

INTERNATIONAL  
STANDARD

**ISO**  
**11443**

First edition  
1995-02-01

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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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Reference number  
ISO 11443:1995(E)

## Foreword

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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.

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

Annexes A to D of this International Standard are for information only.

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International Organization for Standardization  
Case postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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

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

The rheological techniques described herein are not limited to the characterization of wall-adhering thermoplastic 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.

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

## 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based

on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 468:1982, *Surface roughness — Parameters, their values and general rules for specifying requirements*.

ISO 6507-1:1982, *Metallic materials — Hardness test — Vickers test — Part 1: HV 5 to HV 100*.

## 3 Definitions

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

**3.1 Newtonian fluid:** A fluid for which the viscosity is independent of the shear rate and of time.

**3.2 non-Newtonian fluid:** A fluid for which the viscosity varies with the shear rate and/or with time. 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,  $\tau_{ap}$ :** The fictive shear stress to which the melt in contact with the die wall is subjected.

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

It is expressed in pascals (Pa).

**3.4 apparent shear rate,  $\dot{\gamma}_{ap}$ :** The fictive shear rate that the melt at the wall would experience at the observed volume flow rate if its behaviour were Newtonian.

It is expressed in reciprocal seconds ( $\text{s}^{-1}$ ).

**3.5 true shear stress,  $\tau$ :** The actual shear stress to which the melt in contact with the die wall is subjected.

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.

It is expressed in pascals (Pa).

**3.6 true shear rate,  $\dot{\gamma}$ :** The 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 20).

It is expressed in reciprocal seconds ( $s^{-1}$ ).

NOTE 1 For purposes of notation, the absence of a subscript is used to denote true values.

**3.7 viscosity,  $\eta$ :** The viscosity in steady shear, defined as the ratio  $\tau/\dot{\gamma}$  of true shear stress  $\tau$  to true shear rate  $\dot{\gamma}$ .

It is expressed in pascal seconds (Pa·s).

**3.8 apparent viscosity,  $\eta_{ap}$ :** The ratio  $\tau_{ap}/\dot{\gamma}_{ap}$  of apparent shear stress  $\tau_{ap}$  to apparent shear rate  $\dot{\gamma}_{ap}$ .

It is expressed in pascal seconds (Pa·s).

**3.9 volume flow rate,  $Q$ :** The volume of melt flowing through the die per unit time.

It is expressed in cubic millimetres per second ( $mm^3/s$ ).

**3.10 swell ratio at room temperature,  $S_a$ :** The ratio of the diameter of the extrudate to the diameter of the capillary die, both measured at room temperature.

**3.11 swell ratio at the test temperature,  $S_T$ :** The ratio of the extrudate diameter to the capillary diameter, both measured at the test temperature.

**3.12 percent swell at room temperature,  $s_a$ :** The difference between the diameter of the extruded strand and the diameter of the capillary, expressed as a percentage of the diameter of the capillary, both measured at room temperature.

**3.13 percent swell at the test temperature,  $s_T$ :** The difference between the diameter of the extruded strand and the capillary diameter, expressed as a percentage of the capillary diameter, both measured at the test temperature.

NOTE 2 Equivalent extrudate swell measurements can be made on the thickness of slit-die extrudate with reference to the slit-die thickness.

**3.14 preheating time:** The time interval between completion of charging of the barrel and the beginning of measurement.

**3.15 dwell time:** The time interval between the completion of charging of the barrel and the end of measurements.

NOTE 3 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.

**3.16 extrusion time:** Time corresponding to the period of measurement for a given shear rate.

**3.17 critical shear stress:** The value of the shear stresses at the capillary 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.

It is expressed in pascals (Pa).

**3.18 critical shear rate:** The shear rate corresponding to the critical shear stress.

It is expressed in reciprocal seconds ( $s^{-1}$ ).

## 4 General principles

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

If a slit die with pressure transducers positioned along its length 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.

NOTE 4 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 exchangeable capillary or slit die. The test pressure shall be exerted on the melt contained in this barrel by a piston, a screw or gas pressure. Figures 1 and 2 show practical 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  $\pm 0,007$  mm.

The bore shall have a Vickers hardness of at least 800 HV 30 (see ISO 6507-1) and shall have a surface roughness less than  $R_a = 0,25$   $\mu\text{m}$  (average arithmetic discrepancy, see ISO 468).

#### NOTES

5 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.

6 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-tube wall shall be machined to an accuracy of  $\pm 0,007$  mm for the diameter ( $D$ ) and  $\pm 0,025$  mm for the length ( $L$ ) (see figure 1).

The internal surface texture of the capillary shall be smooth and shall correspond to a maximum roughness of  $R_a = 0,25$   $\mu\text{m}$  (average arithmetic discrepancy, see ISO 468).

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

The die shall have a Vickers hardness of at least 800 HV 30 (see ISO 6507-1). (See note 5).

#### NOTES

7 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.

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

9 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 ( $\pm 0,007$  mm) is difficult to obtain. Due to the extreme sensitivity of flow data to capillary dimensions, it is most important that both the capillary dimensions and the precision with which the dimensions are measured be known and reported. This also applies to the dimensions (thickness, width and length) of slit dies (see 5.1.4).

**5.1.3.2** For determining the apparent shear rate  $\dot{\gamma}_{ap}$  and the apparent shear stress  $\tau_{ap}$  with one capillary, unless otherwise specified by the referring standard, the ratio  $L/D$  of the length  $L$  to the diameter  $D$  of the capillary die shall be at least 20 and its inlet angle 180°. Only data obtained with capillaries of the same inlet angle ( $\pm 1^\circ$ ), length ( $\pm 0,025$  mm) and diameter ( $\pm 0,007$  mm) shall be compared. The inlet angle is defined in figure 1.

NOTE 10 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** For determining the true shear rate  $\dot{\gamma}$  and the true shear stress  $\tau$ , capillary dies of the same diameter ( $\pm 0,007$  mm) and inlet angle ( $\pm 1^\circ$ ) and having at least three different  $L/D$  ratios selected from the recommended series  $L/D = 5, 10, 20, 30, 40$  (see also 8.4.2) are required, except under the following conditions:

The use of only two dies, of the same diameter ( $\pm 0,007$  mm) and inlet angle ( $\pm 1^\circ$ ), of  $L/D \leq 5$  and  $L/D \geq 20$  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 separately, in accordance with 8.4, for each class of sample.

Dimensions in millimetres

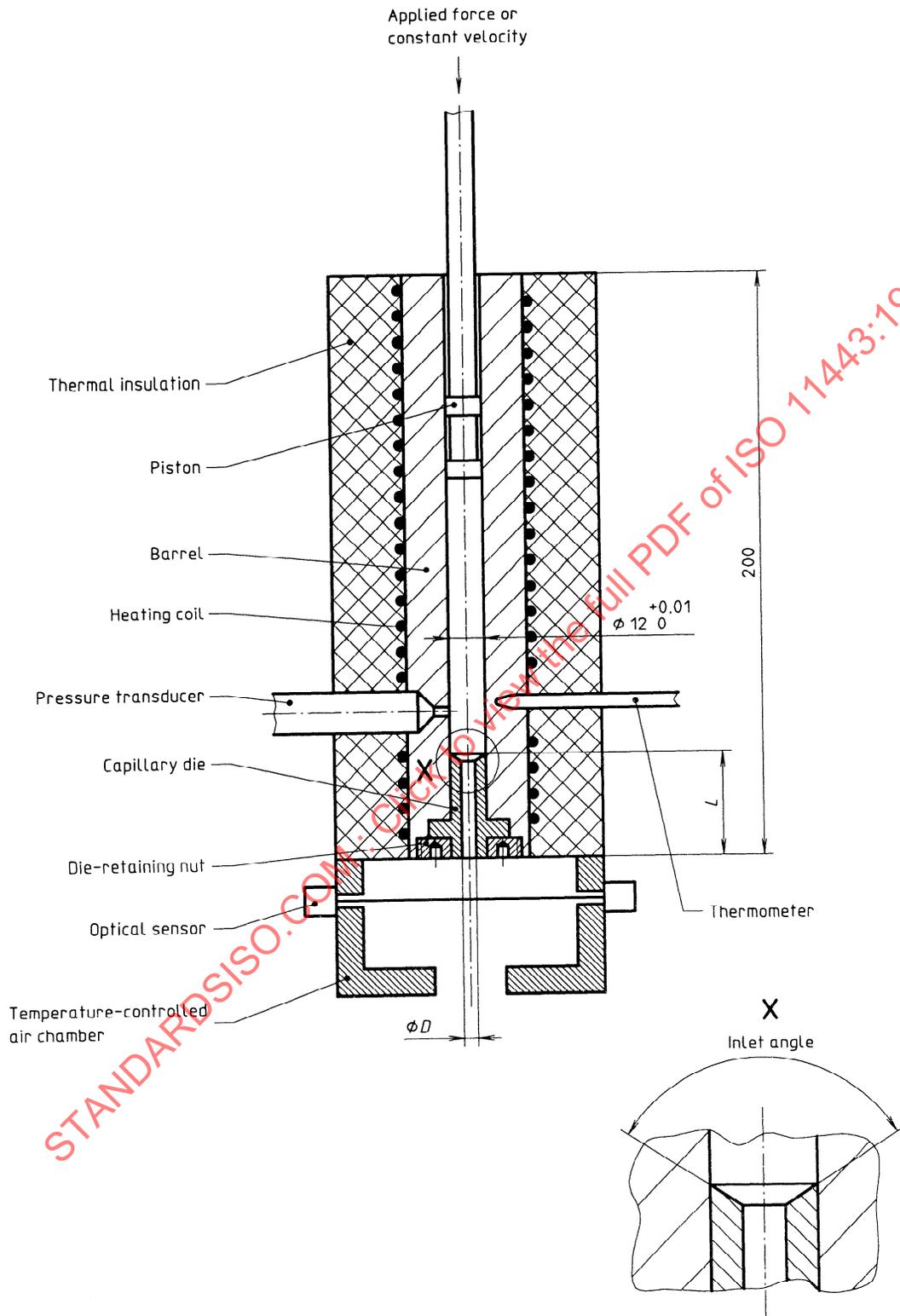
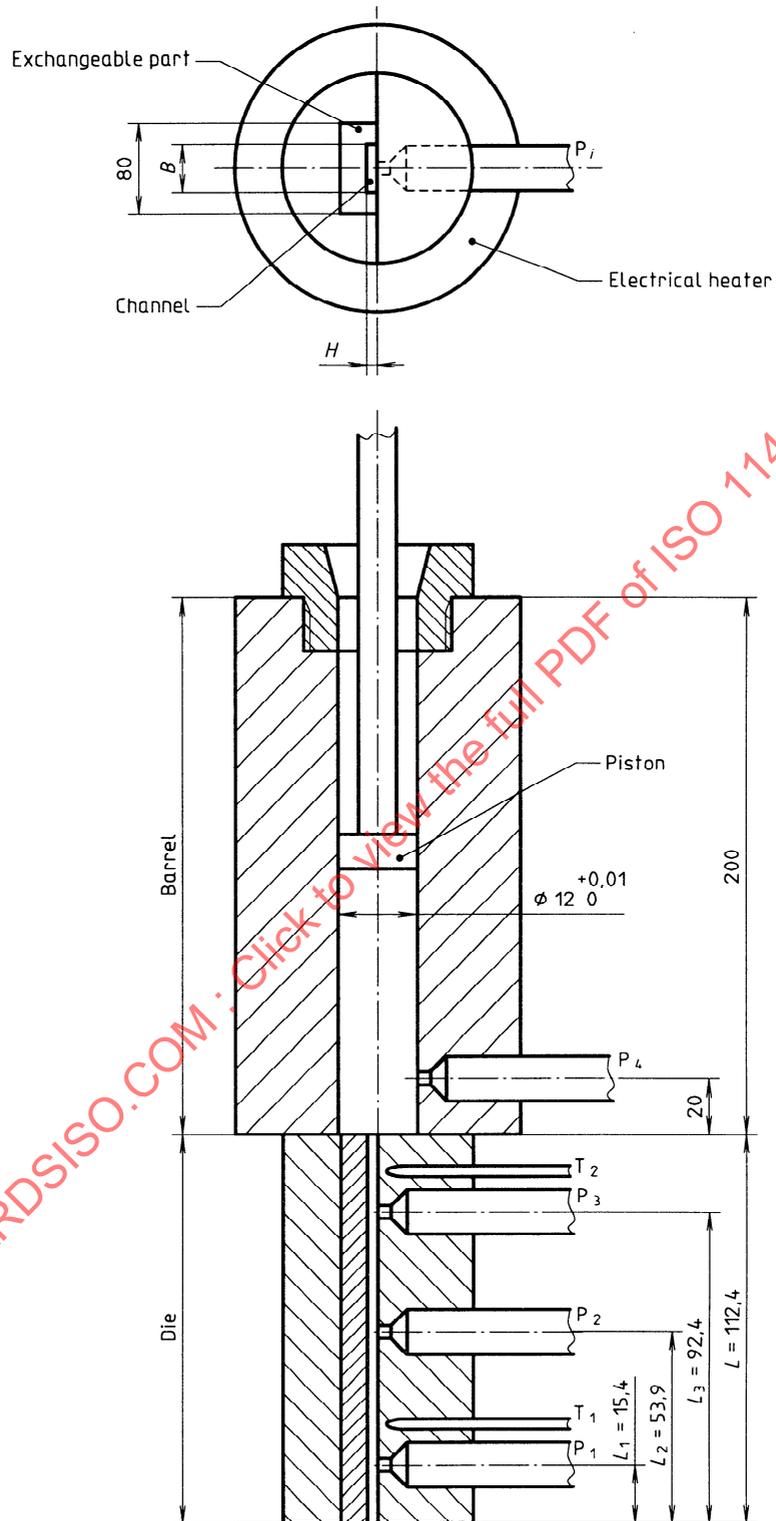


Figure 1 — Typical example of an extrusion rheometer used with a capillary die

Dimensions in millimetres



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**Key**

- $P_i$  = Pressure transducers
- $T_i$  = Thermometers

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

### 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  $\pm 0,007$  mm for the thickness,  $\pm 0,01$  mm for the width and  $\pm 0,025$  mm for the length. As applicable, the distance between the centres of the pressure transducers and the exit plane shall be determined to  $\pm 0,05$  mm. (See note 9.)

The internal surface of the slit die shall be smooth and correspond to a maximum roughness of  $R_a = 0,25$   $\mu\text{m}$  (see ISO 468).

The die shall have a Vickers hardness of at least 800 HV 30 (see ISO 6507-1). (See notes 5 and 8.)

NOTE 11 For slit-die materials, see note 8.

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

**5.1.4.3** To determine the true values of shear rate  $\dot{\gamma}$  and shear stress  $\tau$ , 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  $\pm 0,005$  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 measured at the wall do not exceed those given in table 2 throughout the duration of the test.

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

Values in degrees Celsius

Test temperature $\theta$	Temperature differences	
	as a function of distance	as a function of time
$\leq 200$	$\pm 1,0$	$\pm 0,5$
$200 < \theta \leq 300$	$\pm 1,5$	$\pm 1,0$
$> 300$	$\pm 2,0$	$\pm 1,5$

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. (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 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 die wall not more than 1,5 mm from the wall of 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.

### 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  $\pm 0,1$  °C, whilst complying with the depth of

immersion prescribed for the thermometer concerned. For this purpose, the barrel shall be filled to the top with a low-viscosity melt.

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

## 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 melt-pressure transducers located near the entrance of the die. Otherwise, the test pressure shall be determined by the force exerted, e.g. by the piston, on the melt (see B.1). If testing is to be carried out extruding to a channel or vessel pressurised to a pressure above atmospheric pressure, then the pressure at the die exit shall 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 10 % and 90 % of their nominal capacity.

### 5.4.2 Pressure drop along the length of the 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 (see 8.4.3) modified for slit dies.

### 5.4.3 Calibration

External hydraulic test equipment may 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 shall be less than or equal to 1 % of full scale. 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 rate of flow 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 rate of flow 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 rate of flow shall be determined to within 1 %.

NOTE 12 The specified maximum permissible error for determining the volume rate of flow 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.

## 6 Sampling

From the product 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, the transducer bores, where applicable, the piston and the 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 barrel, piston and capillary or slit die has occurred that might influence the test result.

NOTE 13 For the purpose of cleaning, circular brushes made of a copper/zinc alloy (brass) and linen cloths have proved satisfactory. Cleaning can also be performed by cautious burning out. Using graphite on threads facilitates unlocking after the test.

**WARNING — The operating conditions chosen may entail partial decomposition of the material under test, or cause it to release dangerous volatile substances. The user of this International Standard is therefore responsible for keeping him- or herself informed of possible risks of accident and for providing the appropriate means of protection.**

### 7.2 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 pre-

treatment or conditioning procedures in accordance with the referring standard and/or the relevant material standard, as applicable.

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 the warning in 7.1).

NOTE 14 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 in processing of the material. The shear stress and shear rate applied should also closely approximate those observed in the actual processing.

**Table 3 — Typical test temperatures**

Material	Temperature °C
Polyacetal	190 to 220
Polyacrylate	140 to 300
Acrylonitrile/butadiene/styrene (ABS)	200 to 280
Cellulose esters	190
Polyamide	190 to 300
Poly(chlorotrifluoroethylene)	265
Polyethylene and ethylene copolymers and terpolymers	150 to 250
Polycarbonate	260 to 300
Polypropylene	180 to 270
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

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.

### 7.3 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 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  $\pm 5\%$ , 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.

### 7.4 Determination of the maximum permissible test duration

For each sample and each test temperature, determine by testing, employing several different preheating times, prior to the actual test, 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  $\pm 5\%$ . (See also 7.3.)

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 15.)

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

If the test pressure necessary to maintain a given volume rate of flow is to be determined (see also 5.4.1 and 7.7), use either of the following methods (see table 1):

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

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

If, as an alternative to 7.5, the volume rate of flow for a given test pressure drop is required (see also 7.7), use either of the following methods (see table 1):

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

### 7.7 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 (to e.g.  $\pm 3\%$ ) over a given time period (e.g. 15 s).

NOTE 15 With a single barrel filling it is generally possible to determine several pairs of values for volume flow rate and test pressure.

Insert further waiting periods after changes of volume flow rate or test pressure, before taking measurements, in order to minimize the effects of adiabatic melt-temperature changes due to pressure variation. These additional waiting periods shall be as long as is necessary for the test pressure or volume flow rate, as applicable, to become constant to within the specified limits.

NOTE 16 It is recommended that selected measurements are repeated to check the repeatability.

## 7.8 Measurement of extrudate swelling

### 7.8.1 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 17 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) and the length of the extrudate, as well as the capillary length, the barrel diameter and the capillary diameter. The results obtained can be very sensitive to the details of the measurement technique.

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.

### 7.8.2 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).

### 7.8.3 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 point preferably 10 mm below the die outlet by photographic or optical techniques.

### NOTES

18 Extrudate swelling can be measured at other distances from the die along the extruded strand.

19 In order to minimize cooling of the extruded strand during the measurement of extrudate swelling, it is recommended that the strand be extruded into a temperature-controlled air chamber, such as that shown schematically in figure 1.

## 8 Expression of results

### 8.1 Volume rate of flow

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

$$Q = Av \quad \dots(1)$$

or

$$Q = \frac{\dot{m}}{\rho} \quad \dots(2)$$

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;
- $\rho$  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  $\dot{\gamma}_{ap}$ , in reciprocal seconds, at the die wall, using the equation given in 8.2.2 or 8.2.3, as applicable.

#### 8.2.2 Method A: capillary dies

$$\dot{\gamma}_{ap} = \frac{32Q}{\pi D^3} \quad \dots(3)$$

where

- $D$  is the diameter of the die bore, in millimetres;
- $Q$  is the volume rate of flow, in cubic millimetres per second (see 8.1).

NOTE 20 In the case of Newtonian fluids, equation (3) gives the true shear rate  $\dot{\gamma}$  at the capillary wall. As plastic melts do not generally exhibit Newtonian behaviour, the quantity calculated using this equation is termed the apparent shear rate  $\dot{\gamma}_{ap}$ . The true shear rate  $\dot{\gamma}$  can be determined from the apparent one  $\dot{\gamma}_{ap}$  by a correction procedure (see 8.5.1).

### 8.2.3 Method B: slit dies

$$\dot{\gamma}_{ap} = \frac{6Q}{BH^2} \quad \dots(4)$$

where

- $B$  is the width of the die, in millimetres;
- $H$  is the thickness of the die, in millimetres;
- $Q$  is the volume rate of flow, in cubic millimetres per second (see 8.1).

See also note 20.

NOTE 21 Equation (4) is strictly true only for infinitesimally small thickness-to-width ( $H/B$ ) ratios. The use of equation (4) overestimates the apparent shear rate  $\dot{\gamma}_{ap}$  by less than 3 %, however, if  $H/B \leq 0,1$ . A detailed analysis of the correctness of the approximation involved in using equation (4), together with a correction procedure, are given in annex A.

## 8.3 Apparent shear stress

### 8.3.1 General

Calculate the apparent shear stress  $\tau_{ap}$ , in pascals, at the die wall, using the equation given in 8.3.2 or 8.3.3, as applicable.

### 8.3.2 Method A: capillary dies

$$\tau_{ap} = \frac{pD}{4L} \quad \dots(5)$$

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

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

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 22 The shear stresses calculated using equations (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<sup>[3]</sup> (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 non-linear 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 23 In using capillary- 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. Non-linear plots may 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:

- 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  $\dot{\gamma}_{ap}$  at the die wall as a function of the test pressure  $p$  (see figure 3).
- 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  $\dot{\gamma}_{ap}$  at the die wall.

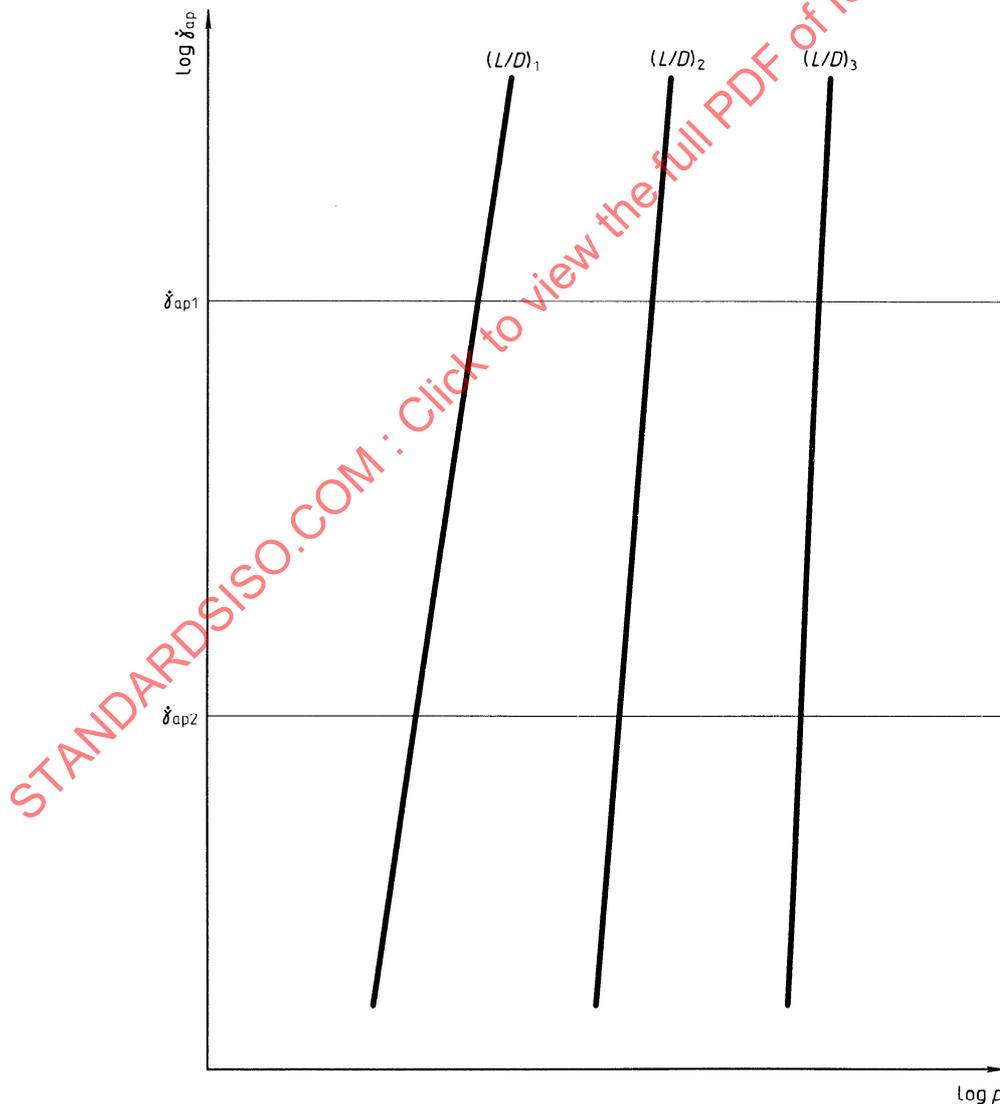
- c) From the data obtained in a) or b), as applicable, plot the test pressure  $p$  as a function of  $L/D$  for different values of the apparent shear rate  $\dot{\gamma}_{ap}$  (see figure 4). The so-called Bagley lines which result have a slope which is four times the true shear stress.

If, when using long capillary dies, deviations from the straight line arise owing to the influence of pressure on the melt viscosity or owing to viscous-dissipation effects, make the measurements using shorter capillary dies, unless otherwise specified by agreement in

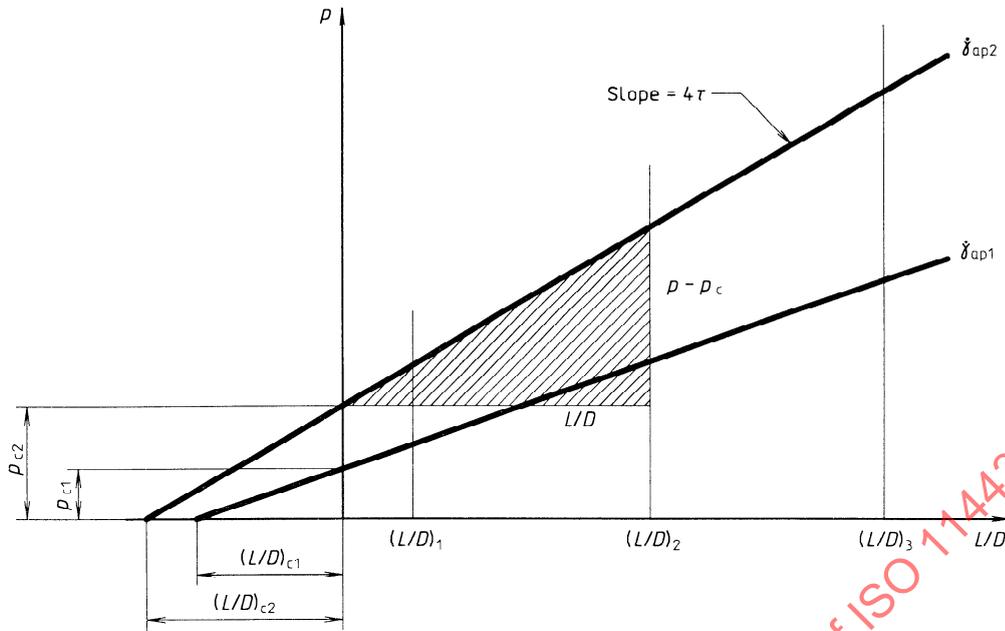
which case the procedure used shall be stated in the test report (see note 23).

NOTE 24 The Bagley correction may be performed using suitable computer programmes. 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  $\dot{\gamma}_{ap}$  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  $\dot{\gamma}_{ap}$  concerned.



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



**Figure 4 — Schematic Bagley plot for capillary dies: 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  $\dot{\gamma}_{ap}$**

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

$$\tau = (p - p_c) \frac{D}{4L} \quad \dots(7)$$

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  $(L/D)_c$  represent die-length corrections. Thus, as an alternative to equation (7), the true shear stress  $\tau$  for the apparent shear rate  $\dot{\gamma}_{ap}$  of interest may be calculated using equation (8):

$$\tau = \frac{p}{4[(L/D) + (L/D)_c]} \quad \dots(8)$$

where

- $p$  is the test pressure, in pascals;
- $L$  is the length of the capillary die, in millimetres;

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

$(L/D)_c$  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  $\dot{\gamma}_{ap}$  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  $\dot{\gamma}_{ap}$  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  $\dot{\gamma}_{ap}$ . The Bagley lines which result have a slope which is twice the true shear stress (see figure 5).

If, when using long slit dies, deviations from the straight line arise owing to the influence of pressure on melt viscosity or owing 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 23 and 24).

Extrapolate the Bagley line for each apparent shear rate  $\dot{\gamma}_{ap}$  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  $\dot{\gamma}_{ap}$  concerned.

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

$$\tau = \frac{HB}{2(H+B)} \times \frac{(p-p_c)}{L} \quad \dots(9)$$

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  $L_c(H+B)/HB$  represent die-length corrections. Thus, as an alternative to equation (9), the true shear stress  $\tau$  for the apparent shear rate  $\dot{\gamma}_{ap}$  of interest may be calculated using equation (10).

$$\tau = \frac{p}{2(L+L_c)} \times \frac{HB}{(H+B)} \quad \dots(10)$$

where

- $p$  is the test pressure, in pascals;
- $H$  is the thickness of the slit die, in millimetres;
- $B$  is the width of the slit die, in millimetres;
- $L$  is the length of the slit die, in millimetres;
- $L_c(H+B)/HB$  is the die-length correction (dimensionless).

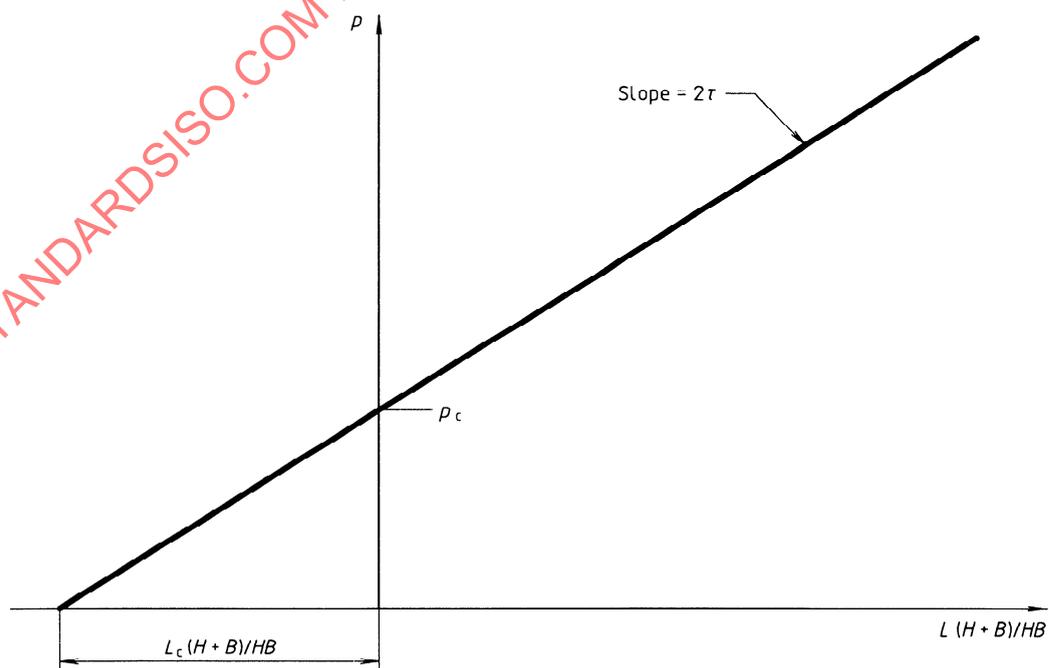


Figure 5 — Schematic Bagley plot for slit dies: the test pressure  $p$  is plotted as a function of  $L(H+B)/HB$  for dies of the same width  $B$  and thickness  $H$  for different values of the apparent shear rate  $\dot{\gamma}_{ap}$

### 8.4.4 Direct determination (method B)

From the longitudinal pressure gradient  $dp/dL$  measured using pressure transducers placed along the length of the slit die, calculate the true shear stress  $\tau$  at the die wall using the following equation:

$$\tau = \frac{HB}{2(H+B)} \times \frac{dp}{dL} \quad \dots(11)$$

where

$\frac{dp}{dL}$  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 and by applying the Weissenberg-Rabinowitsch correction method<sup>[4]</sup>, using equation (12) for method A (see 8.5.2) and equation (13) for method B (see 8.5.3).

### 8.5.2 Method A: capillary dies

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

where

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

### 8.5.3 Method B: slit dies

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

where

$\frac{d \log \dot{\gamma}_{ap}}{d \log \tau}$  is as defined in 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, apparent viscosities result. These shall be identified by the subscript "ap".

## 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 equations (14) and (15):

$$S_a = \frac{D_a}{D} \quad \dots(14)$$

$$s_a = \frac{D_a - D}{D} \times 100 \% \quad \dots(15)$$

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 equations (16) and (17):

$$S_T = \frac{D_m}{D_T} \quad \dots(16)$$

$$s_T = \frac{D_m - D_T}{D_T} \times 100 \% \quad \dots(17)$$

where

$D_m$  is the extrudate diameter, in millimetres, measured at the test temperature 10 mm below the die outlet;

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

NOTE 25 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 equations (14) to (17). Since swelling may be different in the width and thickness directions, it should preferably be determined in both of these directions.

## 9 Precision

An interlaboratory test programme was completed in 1990, involving seven laboratories and two materials (PP and PVC).

Comments and analysis of results:

Two types of apparatus and two measurement procedures were used:

- a rheometer measuring the extrusion pressure at the capillary inlet (4 laboratories) and a rheometer

measuring the force applied to the piston (2 laboratories);

- the shear rates applied during the tests were imposed successively in decreasing order of magnitude (2 laboratories) or increasing order (4 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 ( $< 100 \text{ s}^{-1}$ ) than at high shear rates ( $> 100 \text{ s}^{-1}$ ). Estimated repeatability was  $\pm 10 \%$  and  $\pm 5 \%$ , 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  $\geq 90^\circ$ .

The reproducibility of the method was estimated by measuring, in seven laboratories, the viscosity of PVC at  $180^\circ\text{C}$  and  $190^\circ\text{C}$  and that of PP at  $210^\circ\text{C}$  and  $240^\circ\text{C}$ . Reproducibility was found to be poorer at low shear rates ( $< 100 \text{ s}^{-1}$ ) than at high shear rates ( $> 100 \text{ s}^{-1}$ ), being  $\pm 20 \%$  and  $\pm 10 \%$ , 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 cleanness of the capillary on the results was not investigated in these tests.

See also annexes A, B and C.

## 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 standard;
- b) the information required by 10.2, 10.3 and 10.4, as applicable;
- c) the date of the test.

## 10.2 Test conditions

**10.2.1** A description of the material under test.

**10.2.2** Details of conditioning of the material.

**10.2.3** The method used (A1, A2, B1 or B2).

**10.2.4** A description of the rheometer used and its barrel diameter  $D_B$ .

**10.2.5** If applicable, 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.

**10.2.6** If applicable, the thickness  $H$ , width  $B$  and length  $L$  of the slit die used, and the degree of precision of these measurements.

**10.2.7** A description of the capillary-die or slit-die inlet angle profile, as applicable.

**10.2.8** A description of the technique used to measure the extrudate swelling.

**10.2.9** The temperature at which the measurements were made, and the degree of precision of the measurements.

**10.2.10** 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.

**10.2.11** The sample-preheating time.

**10.2.12** The dwell time.

**10.2.13** Where applicable, the dwell time corresponding to the appearance of an alteration in the material.

**10.2.14** The maximum permissible test duration.

**10.2.15** The extrusion time.

**10.2.16** 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 non-linear.

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

Include the following plots:

- 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 degrees Celsius at constant shear stress or shear rate;
- log critical shear stress or log critical shear rate for each of the observed effects (see 3.17 and 3.18) versus the reciprocal of absolute temperature;
- log critical shear stress or log critical shear rate for each of the observed effects (see 3.17 and 3.18) versus temperature in degrees Celsius;
- log volume rate of flow 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 rate of flow;

- 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.

### 10.3.3 Individual values

These may be given for a given series of test conditions:

- 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.

### 10.4 Visual examination

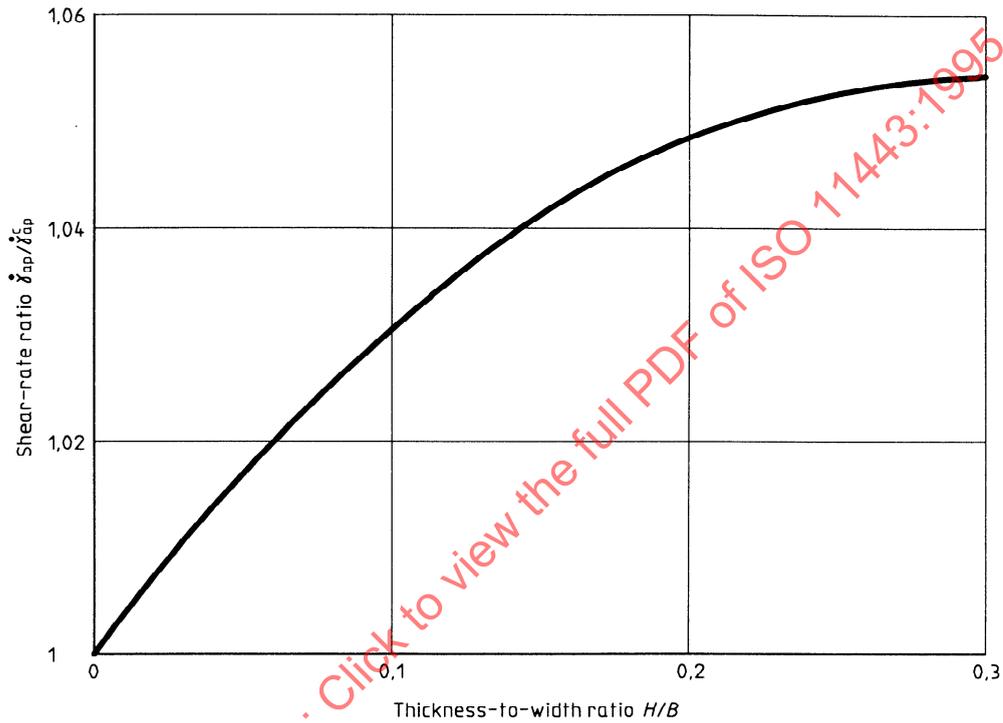
If visual examination is possible, report any change in the surface appearance of the extrudate (e.g. break in flow, distortion of extrudate), noting the test conditions at which the change occurs.

Such changes may 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  $\dot{\gamma}_{ap}$**



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

Equation (4) given in 8.2.3 for the apparent shear rate is valid only for an infinitely wide slit. Equation (4) therefore gives the apparent shear rate which will exist when the rate of volume flow 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  $H/B$ , equation (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 equation (4) and from equation (A.1), a corrected equation given in reference [5]:

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

where  $n$  is an odd integer.

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

$$\frac{\dot{\gamma}_{ap}}{\dot{\gamma}_{ap}^c} = \left(1 + \frac{H}{B}\right) \left[ 1 - 0,6274 \frac{H}{B} \sum_{n=1}^5 \frac{1}{n^5} \tanh \frac{n\pi B}{2H} \right] \dots(A.2)$$

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

The summation term in equation (A.2) is 1,004 4 when  $H/B \leq 0,3$ .

Hence

$$\begin{aligned} \frac{\dot{\gamma}_{ap}}{\dot{\gamma}_{ap}^c} &= \left(1 + \frac{H}{B}\right) \left(1 - 0,630 \frac{H}{B}\right) \\ &= 1 + 0,370 \frac{H}{B} - 0,630 \frac{H^2}{B^2} \dots(A.3) \end{aligned}$$

can be used as the correction equation.