

Methods of testing

Plastics —

Part 7: Rheological properties —

**Method 722B: Determination of the
fluidity of plastics using capillary and
slit-die rheometers**

ICS 83.080

Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee PRI/21, Testing of plastics, upon which the following bodies were represented:

- British Apparel and Textile Confederation
- British Plastics Federation
- Department of the Environment (Building Research Establishment)
- Department of Trade and Industry (National Physical Laboratory)
- Electrical and Electronic Insulation Association
- GAMBICA (BEAMA) Ltd.
- Institute of Materials
- Ministry of Defence
- Packaging and Industrial Films Association
- PIRA International
- RAPRA Technology Ltd.
- Royal Society of Chemistry

This British Standard, having been prepared under the direction of the Sector Board for Materials and Chemicals, was published under the authority of the Standards Board and comes into effect on 15 July 1996

© BSI 10-1999

The following BSI references relate to the work on this standard:
Committee reference PRI/21
Draft for comment 92/45392 DC

ISBN 0 580 26004 6

Amendments issued since publication

Amd. No.	Date	Comments

Contents

	Page
Committees responsible	Inside front cover
National foreword	ii
1 Scope	1
2 Normative references	1
3 Definitions	1
4 General principles	2
5 Apparatus	3
6 Sampling	7
7 Procedure	7
8 Expression of results	9
9 Precision	14
10 Test report	14
Annex A (informative) Method of correcting for the influence of H/B on the apparent shear rate $\dot{\gamma}_{ap}$	16
Annex B (informative) Measurement errors	17
Annex C (informative) Effect of flow conditions on measurement accuracy	18
Annex D (informative) Bibliography	19
Figure 1 — Typical example of an extrusion rheometer used with a capillary die	4
Figure 2 — Typical example of an extrusion rheometer used with a slit die	5
Figure 3 — Application of the Bagley correction method	11
Figure 4 — Schematic Bagley plot for capillary dies	13
Figure 5 — Schematic Bagley plot for slit dies	13
Figure A.1 — Shear-rate ratio $\dot{\gamma}_{ap}/\dot{\gamma}_{ap}^c$ versus thickness-to-width ratio H/B	16
Table 1 — Designation of test methods	2
Table 2 — Maximum allowable temperature differences as a function of distance and as a function of time	6
Table 3 — Typical test temperatures	8
Table C.1 — Errors	19
List of references	Inside back cover

National foreword

This British Standard has been prepared by Technical Committee PRI/21 and is identical with ISO 11443:1995 *Plastics — Determination of the fluidity of plastics using capillary and slit-die rheometers*, published by the International Organization for Standardization (ISO).

Cross-references

The Technical Committee has reviewed the provisions of ISO 468:1982 to which normative reference is made in the text and has decided that they are acceptable for use in conjunction with this standard. It is related to BS 1134-1:1988.

It has also reviewed the provisions of ISO 6507-1:1982 to which normative reference is made in the text and has decided that they are acceptable for use in conjunction with this standard. It is related to BS 427:1990.

NOTE International and European Standards as well as overseas standards are available from Customer Services, BSI, 389 Chiswick High Road, London, W4 4AL.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 20, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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 s^{-1} to 10^6 s^{-1} . 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.

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, τ_{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, $\dot{\gamma}_{\text{ap}}$

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 (s^{-1})

3.5

true shear stress, τ

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 $\dot{\gamma}_{\text{ap}}$ 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, η

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

3.8

apparent viscosity, η_{ap}

the ratio $\tau_{ap}/\dot{\gamma}_{ap}$ of apparent shear stress τ_{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 (mm³/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. Figure 1 and Figure 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 $R_a = 0,25$ μm (average arithmetic discrepancy, see ISO 468).

NOTE 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.

NOTE 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 $R_a = 0,25$ μm (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).

NOTE 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.

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

NOTE 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 τ_{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 τ , 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.

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 $R_a = 0,25$ μm (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.

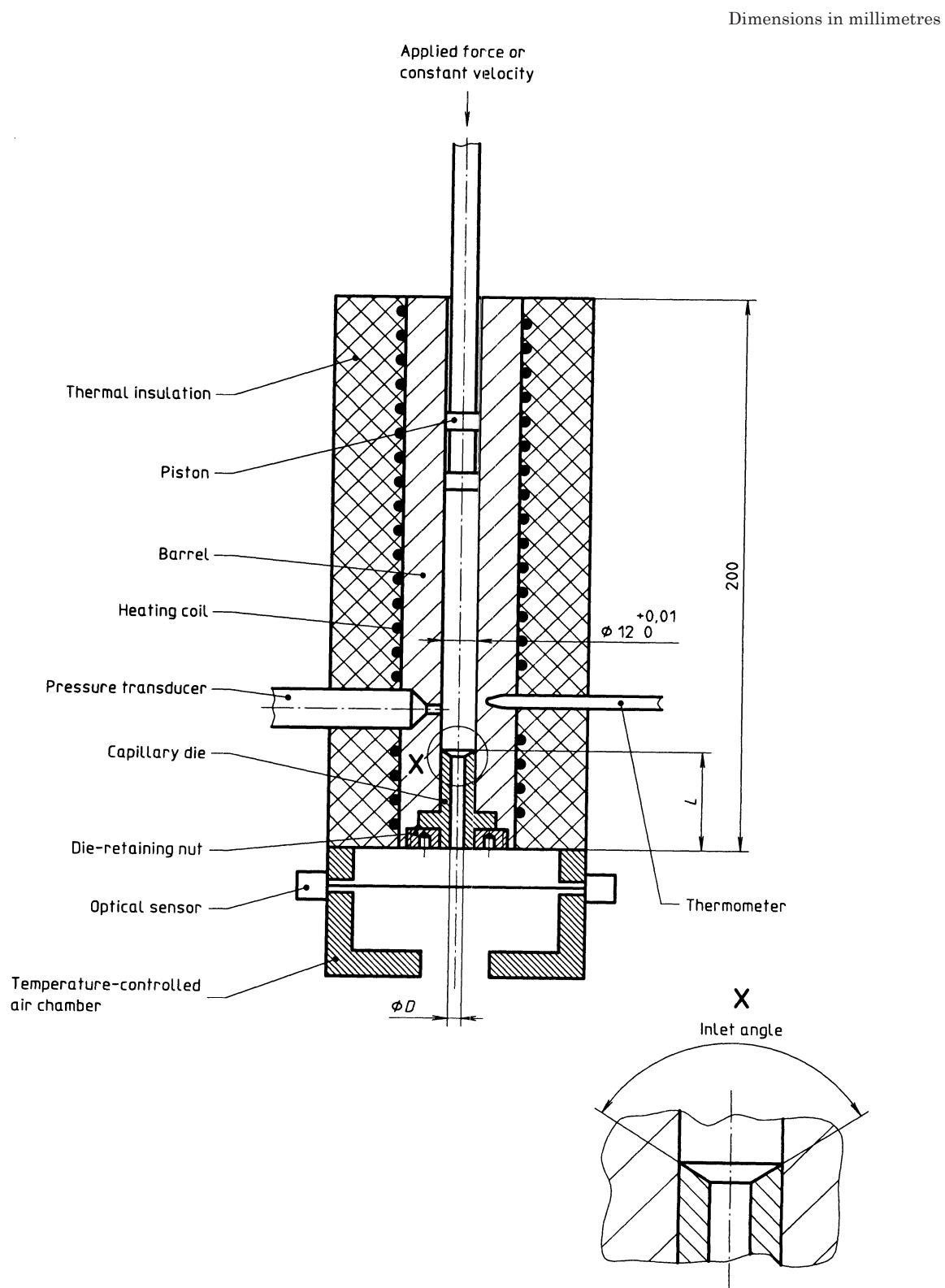


Figure 1 — Typical example of an extrusion rheometer used with a capillary die