rate for a given test pressure drop is required (see also 7.8), use either of the following methods (see Table 1):

- method A1, using capillary dies;
- method B1, using slit dies.

7.8 Waiting periods during measurement

At each measurement, wait until the test pressure (method A2 or B2) or the volume flow rate (method A1 or B1) has become constant (e.g. to ±3 %) over a given time period (e.g. 15 s).

NOTE 1 With a single barrel filling, it is generally possible to determine several pairs of values for volume flow rate and test pressure.

NOTE 2 It is recommended that selected measurements are repeated to check the repeatability.

7.9 Measurement of extrudate swelling

7.9.1 General

Measure the degree of extrudate swelling either at the test temperature during the extrusion process, or after cooling of the extruded strand to room temperature.

NOTE The diameter of the extrudate is dependent on the flow rate, the test temperature, the time since extrusion, the manner of cooling (for the ratio at room temperature), the length of the extrudate, the capillary die length, diameter, and entry geometry, and the barrel diameter. The results obtained can be very sensitive to the details of the measurement technique. Comparable results can only be obtained when all testing conditions are identical.

The following procedures give a measure of the degree of extrudate swelling. Other methods can be used. Although the procedures described are written for capillary dies, they also apply by analogy to slit dies.

7.9.2 Measurement at room temperature

The diameter of the extruded strand is measured with a micrometer. In order to minimize the effects of gravity, use the following procedure:

- remove any extrudate attached to the capillary die by cutting it off as close as possible to the die;
- extrude a length of extrudate not longer than 5 cm and cut off the length of extrudate, marking the end that was extruded first;
- when cutting off the length of extrudate, hold it with tweezers and subsequently allow it to cool, suspended in air, to room temperature;
- measure the diameter of the strand as close as possible to the marked end (outside the area deformed by cutting and marking).

7.9.3 Measurement at the test temperature

Use a photographic or optical method that involves no mechanical contact with the extruded strand. In order to minimize the effect of gravity, use the following procedure:

- remove any extrudate attached to the capillary die by cutting it off as close as possible to the die;
- extrude a length of extrudate not longer than 5 cm;
- measure the diameter of the extruded strand at a fixed point below the die outlet by photographic or optical techniques.

NOTE In order to minimize cooling of the extruded strand during the measurement of extrudate swelling, the strand can be extruded into a temperature-controlled air chamber, such as that shown schematically in Figure 1.

8 Expression of results

8.1 Volume flow rate

Calculate the volume flow rate Q, in cubic millimetres per second, by means of one of the following formulae:

$$Q = Av \tag{1}$$

or

$$Q = \frac{\dot{m}}{\rho} \tag{2}$$

where

A is the piston cross-sectional area, in square millimetres;

v is the velocity of the piston, in millimetres per second;

$\dot{m}$ is the mass flow rate of the sample, in grams per second;

ρ is the density of the sample at the test temperature, in grams per cubic millimetre.

8.2 Apparent shear rate

8.2.1 General

Calculate the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$, in reciprocal seconds, at the die wall, using the formula given in 8.2.2 or 8.2.3, as applicable.

8.2.2 Method A: Capillary dies

$$\dot{\gamma}_{\mathrm{ap}} = \frac{32Q}{\pi D^3} \tag{3}$$

where

D is the diameter of the capillary die bore, in millimetres;

Q is the volume flow rate, in cubic millimetres per second (see 8.1).

NOTE In the case of Newtonian fluids, Formula (3) gives the true shear rate $\dot{\gamma}$ at the capillary wall. As plastics melts do not generally exhibit Newtonian behaviour, the quantity calculated using this formula is termed the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$. The true shear rate $\dot{\gamma}$ can be determined from the apparent one $\dot{\gamma}_{\mathrm{ap}}$ by a correction procedure (see 8.5.1).

8.2.3 Method B: Slit dies

$$\dot{\gamma}_{\text{ap}} = \frac{6Q}{BH^2} \tag{4}$$

where

B is the width of the die, in millimetres;

H is the thickness of the die, in millimetres;

Q is the volume flow rate, in cubic millimetres per second (see 8.1).

See Note to 8.2.2.

NOTE Formula (4) is strictly true only for infinitesimally small thickness-to-width (H/B) ratios. The use of Formula (4) overestimates the apparent shear rate $\dot{\gamma}_{\text{ap}}$ by less than 3 %, however, if $H/B < 0{,}1$. A detailed analysis of the correctness of the approximation involved in using Formula (4), together with a correction procedure, is given in Annex A.

8.3 Apparent shear stress

8.3.1 General

Calculate the apparent shear stress τ_{ap}, in pascals, at the die wall, using the formula given in 8.3.2 or 8.3.3, as applicable.

8.3.2 Method A: Capillary dies

$$\tau_{\text{ap}} = \frac{pD}{4L} \tag{5}$$

where

p is the test pressure, in pascals;

L is the length of the die, in millimetres;

D is the diameter of the die, in millimetres.

8.3.3 Method B: Slit dies

$$\tau_{\text{ap}} = \frac{HB}{2(H+B)} \times \frac{p}{L} \tag{6}$$

where

p is the test pressure above the die inlet, in pascals;

L is the length of the die, in millimetres;

B is the width of the die, in millimetres;

H is the thickness of the die, in millimetres.

NOTE The shear stresses calculated using Formulae (5) and (6) are apparent quantities because the pressure drop along the length of the die is smaller than the test pressure p which is the sum of the pressure losses at the die entrance, in the die, and at the die exit. True shear stresses can be determined by applying appropriate corrections either to the test pressure p or to the die length L (see 8.4).

8.4 True shear stress

8.4.1 General

The true shear stress can be obtained by using the Bagley correction method[3] (see 8.4.2 or 8.4.3, as applicable), or can be determined directly if slit dies (methods B1 and B2) equipped with pressure transducers are used (see 8.4.4).

If nonlinear Bagley or slit-die pressure-drop versus distance plots are obtained, a statement shall be made to that effect in the test report. In such cases, shorter dies shall be used, unless otherwise specified by agreement, in which case the procedure used shall be stated in the test report.

NOTE In using capillary-die or slit-die extrusion rheometers for measuring the shear viscosity of plastics, viscous dissipation and the pressure dependence of the viscosity can affect the results. Nonlinear plots can result.

8.4.2 Bagley correction for capillary dies (method A)

Determine the sum of the entrance and exit pressure losses using the following procedure.

a) For method A1, using at least two, but preferably more, capillary dies of the same inlet angle and diameter but with different L/D ratios such that $(L/D)_1 < (L/D)_2$, determine the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ at the die wall as a function of the test pressure p (see Figure 3).

b) For method A2, using at least two, but preferably more, capillary dies of the same inlet angle and diameter but with different L/D ratios such that $(L/D)_1 < (L/D)_2$, measure the test pressure p as a function of the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ at the die wall.

c) From the data obtained in a) or b), as applicable, plot the test pressure p as a function of L/D for different values of the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ (see Figure 4). The so-called Bagley lines which result have a slope that is four times the true shear stress.

If, when using long capillary dies, deviations from the straight line arise due to the influence of pressure on the melt viscosity or due to viscous-dissipation effects, make the measurements using shorter capillary dies, unless otherwise specified by agreement, in which case the procedure used shall be stated in the test report (see Note to 8.4.1).

NOTE The Bagley correction can be performed using suitable computer programs. It is then not necessary to follow the data-plotting procedure described above. However, if computers are used to apply corrections to measured data, a graphic printout of the Bagley plots can enable the operator to assess the validity of the assumptions made (i.e. to check that the Bagley lines are straight).

Extrapolate the Bagley line for each apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ to zero pressure (see Figure 4). The ordinate distance p_{c} corresponds to the sum of the die-entrance and die-exit pressure losses at the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ concerned.

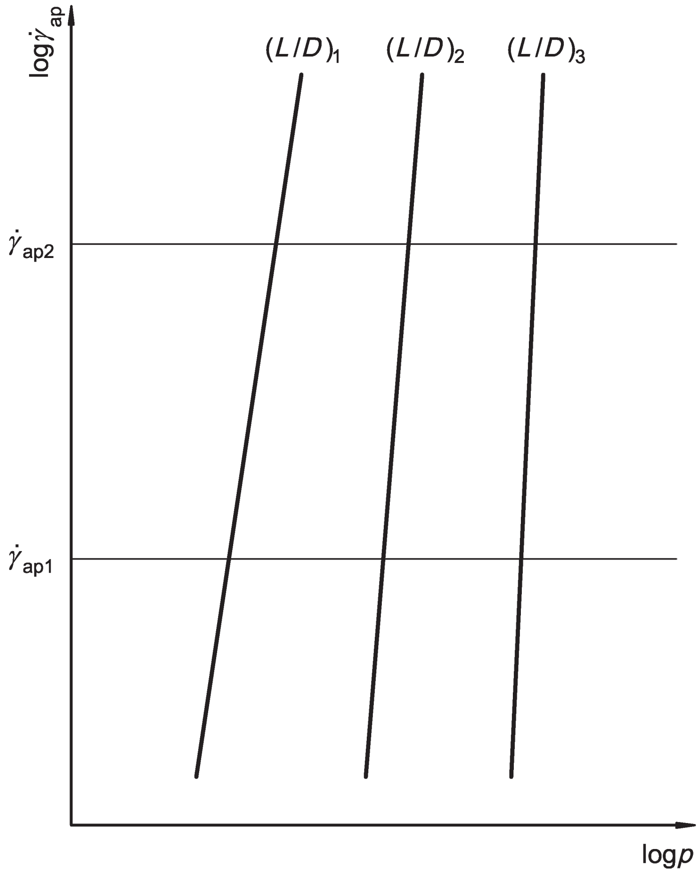
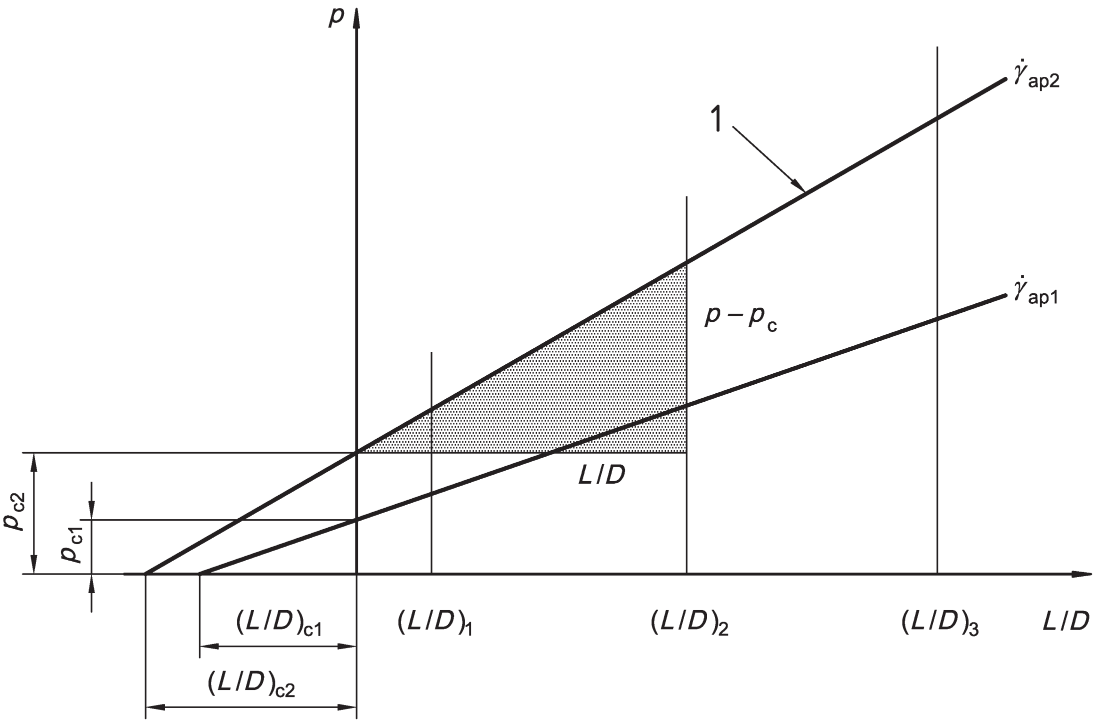


Figure 3 — Application of the Bagley correction method[3] — Plot of the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ versus the test pressure p for different values of L/D



Key

1 slope = 4τ

NOTE The melt pressure p is plotted as a function of L/D for dies of the same diameter for different values of the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$.

Figure 4 — Schematic Bagley plot for capillary dies

Calculate the true shear stress τ for the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ of interest using either Formula (7) or Formula (8):

$$\tau=\left(p-p_{\mathrm{c}}\right)\frac{D}{4L} \tag{7}$$

where

p is the test pressure, in pascals;

p_{c} is the pressure correction, in pascals;

D is the diameter of the capillary die, in millimetres;

L is the length of the capillary die, in millimetres.

Because the diameter D of the die is constant, the abscissa distances $(L/D)_{\mathrm{c}}$ represent die-length corrections. Thus, as an alternative to Formula (7), the true shear stress τ for the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ of interest can be calculated using Formula (8):

$$\tau=\frac{p}{4\left[\left(L/D\right)+\left(L/D\right)_{\mathrm{c}}\right]} \tag{8}$$

where $(L/D)_{\mathrm{c}}$ is the die-length correction (dimensionless).

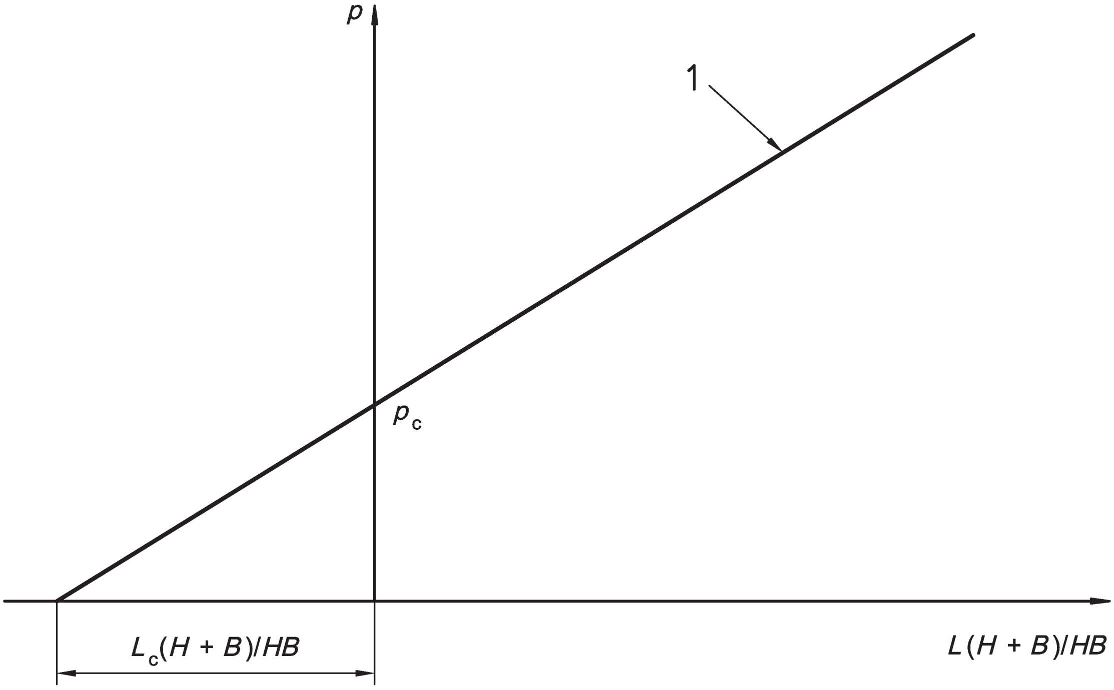
8.4.3 Bagley correction for slit dies (method B)

Determine the sum of the entrance and exit pressure losses using the following procedure.

a) For method B1, using at least two, but preferably more, slit dies of the same inlet angle, width, and thickness, but of different lengths L such that L_1 is less than L_2, determine the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ at the die wall as a function of the test pressure p.

b) For method B2, using at least two, but preferably more, slit dies of the same inlet angle, width, and thickness, but of different lengths L such that L_1 is less than L_2, measure the test pressure p as a function of the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ at the die wall.

c) From the data obtained in a) or b), as applicable, plot the test pressure p as a function of $L(H + B)/HB$ for different values of the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$. The Bagley lines which result has a slope that is twice the true shear stress (see Figure 5).



Key

1 slope = 2τ

NOTE The test pressure p is plotted as a function of $L(H + B)/HB$ for dies of the same width B and thickness H for a single value of the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$.

Figure 5 — Schematic Bagley plot for slit dies

If, when using long slit dies, deviations from the straight line arise due to the influence of pressure on melt viscosity or due to viscous-dissipation effects, make the measurements using shorter slit dies, unless otherwise specified by agreement, in which case the procedure used shall be stated in the test report (see Notes to 8.4.1 and 8.4.2).

Extrapolate the Bagley line for each apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ to zero pressure. The ordinate distance p_{c} corresponds to the sum of the die-entrance and die-exit pressure losses at the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ concerned.

Calculate the true shear stress τ for the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ of interest using either Formula (9) or Formula (10):

$$\tau = \frac{HB}{2(H+B)} \times \frac{(p-p_{\mathrm{c}})}{L} \tag{9}$$

where

H is the thickness of the slit die, in millimetres;

B is the width of the slit die, in millimetres;

p is the test pressure, in pascals;

p_{c} is the pressure correction, in pascals;

L is the length of the slit die, in millimetres.

Because the die dimensions H and B are constant, the abscissa distances $L_{\mathrm{c}}(H + B)/HB$ represent die-length corrections. Thus, as an alternative to Formula (9), the true shear stress τ for the apparent shear rate $\dot{\gamma}_{\mathrm{ap}}$ of interest can be calculated using Formula (10):

$$\tau = \frac{p}{2(L+L_{\mathrm{c}})} \times \frac{HB}{(H+B)} \tag{10}$$

where $L_{\mathrm{c}}(H + B)/HB$ is the die-length correction (dimensionless).

8.4.4 Direct determination using slit dies (method B)

From the longitudinal pressure gradient $\mathrm{d}p/\mathrm{d}L$ measured using pressure transducers placed along the length of the slit die, calculate the true shear stress τ at the die wall using the following formulae:

$$\tau = \frac{HB}{2(H+B)} \times \frac{\mathrm{d}p}{\mathrm{d}L} \tag{11}$$

where

$\frac{\mathrm{d}p}{\mathrm{d}L}$ is the longitudinal pressure gradient, in pascals per millimetre;

B is the width of the slit die, in millimetres;

H is the thickness of the slit die, in millimetres.

8.5 True shear rate

8.5.1 General

Calculate the true shear rate $\dot{\gamma}$ at the capillary-die or slit-die wall from the apparent shear rate by applying the Weissenberg-Rabinowitsch correction method[4], using Formula (12) for method A (see 8.5.2) and Formula (13) for method B (see 8.5.3).

8.5.2 Method A: Capillary dies

$$\dot{\gamma} = \frac{\dot{\gamma}_{\mathrm{ap}}}{4}\left(3 + \frac{\mathrm{d}\log\dot{\gamma}_{\mathrm{ap}}}{\mathrm{d}\log\tau}\right) \tag{12}$$

where $\frac{\mathrm{d}\log\dot{\gamma}_{\mathrm{ap}}}{\mathrm{d}\log\tau}$ is the slope of the curve $\log\dot{\gamma}_{\mathrm{ap}} = f(\log\tau)$.

NOTE It is noted that the method by which this correction is applied, specifically either the choice of the function used to fit the $\log\dot{\gamma}_{\mathrm{ap}}$ versus $\log\tau$ data from which the slope is determined or the use of an alternative method of determination of the slope of the data, can result in significant errors in the corrected (true) values of shear rate, and consequently true shear viscosity. This is particularly the case when the slope of the curve is large or where the selected curve does not fit the data well, for example at the highest and lowest shear rate data points.

8.5.3 Method B: Slit dies

$$\dot{\gamma} = \frac{\dot{\gamma}_{\mathrm{ap}}}{3}\left(2 + \frac{\mathrm{d}\log\dot{\gamma}_{\mathrm{ap}}}{\mathrm{d}\log\tau}\right) \tag{13}$$

where $\frac{\mathrm{d}\log\dot{\gamma}_{\mathrm{ap}}}{\mathrm{d}\log\tau}$ is as defined in 8.5.2.

See Note to 8.5.2.

8.6 Viscosity

Calculate the viscosity as the ratio of the shear stress to the shear rate.

If the ratio is not derived exclusively from true quantities of shear stress and shear rate, then one of several apparent viscosities will result. These shall be named and identified by subscripts as defined in 3.8 to 3.10.

8.7 Determination of extrudate swelling

8.7.1 Measurement at room temperature

Calculate the extrudate swell ratio at room temperature S_{a} and the percent swell at room temperature s_{a}, using Formulae (14) and (15):

$$S_{\mathrm{a}} = \frac{D_{\mathrm{a}}}{D} \tag{14}$$

$$s_{\mathrm{a}} = \frac{D_{\mathrm{a}} - D}{D} \times 100\,\% \tag{15}$$

where

D_{a} is the extrudate diameter, in millimetres, measured at room temperature;

D is the capillary-die diameter, in millimetres, measured at room temperature.

8.7.2 Measurement at the test temperature

Calculate the swell ratio at the test temperature S_{T} and the percent swell at the test temperature s_{T}, using Formulae (16) and (17):

$$S_{\mathrm{T}} = \frac{D_{\mathrm{m}}}{D_{\mathrm{T}}} \tag{16}$$

$$s_T = \frac{D_m - D_T}{D_T} \times 100\,\% \tag{17}$$

where

D_m is the extrudate diameter, in millimetres, measured at the test temperature;

D_T is the capillary-die diameter, in millimetres, measured at the test temperature.

NOTE In the case of slit dies, these calculations can be made using the thickness (or width) of the extrudate instead of the extrudate diameter and the die thickness (or die width) instead of the die diameter in Formulae (14) to (17). Since swelling can be different in the width and thickness directions, it should preferably be determined in both of these directions.

9 Precision

Two interlaboratory test programmes have been carried out. The first interlaboratory test programme was completed in 1990, involving seven laboratories and two materials (PP and PVC).

In the first interlaboratory comparison, two types of apparatus and two measurement procedures were used:

- a rheometer measuring the extrusion pressure at the capillary inlet (four laboratories) and a rheometer measuring the force applied to the piston (two laboratories);
- the shear rates applied during the tests were imposed successively in decreasing order of magnitude (two laboratories) or increasing order (four laboratories).

Repeatability was examined by two laboratories, and was found to be improved if the pressure was measured at the inlet to the capillary rather than determined from the force applied to the piston, and to be less good at low shear rates (<100 s^{-1}) than at high shear rates (>100 s^{-1}). Estimated repeatability was ±10 % and ±5 %, respectively. If long dies are used ($L/D > 20$), the effect of the geometry of the capillary inlet can be disregarded if the inlet angle is ≥90°.

The reproducibility of the method was estimated by measuring, in seven laboratories, the viscosity of PVC at 180 °C and 190 °C and that of PP at 210 °C and 240 °C. Reproducibility was found to be poorer at low shear rates (<100 s^{-1}) than at high shear rates (>100 s^{-1}), being ±20 % and ±10 %, respectively.

From an examination of the results, it can be seen that reproducibility is affected by

- the order in which the various shear rates are examined during a single test;
- the sensitivity of the pressure-sensor or force-sensor used: measurements cannot be carried out with the same precision at high pressures (high shear rates) and at low pressures (low shear rates) using the same sensor;
- the method used to determine the shear stress: measurement of pressure at the capillary inlet is preferred since it is more accurate.

The effect of the cleanliness of the capillary on the results was not investigated in these tests.

In the second interlaboratory test programme, completed in 1996, 20 laboratories took part in total, using polyethylene (PE) and glass-fibre-filled polypropylene (GFPP) samples[6]. The precision data presented in Table 4 on the measurement and determination of extrusion pressure, entrance pressure drop, and shear viscosity corrected for both entrance effects and non-Newtonian velocity profile were determined. Values presented are for 95 % confidence levels, these values having been determined using a factor of 2,8 times the calculated standard deviation values.

NOTE 1 The contraction ratio is defined as the ratio of the barrel diameter to the die diameter.

NOTE 2 The standard deviations and repeatability and reproducibility limits (95 % confidence values) were determined in accordance with Reference [7].

NOTE 3 See also Annexes A to C.

Table 4 — Precision data for extrusion rheometry

<table>
<tr><th colspan="4">Extrusion pressure measurement</th></tr>
<tr><td>Material</td><td colspan="2">Polyethylene</td><td>Glass-fibre-filled polypropylene</td></tr>
<tr><td>Test temperature, °C</td><td colspan="2">190</td><td>230</td></tr>
<tr><td>Repeatability (95 % confidence)</td><td colspan="2">20 %</td><td>38 %</td></tr>
<tr><th colspan="4">Shear viscosity measurement [corrected for entrance pressure drop and non-Newtonian velocity profile (Weissenberg-Rabinowitsch correction)]</th></tr>
<tr><td>Test temperature, °C</td><td colspan="2">190</td><td>230</td></tr>
<tr><td>Repeatability (95 % confidence)</td><td colspan="2">20 %</td><td>24 %</td></tr>
<tr><td>Reproducibility (95 % confidence)</td><td colspan="2">28 %</td><td>34 %</td></tr>
<tr><th colspan="4">Entrance pressure drop measurement</th></tr>
<tr><td>Material</td><td>Polyethylene</td><td>Polyethylene</td><td>Glass-fibre-filled polypropylene</td></tr>
<tr><td>Test temperature, °C</td><td>190</td><td>190</td><td>230</td></tr>
<tr><td>Contraction ratio</td><td>15</td><td>9,55 to 15,5</td><td>15</td></tr>
<tr><td>Reproducibility (95 % confidence)</td><td>42 %</td><td>50 %</td><td>56 %</td></tr>
</table>

10 Test report

10.1 General

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

a) a reference to this International Standard and any referring standards;

b) the information specified in 10.2, 10.3, and 10.4, as applicable;

c) the date of the test.

10.2 Test conditions

a) a description of the material under test;

b) details of any conditioning or preparation of the material or sample, e.g. drying or compounding;

c) the method used (A1, A2, B1, or B2);

d) a description of the rheometer used and its barrel diameter, D_b;

e) the diameter D, length L, and ratio of length to diameter L/D of the straight section of the capillary die used, and the degree of precision of these measurements, if applicable;

f) the thickness H, width B, and length L of the slit die used, and the degree of precision of these measurements, if applicable;

g) a description of the capillary-die or slit-die inlet angle profile;

h) a description of the technique used to measure the extrudate swelling;

i) the temperature at which the measurements were made;

j) the pressure below the die exit when extruding to pressures other than atmospheric pressure, a description of the method used to obtain and measure this pressure, and the precision of measurement of the pressure, if applicable;

k) the sample preheating time;

l) the dwell time;

m) the dwell time corresponding to the appearance of an alteration in the material, if applicable;

n) the maximum permissible test duration, if applicable;

o) the extrusion time;

p) details of any deviations from the requirements of this International Standard and any incidents likely to have influenced the results.

10.3 Flow characteristics

10.3.1 General

Report whether the shear rate, the shear stress, and the viscosity are “apparent” or “true” values.

Report the method of viscosity determination if the Bagley or pressure drop versus distance plots are nonlinear.

For plastics which are not wall-adhering, present the results in the form of apparent shear stress plotted as a function of flow rate Q, or vice versa.

10.3.2 Graphical representation

The following plots can be included, as necessary:

- log shear stress versus log shear rate, or vice versa;
- log viscosity versus log shear stress or log shear rate;
- log viscosity versus the reciprocal of absolute temperature at constant shear stress or shear rate;
- log viscosity versus temperature in °C at constant shear stress or shear rate;
- log critical shear stress or log critical shear rate for each of the observed effects (see 3.19 and 3.20) versus the reciprocal of absolute temperature;
- log critical shear stress or log critical shear rate for each of the observed effects (see 3.19 and 3.20) versus temperature in °C;
- log volume flow rate versus log shear stress, or vice versa;
- pressure versus die length;
- pressure versus distance of pressure transducer from die exit (slit dies);
- log pressure correction versus log shear stress or log shear rate or log volume flow rate;
- capillary-die or slit-die inlet and exit pressure loss versus shear stress or shear rate or volume flow rate;

- swell ratio at room temperature or at the test temperature versus shear rate or volume flow rate;
- percent swell at room temperature or at the test temperature versus shear rate or volume flow rate.

Apparent and/or true values of shear rate, shear stress, and viscosity can also be presented.

10.3.3 Individual values

These can be given for a given series of test conditions, as necessary:

- shear stress, in pascals;
- shear rate, in reciprocal seconds;
- viscosity, in pascal seconds;
- swell ratio at room temperature;
- percent swell at room temperature;
- swell ratio at the test temperature;
- percent swell at the test temperature.

Apparent and/or true values of shear rate, shear stress, and viscosity can also be presented.

10.4 Visual examination

If visual examination is possible, report any change in the surface appearance of the extrudate (e.g. break in flow, distortion of extrudate), noting the test conditions at which the change occurs.

Such changes can correspond to the critical shear stresses. In this case, note the values individually in the test report as “visual” critical shear stresses.

In addition, if the material changes colour, report the corresponding dwell time.

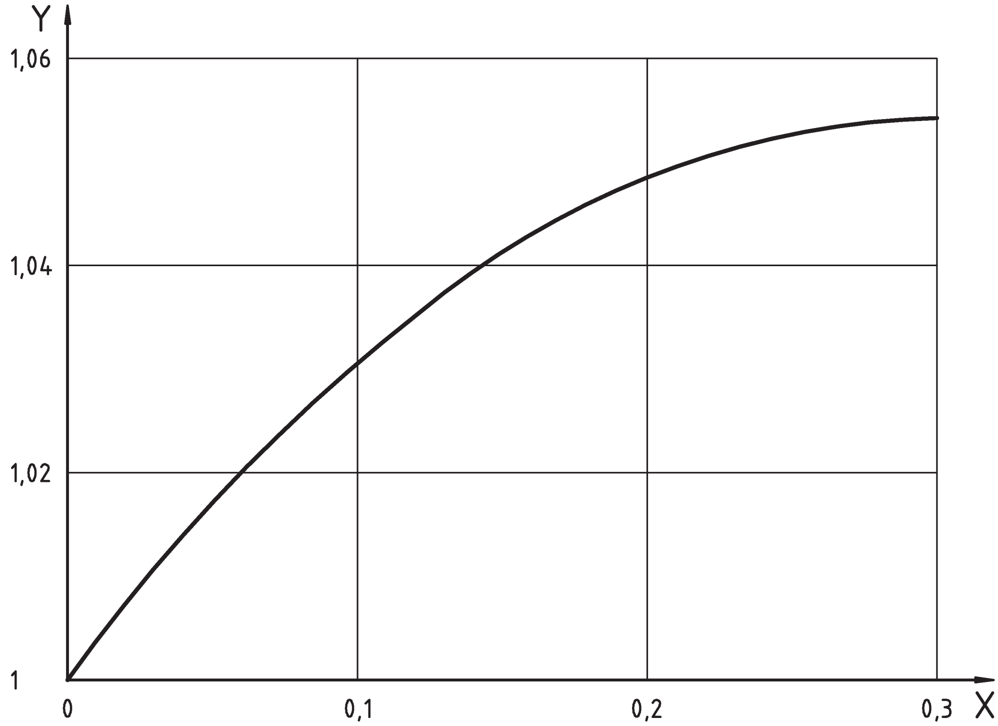
Annex A
(informative)

Method of correcting for the influence of *H*/*B* on the apparent shear rate

Formula (4) given in 8.2.3 for the apparent shear rate is valid only for an infinitely wide slit. Formula (4) therefore gives the apparent shear rate which will exist when the volume flow rate in the die-length direction is Q over a distance B in the die-width direction and assuming that no flow takes place in the width direction or in the thickness direction. At finite ratios of H/B, Formula (4) is still a good approximation, as shown in Figure A.1. This shows the ratio of the apparent shear rates obtained, at identical volume flow rates Q, from Formula (4) and from Formula (A.1), a corrected formula given in Reference [5]:

$$\dot{\gamma}_{\mathrm{ap}}^{\mathrm{c}} = \frac{QBH}{2(B+H)\left[\frac{BH^3}{12} - \frac{16H^4}{5}\sum_{n=1}^{5}\left(\frac{1}{n^5}\tanh\frac{n\pi B}{2H}\right)\right]} \qquad \text{(A.1)}$$

where n is an odd integer.



Key

X thickness-to-width ratio H/B

Y shear rate ratio $\dot{\gamma}_{\mathrm{ap}} / \dot{y}_{\mathrm{ap}}^{\mathrm{c}}$

Figure A.1 — Shear-rate ratio $\dot{\gamma}_{\mathrm{ap}} / \dot{y}_{\mathrm{ap}}^{\mathrm{c}}$ versus thickness-to-width ratio *H*/*B*

Dividing Formula (4) by Formula (A.1) gives Formula (A.2):

$$\frac{\dot{\gamma}_{\mathrm{ap}}}{\dot{\gamma}_{\mathrm{ap}}^{\mathrm{c}}}=\left(1+\frac{H}{B}\right)\left[1-0{,}627\,4\,\frac{H}{B}\sum_{n=1}^{5}\left(\frac{1}{n^{5}}\tanh\frac{n\pi B}{2H}\right)\right] \tag{A.2}$$

which expresses the ratio of the apparent shear rates as a function of the thickness-to-width ratio H/B.

The summation term in Formula (A.2) is 1,004 4 when $H/B \leq 0{,}3$. Thus the corrected apparent wall shear rate is given (using Formula 4) by

$$\dot{\gamma}_{\mathrm{ap}}^{\mathrm{c}}=\frac{6Q}{BH^{2}}\left[\left(1+\frac{H}{B}\right)\left(1-0{,}630\,\frac{H}{B}\right)\right]^{-1} \tag{A.3}$$

The error introduced by using Formula (4) instead of Formula (A.3) is less than 3 % for thickness-to-width ratios less than 0,1.

Annex B
(informative)

Measurement errors

B.1 Error due to piston friction

Friction is caused by contact between the piston and the barrel. Usually, the effect of the frictional force thus produced is negligible compared with the pressure drop through the capillary or slit die. It should, however, be confirmed that the frictional force is negligible by carrying out a dry run at the test temperature.

This precaution can be disregarded if measurements are made at constant velocity, and pressure is measured using a pressure sensor placed in the immediate vicinity of the die inlet.

B.2 Error due to back-flow of material

The clearance between the piston head and barrel can allow a small quantity of the sample to flow back past the piston instead of through the capillary or slit die. This results in a measured shear rate lower than that calculated from the piston velocity. Usually, this error is negligible. In certain cases, however, particularly when the piston is operating at low speed and with a sizeable load, a correction can be necessary. Material which has flowed back past the piston head is collected and weighed, and its mass compared with that of the extrudate produced during the same time interval, in order to determine the percentage error due to back-flow.

B.3 Error due to melt compressibility

Some liquids exhibit very high compressibility. Given that the shear rate at the die wall is calculated from the piston travel speed, an error is therefore introduced owing to the drop in hydrostatic pressure (and hence the decrease in density) along the length of the die. With the decrease in density of the liquid, the flow velocity increases, resulting in an increase in shear rate towards the exit of the die.

B.4 Error due to non-zero liquid velocity at the die wall

The calculations relating to flow in the die are based on the assumption that the velocity of the liquid is zero at the die wall. For high-viscosity polymer melts, slippage can occur between the polymer and die wall.

Annex C
(informative)

Uncertainties in the determination of shear viscosity by capillary extrusion rheometry testing

C.1 Analysis of uncertainties

The combined uncertainty $u_c(y)$ of the measurand y (the quantity to be measured) can be determined from the partial derivatives of the function $y = f(x_i)$ and the uncertainties $u(x_i)$ in the parameters x_i of that function. Assuming that individual uncertainty sources are uncorrelated, the combined uncertainty $u_c(y)$ can be computed using the root sum squares:

$$u_c(y) = \sqrt{\sum_{i=1}^{m}[c_i u(x_i)]^2} \tag{C.1}$$

where

c_i is the sensitivity coefficient (partial derivative) associated with the quantity x_i;

$u(x_i)$ is the uncertainty in that quantity.

The combined uncertainty $u_c(y)$ corresponds to one standard deviation and therefore has an associated confidence level of approximately 68 %. Assuming a normal distribution, then an expanded uncertainty U, equivalent to a 95 % confidence level, can be determined using a coverage factor of 2, i.e. twice the combined uncertainty value. The relative uncertainty is the ratio of the uncertainty in the parameter to the value of the parameter.

To determine the uncertainties, an expression relating the shear viscosity to the measurement parameters shall first be derived. From Formulae (3), (7), and (12), 3.7, and the formulae:

$$\dot{\gamma} = \dot{\gamma}_{\mathrm{ap}}\left[\frac{3n+1}{4n}\right] \tag{C.2}$$

$$n = \left[\frac{\mathrm{d}\log\dot{\gamma}_{\mathrm{ap}}}{\mathrm{d}\log\tau}\right]^{-1} \tag{C.3}$$

$$\dot{\gamma}_{\mathrm{ap}}=\frac{32Q}{\pi D^3} \tag{C.4}$$

and

$$Q=\frac{v\pi {D_{\mathrm{b}}}^2}{4} \tag{C.5}$$

the shear viscosity corrected for entrance pressure drop and non-Newtonian velocity profile (Weissenberg-Rabinowitsch correction) is given by:

$$\eta=\frac{D^4}{\left(32LvD_{\mathrm{b}}^2\right)}\times\left(\frac{4n}{3n+1}\right)\times\left(p-p_{\mathrm{c}}\right) \tag{C.6}$$

where

$\dot{\gamma}_{\mathrm{ap}}$ is the apparent shear rate;

Q is the volume flow rate;

D_{b} is the barrel diameter;

v is the piston speed;

p is the extrusion pressure;

p_{c} is the entrance pressure drop correction;

D is the capillary-die diameter;

L is the capillary-die length.

Thus, using Formulae (C.1), (C.2), and (C.6), it can be shown that the combined uncertainty in the measurement of shear viscosity $u_{\mathrm{c}}(\eta)$ is given by:

$$u_{\mathrm{c}}(\eta)=\eta\sqrt{\left[\left(\frac{4u(D)}{D}\right)^2+\left(\frac{u(L)}{L}\right)^2+\left(\frac{u(v)}{v}\right)^2+\left(\frac{2u(D_{\mathrm{b}})}{D_{\mathrm{b}}}\right)^2+\left(\frac{u(p)^2+u(p_{\mathrm{c}})^2}{(p-p_{\mathrm{c}})^2}\right)+\left(\frac{u(n)}{n(3n+1)}\right)^2\right]} \tag{C.7}$$

and the combined uncertainty in the shear rate $u_{\mathrm{c}}(\dot{\gamma})$ by:

$$u_{\mathrm{c}}(\dot{\gamma})=\dot{\gamma}\sqrt{\left[\left(\frac{3u(D)}{D}\right)^2+\left(\frac{u(v)}{v}\right)^2+\left(\frac{2u(D_{\mathrm{b}})}{D_{\mathrm{b}}}\right)^2+\left(\frac{u(n)}{n(3n+1)}\right)^2\right]} \tag{C.8}$$

The ratio of the uncertainty in the quantity $u(x_i)$ to the value of the quantity x_i is the relative uncertainty for that parameter, e.g. $u(D)/D$ is the relative uncertainty in D.

The effects of the temperature dependence of viscosity and degradation can be incorporated into Formula (C.7) as additional terms, thus:

$$u_c(\eta)=\eta\sqrt{\left[\left(\frac{4u(D)}{D}\right)^2+\left(\frac{u(L)}{L}\right)^2+\left(\frac{u(v)}{v}\right)^2+\left(\frac{2u(D_b)}{D_b}\right)^2+\left(\frac{u(p)^2+u(p_c)^2}{(p-p_c)^2}\right)+\left(\frac{u(n)}{n(3n+1)}\right)^2+f(d)^2+f(\theta)^2\right]}$$

(C.9)

where $f(d)$ is the relative uncertainty due to degradation and $f(\theta)$ is the relative uncertainty due to the combined effect of the temperature dependence of viscosity and errors in the test temperature. The latter, $f(\theta)$, can be expressed as:

$$f(\theta)=\frac{\partial\eta}{\partial\theta}u(\theta) \tag{C.10}$$

Thus, Formula (C.9) can be used to estimate the uncertainty in the measurement of shear viscosity given the uncertainties in, and magnitude of, each term. Formulae (C.7) and (C.9) are for true shear viscosity corrected for both entrance effects and the non-Newtonian velocity profile (Weissenberg-Rabinowitsch). Formula (C.8) is for true shear rates. Equivalent expressions can be deduced for apparent shear viscosity and apparent shear rate by removing the terms involving n and $u(n)$ from these formulae [effectively setting $u(n)$ to zero] and replacing the true shear viscosity term η in Formula (C.7) by the apparent shear viscosity term η_{ap}, and the true shear rate term $\dot{\gamma}$ in Formula (C.8) by the apparent shear rate term $\dot{\gamma}_{ap}$. Similarly, an expression for apparent shear viscosity that has not been corrected for entrance effects can be obtained by removing the terms $u(p_c)$ and p_c in Formula (C.7) [effectively equating $u(p_c)$ and p_c to zero].

C.2 Worked example

As a specific example, uncertainty values for the various components in the measurement of shear viscosity have been calculated and presented in Table C.1, based on the tolerance values and assumptions stated in this International Standard. To avoid excessive over-complexity of the uncertainty analysis, the calculations have been based on the measurement of shear viscosity using two dies: a long die of 20 mm length and a short die of negligible length that is used to determine the entrance pressure drop.

In carrying out an uncertainty analysis on a specific instrument, it would be preferable to utilize the calibration data to determine actual quantity ranges and, if appropriate, assume normal probability distributions for those ranges rather than rectangular distributions as assumed here.

Table C.1 — Uncertainty terms and estimates for shear viscosity measurement

Quantity, symbol, and units	Type[a]	Probability distribution[b]	Divisor[c]	Quantity value, x	Quantity range[d]	Standard uncertainty, $u(x)$[e]	Relative uncertainty, $u(x)/x$
Capillary-die length L, mm	B	R	$\sqrt{3}$	20	±0,025	0,014	0,000 7
Capillary-die diameter D, mm	B	R	$\sqrt{3}$	1	±0,007	0,004	0,004 0
Barrel diameter D_b, mm	B	R	$\sqrt{3}$	15	±0,007	0,004	0,000 27
Pressure measurement p, Pa	B	R	$\sqrt{3}$	variable	±1 % of range	—	0,005 8[f]
Pressure correction p_c, Pa	B	R	$\sqrt{3}$	variable (assume $p_c = 0{,}2p$)	±1 % of range	—	0,005 8[f]
Flow rate measurement Q, mm^3/s	B	R	$\sqrt{3}$	—	±1 %	—	—
Piston speed v, mm/s	B	R	$\sqrt{3}$	—	±1 %[g]	—	0,005 8[g]
Gradient of log τ versus log $\dot{\gamma}_{ap}$ plot, n	A	N	1	0,4	0,03[h]	0,03	0,075
Temperature θ, °C (for $\theta \leq 200$ °C)	B	R	$\sqrt{3}$	—	±1,5 °C	0,87	0,008 7[i]
Temperature θ, °C (for 200 °C < $\theta \leq 300$ °C)	B	R	$\sqrt{3}$	—	±2,5 °C	1,4	0,014[j]

[a, b] The uncertainties in type A quantities are estimated by statistical means. They are based on experimental observations of the variation in the values and are assumed to have a normal distribution (N). The uncertainties in type B quantities are estimated from the specification of tolerances given in this International Standard. In all type B cases, the quantities are assumed to have a rectangular distribution (R), i.e. the actual value has an equal probability of lying anywhere in the quoted tolerance range.

[c, d, e] The standard uncertainty is obtained by dividing the quantity range (d) by the divisor (c) that is based on the assumed probability distribution (b) of that quantity's range.

[f] The values quoted are for using the pressure transducer at its full range. When using the transducer in a lower part of its range, this value has to be factored accordingly, e.g. at 50 % of range, the relative uncertainties are doubled to 0,012. The uncertainties in the measurement of extrusion pressure p and pressure correction p_c were taken to be the same, specifically 1 % of the full-scale range of the pressure transducer. Also it was assumed that the magnitude of the pressure correction p_c was one-fifth of that of the extrusion pressure for the long die, thus $p_c = 0{,}2 \times p$.

The assumptions stated here imply that different transducers, of appropriate range, were used for each of the long and short dies. Where the same transducer is used in both cases, the uncertainties in p_c could be greater, estimated to be by a factor of approximately 5.

[g] The uncertainty in the piston speed is approximately the same as that in the flow rate as the contribution due to the relative uncertainty in the barrel diameter is negligible.

[h] The quantity range estimated for n is for one standard deviation.

[i, j] The uncertainty in the measurement of shear viscosity is related to the temperature dependence of the test sample. For the high-density polyethylene and glass-fibre-filled polypropylene samples used in the intercomparison,[6] it was estimated, based on measurements made at different temperatures, that the temperature dependence of shear viscosity was of the order of 1 %/°C. For polymers having temperature dependence factors different from this, the value of the relative uncertainty has to be factored accordingly, e.g. for a material having a temperature dependence of 2 %/°C, then the relative uncertainty is 0,028 for $\theta \leq 200$ °C. It also assumes that the quantity range is equal to the sum of the permitted spatial and temporal variations in temperature.

[k] In determining the values of the uncertainty in the measurement of shear viscosity where degradation can occur, it was also assumed, in accordance with this International Standard, that testing is carried out over a period during which the extrusion pressure (and hence viscosity) does not change by more than 5 %.

Table C.1 *(continued)*

Quantity, symbol, and units	Type[a]	Probability distribution[b]	Divisor[c]	Quantity value, x	Quantity range[d]	Standard uncertainty, $u(x)$[e]	Relative uncertainty, $u(x)/x$
Temperature θ, °C (for $\theta > 300$ °C)	B	R	$\sqrt{3}$	—	±3,5 °C	2,0	0,020[j]
Variation in extrusion pressure with time due to degradation effects	B	R	$\sqrt{3}$	—	±5 %	—	0,029[k]

[a, b] The uncertainties in type A quantities are estimated by statistical means. They are based on experimental observations of the variation in the values and are assumed to have a normal distribution (N). The uncertainties in type B quantities are estimated from the specification of tolerances given in this International Standard. In all type B cases, the quantities are assumed to have a rectangular distribution (R), i.e. the actual value has an equal probability of lying anywhere in the quoted tolerance range.

[c, d, e] The standard uncertainty is obtained by dividing the quantity range (d) by the divisor (c) that is based on the assumed probability distribution (b) of that quantity's range.

[f] The values quoted are for using the pressure transducer at its full range. When using the transducer in a lower part of its range, this value has to be factored accordingly, e.g. at 50 % of range, the relative uncertainties are doubled to 0,012. The uncertainties in the measurement of extrusion pressure p and pressure correction p_c were taken to be the same, specifically 1 % of the full-scale range of the pressure transducer. Also it was assumed that the magnitude of the pressure correction p_c was one-fifth of that of the extrusion pressure for the long die, thus $p_c = 0{,}2 \times p$.

The assumptions stated here imply that different transducers, of appropriate range, were used for each of the long and short dies. Where the same transducer is used in both cases, the uncertainties in p_c could be greater, estimated to be by a factor of approximately 5.

[g] The uncertainty in the piston speed is approximately the same as that in the flow rate as the contribution due to the relative uncertainty in the barrel diameter is negligible.

[h] The quantity range estimated for n is for one standard deviation.

[i, j] The uncertainty in the measurement of shear viscosity is related to the temperature dependence of the test sample. For the high-density polyethylene and glass-fibre-filled polypropylene samples used in the intercomparison,[6] it was estimated, based on measurements made at different temperatures, that the temperature dependence of shear viscosity was of the order of 1 %/°C. For polymers having temperature dependence factors different from this, the value of the relative uncertainty has to be factored accordingly, e.g. for a material having a temperature dependence of 2 %/°C, then the relative uncertainty is 0,028 for $\theta \leq 200$ °C. It also assumes that the quantity range is equal to the sum of the permitted spatial and temporal variations in temperature.

[k] In determining the values of the uncertainty in the measurement of shear viscosity where degradation can occur, it was also assumed, in accordance with this International Standard, that testing is carried out over a period during which the extrusion pressure (and hence viscosity) does not change by more than 5 %.

Given these assumptions, and the values presented in Table C.1, expanded uncertainty values for shear viscosity have been calculated for various temperature errors and temperature dependencies, for utilization of only part of the pressure transducer range, and with and without a contribution due to degradation effects, as shown in Table C.2. The values calculated assuming a 5 % effect of degradation are presented in brackets.

Table C.2 — Expanded uncertainties in measurement of true shear viscosity

All values expressed as percentages (%)

Temperature dependence of viscosity %/°C	**Temperature error** ± °C	**Expanded uncertainties (95 % confidence level) in viscosity measurement due to use of only part of the range of the pressure transducer, excluding degradation effects** [values in brackets include 5 % effect due to degradation, *f*(*d*) = 0,05]			
		using 100 % of range of pressure transducer	**using 50 % of range of pressure transducer**	**using 20 % of range of pressure transducer**	**using 10 % of range of pressure transducer**
0	—	7,9 (9,7)	8,7 (10,4)	11,1 (12,6)	21,8 (22,5)
1	1,5	8,1 (9,9)[a]	8,8 (10,6)	11,3 (12,7)	21,9 (22,6)
1	2,5	8,4 (10,2)	9,1 (10,8)	11,5 (12,9)	22,0 (22,7)
3	1,5	9,5 (11,1)	10,0 (11,6)	12,3 (13,6)	22,4 (23,1)

NOTE Values quoted are to one decimal place to show the trends in values, and not as an indication of the accuracy of such values.

[a] Corresponds to the case for the relative uncertainties presented in Table C.1, using a relative uncertainty in the temperature of 0,008 7.

The expanded uncertainty (95 % confidence level) in the apparent shear rate is calculated to be approximately 2,7 % and in true shear rate approximately 7 %, the difference being due to the Weissenberg-Rabinowitsch correction for the non-Newtonian velocity profile and the uncertainty associated with it (via the uncertainty in the gradient n of the log τ versus log $\dot{\gamma}_{\mathrm{ap}}$ plot). This correction is also a significant factor contributing to the overall uncertainty in the Weissenberg-Rabinowitsch-corrected true shear viscosity. In the above example, this factor alone gives an expanded uncertainty in true shear viscosity of 6,8 %.

The significant effect of using the pressure transducer in only the lower part of its range is clearly apparent in Table C.2. Using a transducer at only 10 % of its range can increase the uncertainty by a factor of up to about ×3.

These uncertainty values do not take into account errors due to viscous heating and the pressure dependence of viscosity. Both these factors will increase the level of uncertainty of the measurements and the reproducibility of the results, particularly when different test conditions are used to obtain viscosities at similar shear rates, for example by using different die dimensions.

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