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भारतीय मानक मसौदा

फ्लोरोपॉलीमर डिस्पर्स तथा संकचन और बहिर्वेधन के लिए सामग्री  
भाग 2 परीक्षण नमूनों की तैयारी एवं गुणधर्म ज्ञात करना

*Draft Indian Standard*

**FLUOROPOLYMER DISPERSIONS AND MOULDING AND EXTRUSION  
MATERIALS  
PART 2 PREPARATION OF TEST SPECIMENS AND DETERMINATION OF  
PROPERTIES**

*[Second Revision of IS 14635 (Part 2)]*

(ICS 83.080.20)

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Plastics Sectional Committee, PCD 12

Last date for Comments:

**12 August 2024**

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**FOREWORD**

This Indian Standard was originally published in 1999 and subsequently revised in 2020. Original Standard was based on ISO 899-1 : 1993 ‘Plastics — Determination of creep behaviour Part 1 : Tensile creep’ issued by the International Organization for Standardization (ISO) and ASTM D 1457 : 1990 ‘Standard specification for polytetrafluoroethylene (PTFE) moulding and extrusion materials’ issued by the American Society for Testing and Materials, ANSI, USA.

During the first revision, the Committee split the standard into two parts, as given below:

Part 1 Designation system

Part 2 Preparation of test specimens and determination of properties

In this revision (*second*), the major changes are as follows:

- a) Preparation of test specimens of melt processable polymers by compression moulding and injection moulding has been included;
- b) Conditioning for melt processable fluoropolymers has been included;
- c) Test method for determination of tensile properties has been included;
- d) Test method for determination of dielectric strength of fluoropolymers has been included; and
- e) Editorial corrections have been made.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

## 1 SCOPE

This standard (Part 2) prescribes the preparation of test specimens and test methods to define characteristics of thermoplastic fluoropolymer resins. Results from the testing can be used as the basis for designation, material specifications or both.

## 2 REFERENCES

The Indian Standards/other publications given below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No. Other Publication</i>	<i>Title</i>
IS 13360	Plastics — Methods of Testing
(Part 3/Sec 10): 2021/ISO 1183-1: 2019	Plastics — Methods of Testing : Part 3 Physical and Dimensional Properties, Section 10 Determination of Density of Non-cellular Plastics — Immersion Method Liquid Pyknometer Method and Titration Method ( <i>first revision</i> )
(Part 3/Sec 11): 2021/ISO 1183-2: 2019	Plastics — Methods of Testing : Part 3 Physical and Dimensional Properties, Section 11 Determination of Density of Non-cellular Plastics — Density Gradient Column Method ( <i>first revision</i> )
(Part 5/Sec 1): 2021/ISO 527-1: 2019	Plastics — Methods of testing : Part 5 Mechanical properties, Section 1 Determination of tensile properties — General requirements ( <i>second revision</i> )
(Part 5/Sec 2): 2017/ISO 527-2 : 2012	Mechanical properties, Section 2 Determination of tensile properties — Test conditions for moulding and extrusion plastics ( <i>first revision</i> )
(Part 5/Sec 3): 2022/ISO 527-3 : 2018	Plastics — Method of testing : Part 5 mechanical properties, Section 3 determination of tensile properties — Test conditions for films and sheets ( <i>second revision</i> )
14635 (Part 1): 2020	Fluoropolymer dispersions and moulding and extrusion materials : Part 1 Designation system ( <i>first revision</i> )
ISO 291 : 2008	Plastics — Standard atmospheres for conditioning and testing
ISO 293 :2023	Plastics — Compression moulding of test specimens of thermoplastic materials
ISO 565 : 1990	Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings
ISO 1133-1: 2022	Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics : Part 1 Standard method
ISO 11357-3: 2018	Plastics — Differential scanning calorimetry (DSC) : Part 3 Determination of temperature and enthalpy of melting and crystallization

ASTM D 4052: 2022	Standard test method for density, relative density, and API gravity of liquids by digital density meter
ASTM D 4591: 2022	Standard test method for determining temperatures and heats of transitions of fluoropolymers by differential scanning calorimetry
ASTM D 4894: 2019	Standard specification for polytetrafluoroethylene (PTFE) granular moulding and ram extrusion materials
ASTM D 4895: 2018	Standard specification for polytetrafluoroethylene (PTFE) resin produced from dispersion
ASTM E11: 2024	Standard specification for woven wire test sieve cloth and test sieves
ASTM E83: 2016	Standard Practice for Verification and Classification of Extensometer Systems
ASTM D5947: 2018	Standard Test Methods for Physical Dimensions of Solid Plastics Specimens
BS-EN-ISO 6158: 2018	Metallic and other inorganic coatings — Electrodeposited coatings of chromium for engineering purposes
IEC 60243-1: 2013	Electric strength of insulating materials — Test methods : Part 1 Tests at power frequencies

### 3 TERMS AND DEFINITIONS

For the purposes of this standard, the definitions as given in IS 14635 (Part 1) shall apply.

### 4 PREPARATION OF TEST SPECIMENS

Where applicable, Indian Standard or ISO standards shall be followed for the preparation of test specimens. In some instances, special procedures are required that are described either in the general discussion or in the method.

#### 4.1 Preparation of Test Specimens for non-melt processable fluoropolymers

##### 4.1.1 *Test Discs for Tensile Properties of PTFE and Closely Related Materials*

###### 4.1.1.1 *Apparatus*

###### 4.1.1.1.1 *Mould assembly, as illustrated in Fig. 1.*

The mould shall be assembled as shown in Fig. 1, leaving aside the upper pressing piece.

**4.1.1.1.2** *Hydraulic press*, capable of exerting a pressure of at least 35 MPa on the mould, or 70 MPa if filled compositions of PTFE are to be performed. The gauges on many presses read in absolute force units such as newtons or kilograms force. Care shall be taken to be sure that the required pressures are calculated correctly.

###### 4.1.2 *Procedure*

**4.1.2.1** Condition the material to be performed for at least 6 h in accordance with 5.

**4.1.2.2** Cover the inner surface of the bottom of the mould with a disc of aluminium foil slightly smaller in diameter than the inside of the mould cavity. This disc is used to prevent adhesion of the resin to the metal surface.

**4.1.2.3** Sieve a sample of approximately 20 g of the powder to be tested through a 2.0 mm aperture (No. 10) sieve. Weigh  $14.5 \pm 0.1$  g of the sieved powder and transfer to the mould. Adjust the height of the mould cavity so that the powder can be levelled by drawing a steel straight edge in contact with and across the top of the mould cavity.

**4.1.2.4** Position a second disc of aluminium foil on top of the powder.

**4.1.2.5** Insert the upper pressing piece, ensuring free movement within the body of the mould.

**4.1.2.6** Place the mould assembly between the platens of a suitable hydraulic press and increase the pressure applied to the moulding uniformly for 10 min. Use a pressure of 15 MPa to 35 MPa for PTFE-S (granular PTFE), 35 MPa to 70 MPa for filled compositions of PTFE, 15 MPa for PTFE-E (coagulated dispersion or fine powder).

**4.1.2.7** Hold the assembly for 3 min while maintaining the pressure at the specified value. Release the pressure slowly over a period of  $10 \pm 2$  s and carefully remove the preform from the mould. Remove the aluminium release foil from the moulding and identify the preform. Sinter the preform under the conditions given in **6.1.4** and Table 2.

#### NOTES

- 1) The adjustable cavity depth permits easier levelling of powders of different bulk densities.
- 2) Small-particle-size, low-powder-flow forms of PTFE-S can usually be performed satisfactorily at a pressure of 15 MPa. The pressure used with the filled compositions of PTFE should preferably be as high as possible (up to 70 MPa) without evidence of cracking of the preform.

#### **4.1.3** *Preparation of Test Billets for Determining Tensile Properties of PTFE on Skived Specimens or Specimens Cut in Other Ways*

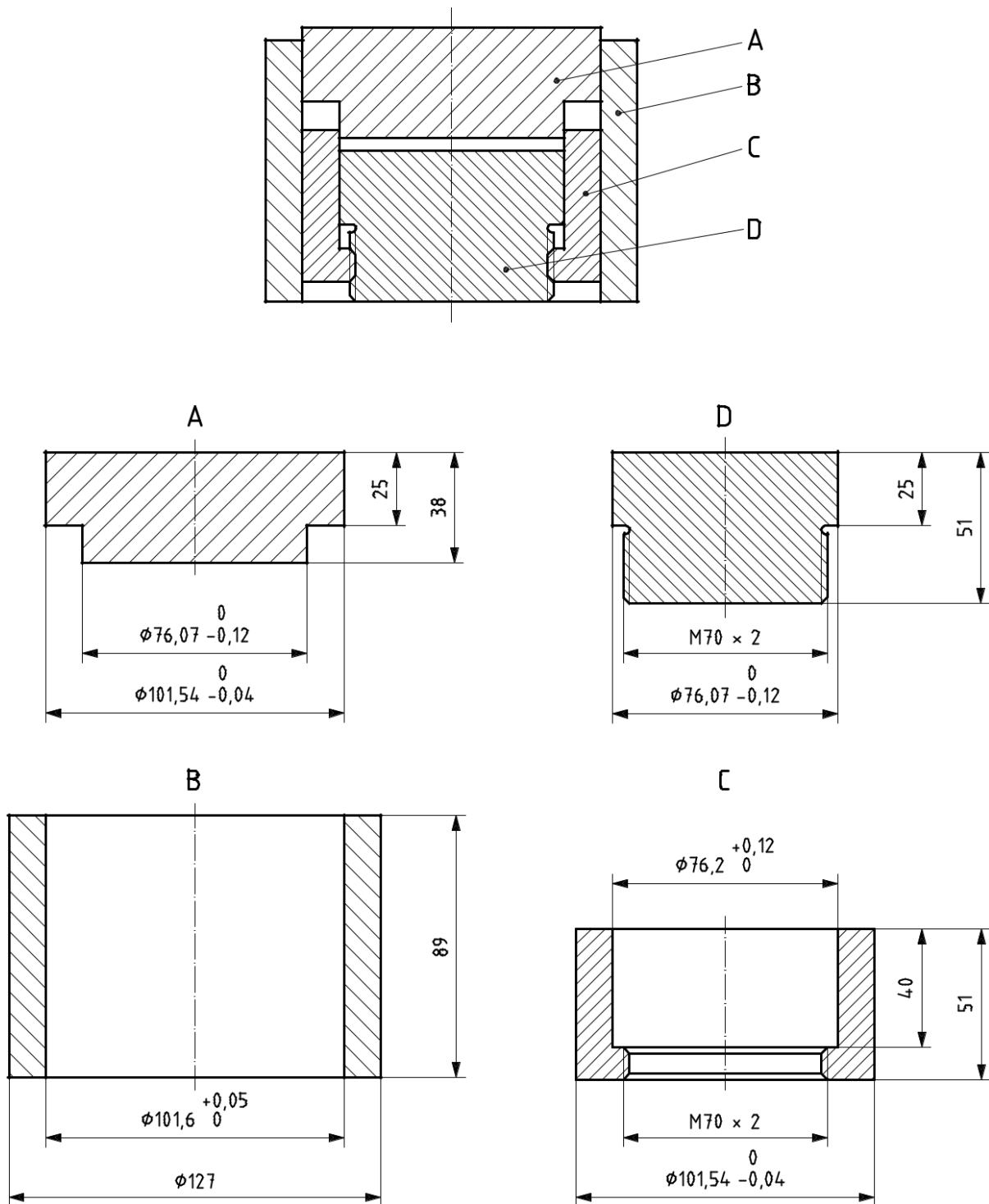
**4.1.3.1** Film or sheet skived or cut Skived sheet from small billets may can be used as alternatives to the test discs described in **4.1.1**. The apparatus for preforming a test billet is shown in Fig. 2. The preforming and sectioning of the test billet shall be done as described in ASTM D4894.

**4.1.3.2** Preform pressures shall be:

- a) 15 MPa for particle sizes less than 125  $\mu\text{m}$  for PTFE-S (granular PTFE)
- b) 25 MPa for particle size 125  $\mu\text{m}$  to 355  $\mu\text{m}$  for PTFE-S (granular PTFE)
- c) 35 MPa for particle size greater than 355  $\mu\text{m}$  for PTFE-S (granular PTFE); and
- d) 35 MPa to 70 MPa for filled PTFE.
- e) 14 MPa for PTFE-E (resin produced from coagulated dispersion)

**4.1.3.3** The nominal thickness of unfilled skived film shall be between 0.125 mm to 1 mm.

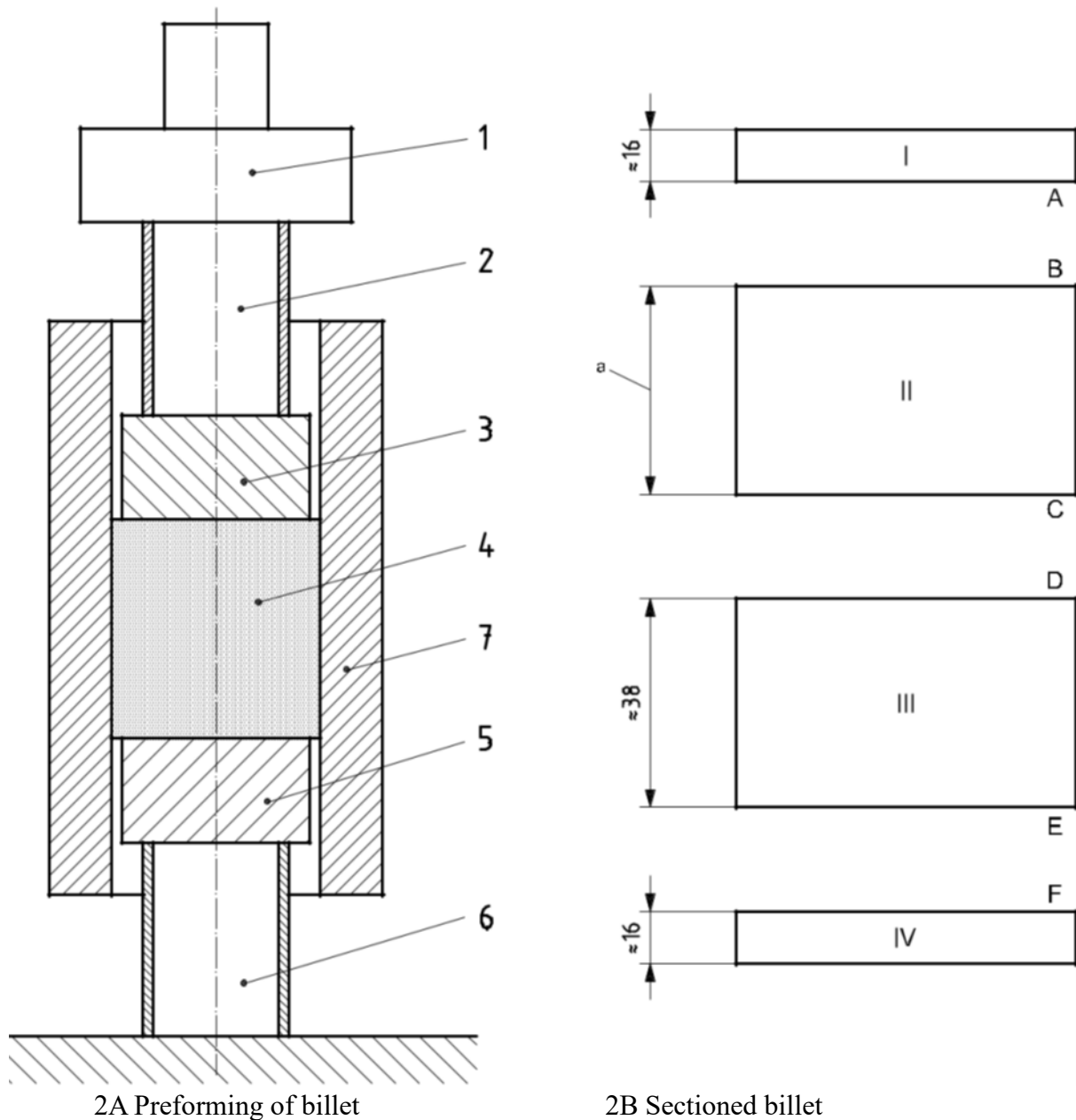
**4.1.3.4** The nominal thickness of filled skived film shall be between 0.5 mm to 1.2 mm.



NOTE — Components A, B, C and D of the assembly are all made of steel.

All dimensions in millimetres.

**FIG. 1 MOULD ASSEMBLY FOR PREPARATION OF SPECIMENS FOR THE DETERMINATION OF TENSILE PROPERTIES**



- 1 Ram
- 2 Steel pusher
- 3 Brass plug
- 4 PTFE resin
- 5 Plug
- 6 Pusher

- I, II, III, IV Designations of sections of billet
- A, B, C, D, E, F Designations of faces of billet sections

7 Steel mould, int. diam. H 57 mm,  
length 305 mm to 380 mm  
a Variable between 24 mm and 40 mm, depending on the total height of the billet after sintering.

All dimensions in millimetres.

**FIG. 2 APPARATUS FOR PREFORMING PTFE TEST BILLET**

## 4.2 Preparation of Test Specimens of melt processable polymers by Compression Moulding and Injection Moulding

### 4.2.1 Apparatus

Picture frame type moulding machine and injection moulding machine as given in ISO 293.

### 4.2.2 Procedure

**4.2.2.1** For tests that require a compression-moulded sheet, the principles of ISO 293 shall be followed with modification of details as presented in **4.2.2.2**. For tests that require an injection-moulded specimen, consult the manufacturer of the resin for suitable moulding conditions.

**4.2.2.2** Prepare a moulded sheet  $1.5 \text{ mm} \pm 0.25 \text{ mm}$  thick. Use a picture-frame- type chase having a suitable blanked-out section and thickness to produce the desired sheet. Use a release foil in contact with the resin (*see Note*). Use steel moulding plates at least 1.0 mm thick and of an area adequate to cover the chase.

**4.2.2.3** Cover the lower mould plate with a smooth piece of the release foil. Place the mould chase on top of this assembly. Place sufficient moulding material to produce the required sheet as a mound in the middle of the mould chase. Place a second sheet of the release foil on top of the granules and add the top mould plate. Place the assembly in a compression-moulding press having platens that have been heated to the temperature specified in Table 1 for the fluoropolymer being used.

**Table 1**  
(Clause 4.2.2.3 and 4.2.24)  
**Conditions for moulding test sheets**

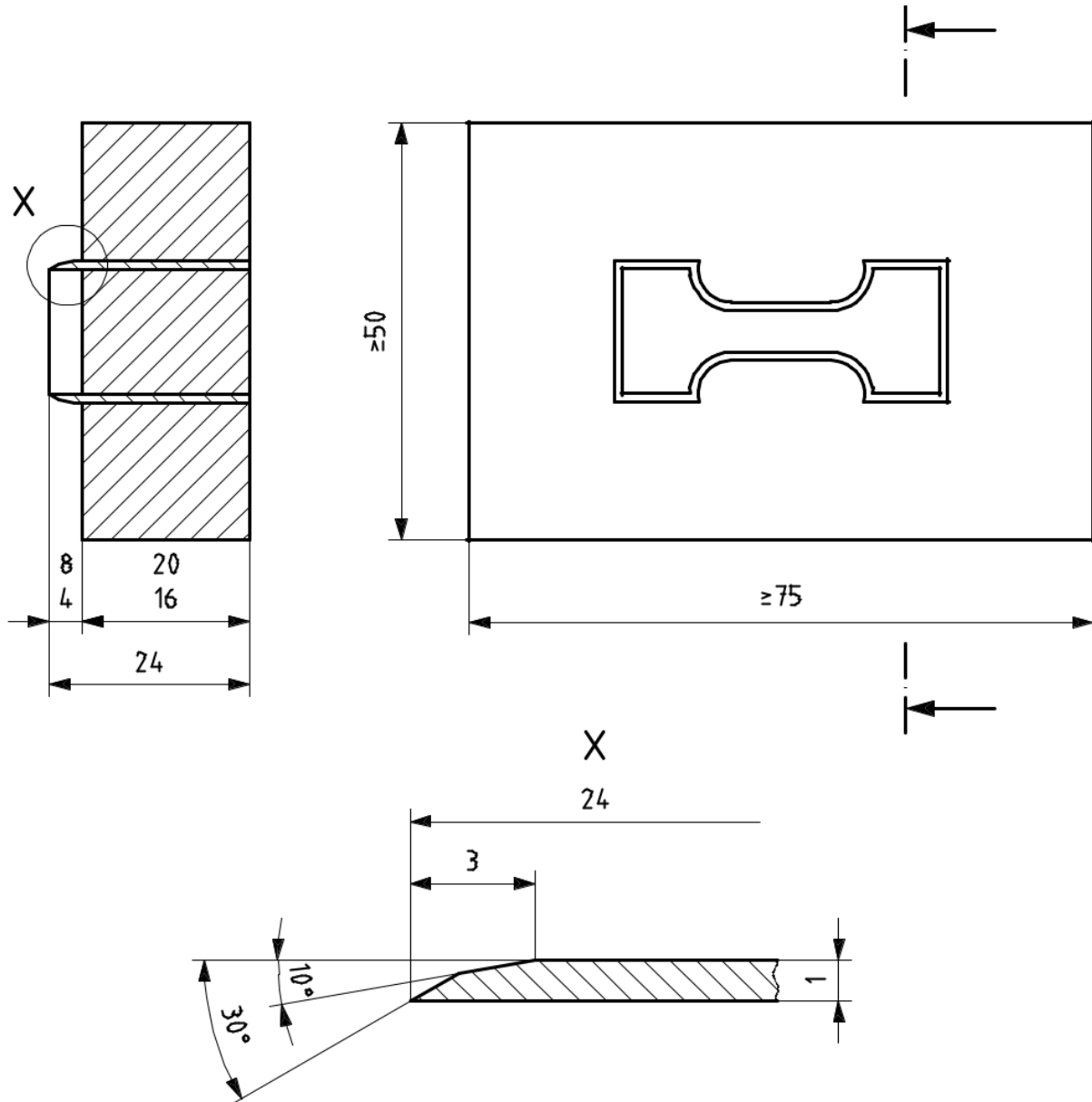
Fluoropolymer	Moulding temperature °C
FEP	325 ±5
PFA	380 ±5
ETFE	300 ±5
PVDF	205 ±5
PCTFE	265 ±5
ECTFE	265 ±5

**4.2.2.4** Bring the press platens to incipient contact with the mould assembly and hold for 5 min without pressure. Apply a pressure of at least 1 MPa and hold for 2 min. Then apply to 2 MPa to 4 MPa and hold for 1 min to 1.5 min. Maintain the press at the temperature specified in Table 1 for the fluoropolymer being used. Remove the assembly from the press and place between two  $20 \text{ mm} \pm 7 \text{ mm}$  steel plates whose temperature is less than 40 °C. Acceptable alternative procedures are to cool within the press or to move the mould assembly to a cooling cassette. For some materials, it is preferable to cool under pressure at a defined cooling rate.

**4.2.2.5** When the assembly has cooled to about 50 °C to 60 °C, it is cool enough to handle. Remove the release foil from the moulded sheet and arrange for the conditioning required

before testing. (If aluminium foil has been used as the barrier film and the assembly is allowed to cool to room temperature, the foil usually cannot be pulled free from the moulded sheet.)

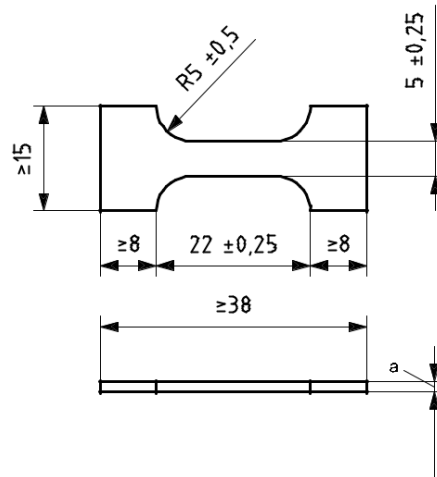
4.2.2.6 Prepare five specimens using the micro tensile die described in Fig 3. The die shall be of the steel-rule type with a curvature of  $5 \pm 0.5$  mm.



Inside dimensions of die are same as those of test specimen.  
Die to be sharpened on outside of knife edge only (as shown in figure).  
Rockwell C hardness of die: 45 to 50.

3(a) Steel Rule Die





3 (b) Micro-tensile specimen

Possible thickness:  $1.5 \pm 0.3$   
 $0.8 \pm 0.15$   
 $0.5 \pm 0.1$   
 $0.125 \pm 0.03$

**FIG 3 STEEL RULE DIE AND MICRO-TENSILE SPECIMEN**

## 5 CONDITIONING AND TEST CONDITIONS

### 5.1 Non-melt Processable Fluoropolymers

**5.1.1** For determinations of specific gravity, condition the moulded test specimens in the atmosphere as given in ISO 291 for a period of at least 4 h prior to testing. The other determinations require no conditioning.

**5.1.2** Conduct tests at a laboratory temperature of  $23 \pm 2$  °C for determining specific gravity, tensile and electrical properties.

### 5.2 Melt Processable Fluoropolymers

**5.2.1** For determinations of specific gravity, condition the moulded test specimens in the atmosphere as given in ISO 291 for a period of at least 4 h for PFA prior to testing. The other determinations require no conditioning.

**5.2.2** Test specimens defined in section 4.3 for PVDF polymer must be conditioned at  $23 \pm 2$  °C, at 50 percent relative humidity for 40 h before conducting tensile and elongation testing. Conduct tests at a laboratory temperature of  $23 \pm 2$  °C for determining specific gravity, tensile and electrical properties.

## 6 DETERMINATION OF PROPERTIES

Properties required for designation or specification, or both, shall be determined in accordance with the procedure given in this standard.

## 6.1 Standard Specific Gravity (SSG), Extended Specific Gravity (ESG) and Thermal Instability Index (TII)

6.1.1 Use the PTFE powder as received.

6.1.2 A cylindrical preforming mould is used to prepare the preforms prior to sintering. The mould is a tube 28.6 mm in internal diameter by at least 76.2 mm deep, with a removable bottom insert and a piston. Clearance between the piston and wall of the mould shall be sufficient to ensure escape of entrapped air during compression. Place flat aluminium foil discs, normally 0.13 mm thick and 28.6 mm in diameter, on each side of the resin. The test resin shall be near ambient temperature prior to preforming. For maximum precision, the weighing and performing operations shall be carried out in a constant-temperature room at  $23 \pm 1$  °C. The method shall not be run below 22 °C due to the 'room temperature' crystalline transition of PTFE which may lead to cracks in sintered specimens and differences in specimen density. ASTM D 4895 provides additional details.

6.1.3 Weigh out  $12.0 \pm 0.1$  g of resin and place it in the preforming mould. Screen non-free-flowing resins through a 2.0 mm (No. 10) sieve. Compacted resins can be broken up by hand-shaking cold resin in a half-filled sealed glass container. To do this, first condition the resin in the sealed glass container in a freezer or dry-ice chest. After shaking to break up resin lumps, allow the sealed container to equilibrate to near ambient temperature. Then screen and weigh  $12.0 \pm 0.1$  g test sample. Insert the mould in a suitable hydraulic press and apply pressure gradually (*see* Note) until the desired pressure is attained. The pressure shall be 34.5 MPa for PTFE granular moulding and ram extrusion materials, and 14 MPa for PTFE resin produced from coagulation of dispersion. Hold the pressure on the preform for 2 min. Release the pressure and remove the preform from the mould. A wax pencil may be used at this time to write an identification marking on the preform.

NOTE — As a guide, increasing the pressure at a rate of 3.5 MPa/min is suggested until the desired maximum is attained.

6.1.4 Place the sintering oven in a laboratory hood (or equip it with an adequate exhaust system) and sinter the preforms in accordance with the schedule as given in 6.1.4.1.

**WARNING** — PTFE resin can evolve small quantities of gaseous products when heated above 260 °C. Some of these gases are harmful. Consequently, exhaust ventilation must be used whenever the resins are heated above this temperature.

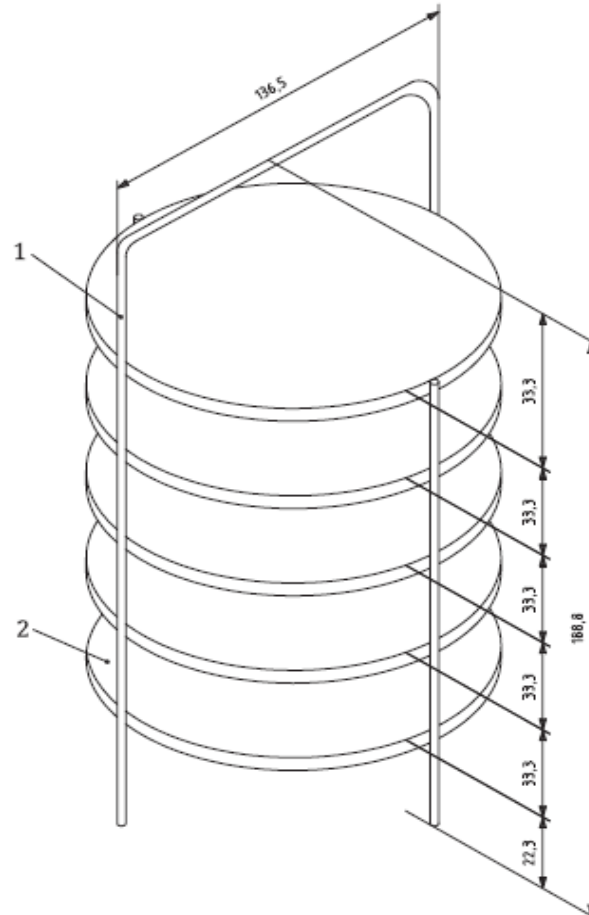
### 6.1.4.1 Sintering Conditions for Preparing SSG/ESG Specimens

Improved precision in the test values for standard specific gravity has been obtained with the use of an upright cylindrical oven and an aluminium sintering rack. The cylindrical oven has an inside diameter of 140 mm and a depth of 203 mm, plus additional depth to accommodate a 50.8 mm cover, and is equipped with adequate band heaters and controls to accomplish the sintering of specimens in accordance with Table 2. The rack, as shown in Fig. 4, allows preforms to be placed symmetrically in the centre region of the oven. Place six preforms on each of the middle oven rack shelves (if six or fewer preforms are to be sintered, place them on the middle rack, filling in with 'dummy' specimens as needed). Place dummy specimens on the top and bottom shelves. Space the specimens evenly in a circle on each shelf, with none of them touching. An oven load shall be no less than 18 pieces, including the additional dummy pieces. (Dummies are defined as normal 12 g specimens that have previously been through the

sintering cycle. Dummies shall be used only for an additional two or three thermal cycles, due to eventual loss of thermal stability and physical form.) ASTM D 4894 or ASTM D 4895 provides additional details.

**Table 2**  
(Clause 4.1.2.7 and 6.1.4.1)  
**Sintering Conditions for Preparing SSG/ESG Specimens**

a)	Initial temperature, °C	290
b)	Rate of heating, °C/h	120 ± 10
c)	Hold temperature, °C	380 ± 6
d)	Hold time, minutes	
	i) for SSG specimens	30 <sub>0</sub> <sup>+2</sup>
	ii) for ESG specimens	360 ± 5
e)	Cooling rate to 294 °C, °C/h	60 ± 5
f)	Second hold temperature, °C	294 ± 6
g)	Second hold time, min	24 <sub>-0</sub> <sup>+0.5</sup>
h)	Time to room temperature, min	>30



- 1 Support rods, diam. 6.35 mm (four required).  
2 Shelves, made of type 3003-H14 20 GA aluminium (five required).

NOTE —Aluminium plates tack-welded to rods.

**FIG. 4 RACK FOR SINTERING OVEN**

### 6.1.5 Determination of Standard Specific Gravity (SSG) or Extended Specific Gravity (ESG)

Determine the standard specific gravity (SSG) or extended specific gravity (ESG) of the moulded piece in accordance with one of the methods described in IS 13360 (Part 3/Sec 10) or IS 13360 (Part 3/Sec 11). Add two drops of a wetting agent (*see* 6.3.3.2.1) to the water in order to reduce the surface tension and ensure complete wetting of the specimen.

NOTE — When the specific gravity of PTFE is measured using immersion procedures, problems caused by the effect of temperature can be minimized if a sensitive thermometer (for example one reading to  $\pm 0.1$  °C) is used in the liquid and the temperature is kept at least 22 °C.

### 6.1.6 Determination of Thermal Instability Index (TII)

Calculate the thermal-instability index (TII) from the following equation:

$$TII = (ESG - SSG) \times 1\,000$$

where

*ESG* = Extended specific gravity, and

*SSG* = Standard specific gravity.

## 6.2 Bulk Density

Bulk density gives an indication of how a resin can perform during the filling of processing equipment. PTFE resins tend to compact during shipment and storage and, even though the material may be broken up by screening or some other means, original ‘as produced’ results may not be duplicated. Because of this tendency to pack under small amounts of compression or shear, the procedure given in 6.2.2 shall be used to measure this property for all fluoropolymer powders.

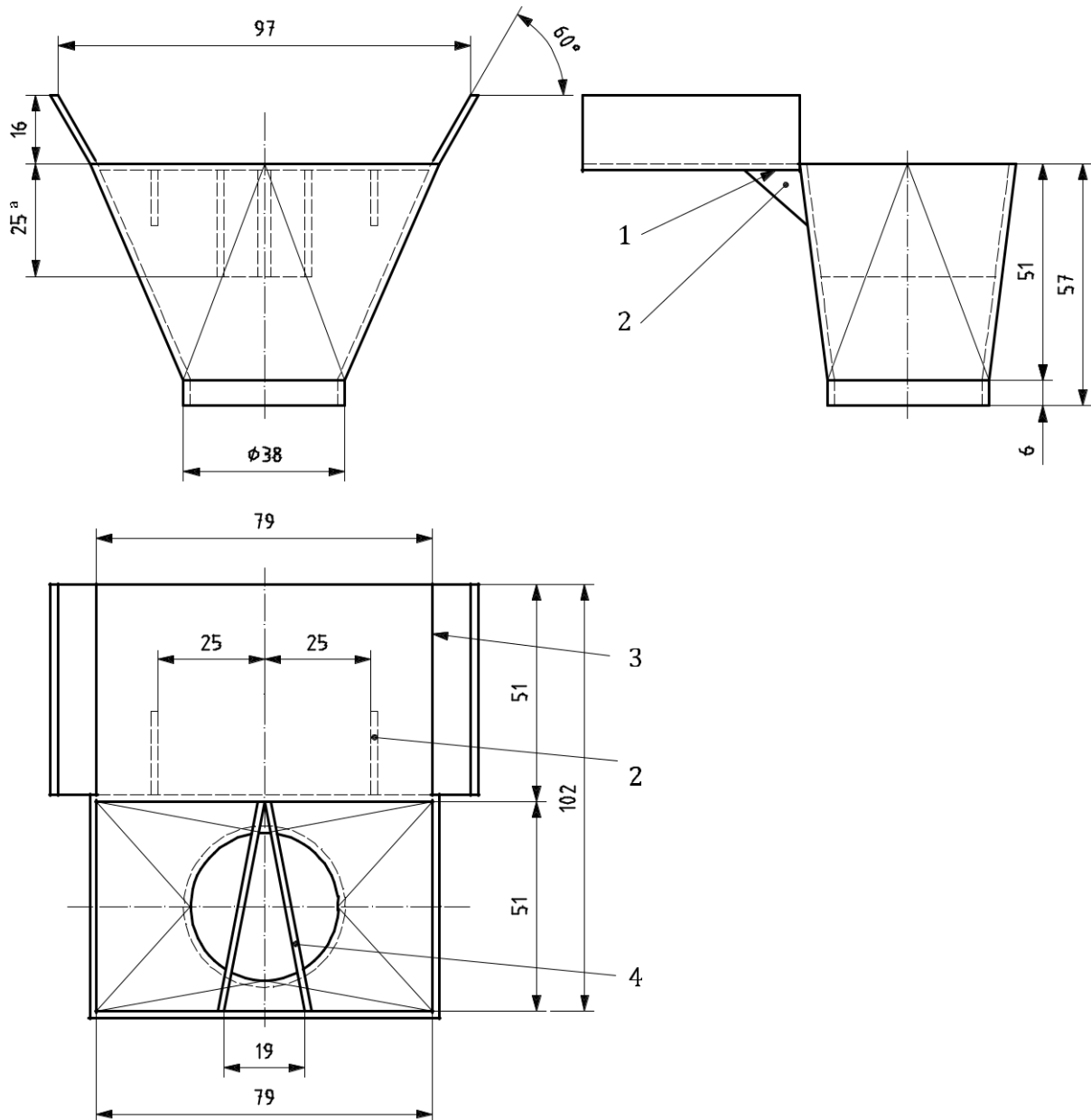
### 6.2.1 Apparatus

6.2.1.1 *Funnel*, as shown in Fig. 5.

6.2.1.2 *Feeder*, with a wire screen having 2.38 mm openings placed over approximately the top two-thirds of the trough. The funnel shall be mounted permanently in the feeder outlet (*see* Note 1).

6.2.1.3 *Controller* (*see* Note 2).

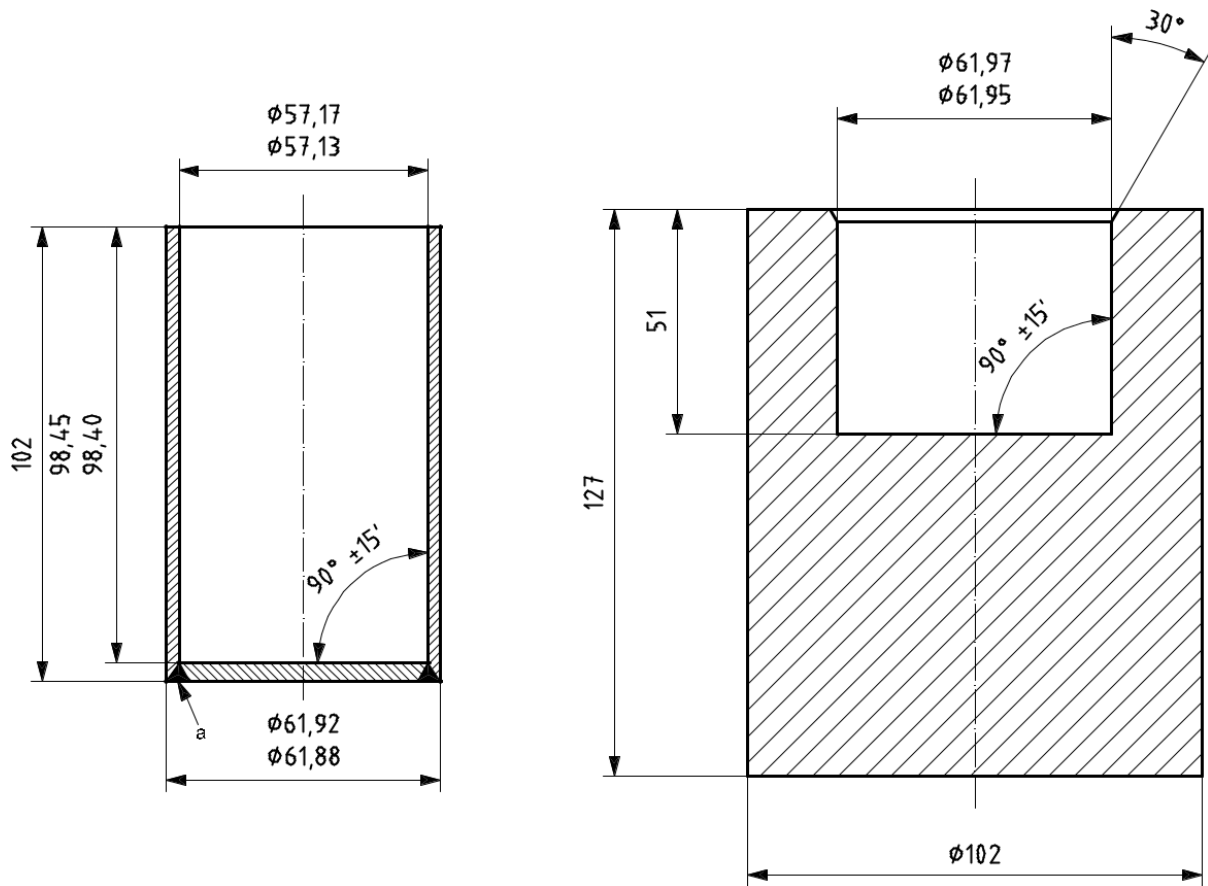
6.2.1.4 *Volumetric cup and cup stand*, as shown in Fig. 6. The top and bottom of both cup and stand shall be flat and parallel to within 0.05 mm. The inside bottom corner of the cup shall be square, as shown in the figure and the bottom of the hole in the cup stand shall be square with the centreline and with the top surface of the stand. All sharp external corners shall be removed from the cup stand. The volumetric cup shall be calibrated initially to 250 ml by filling it with distilled water, placing a planar glass plate on top, drying the outside of the cup, and weighing. The net mass shall be  $250 \pm 0.5$  g.



- 1 Weld
  - 2 Two support gussets, approx. 13 mm  $\times$  13 mm  $\times$  1.6 mm thick, located in positions shown
  - 3 Bend
  - 4 Straightening vanes (locate two partitions as shown)  
a Depth of partitions.
- Funnel material: type 304 stainless steel, 16 gauge (1.6 mm thickness).

All dimensions in millimetres.

**FIG. 5 DETAILS OF FUNNEL USED FOR DETERMINATION OF BULK DENSITY**



<b>6A Volumetric cup</b>	<b>6B Cup stand</b>
(Material: type 304 stainless seamless tubing)	(Material: 17 S-T aluminium or equivalent)
a Weld all round and grind smooth.	

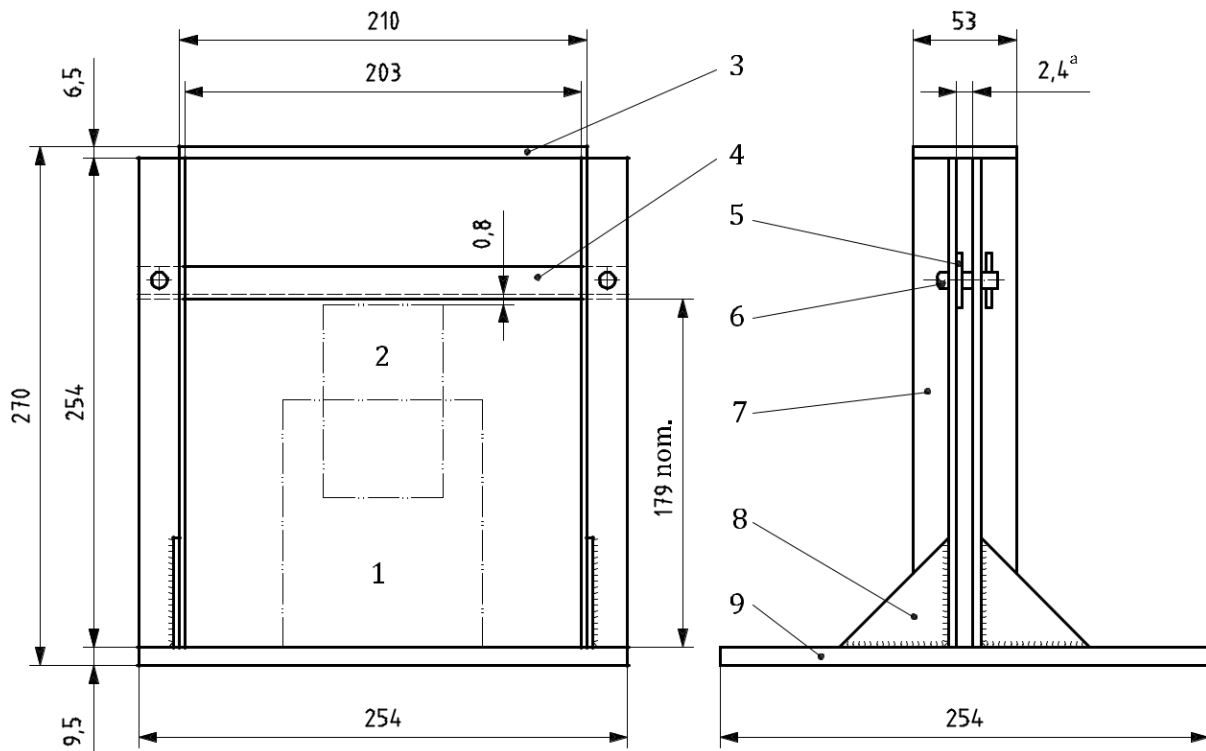
All dimensions in millimetres.

**FIG. 6 VOLUMETRIC CUP AND CUP STAND FOR DETERMINATION OF BULK DENSITY**

**6.2.1.5** *Levelling device*, as shown in Fig. 7, affixed permanently on the work table, and adjusted so that the sawtooth edge of the leveller blade passes within 0.8 mm of the top of the volumetric cup.

**6.2.1.6** *Work surface*, for holding the volumetric cup and leveller. It shall be essentially free from vibration. The feeder, therefore, shall be mounted on an adjoining table or wall bracket.

**6.2.1.7** *Balance*, having an extended beam, and with a capacity of 500 g and a sensitivity of 0.1 g or equivalent.



1	Cup stand
2	Cup
3	210 mm × 53 mm × 6 mm type 304 stainless-steel plate (or strap-welded across top for rigidity)
4	Leveller blade (e.g. Atkins saw blade No. 614-P), sawtooth edge, six teeth per 25 mm, 1.6 mm deep
5	Use shimstock or washers to take up clearance in 2.4 mm wide gap between the angle and saw blade
6	6 mm diam × 19 mm long brass rivet (two required) plus 2.4 mm diam. × 19 mm long brass cotter pin (two required) for mounting saw blade firmly in position (drill hole through angle and blade to 0.12 mm clearance with diameter of cotter pin)
7	25 mm × 25 mm × 3 mm type 304 stainless-steel angles (four required — two each end)
8	51 mm × 51 mm × 3 mm stainless-steel gussets (four required — two each end)
9	Type 304 stainless-steel plate

\* Gap left between angles for mounting saw blade.

All dimensions in millimetre.

**FIG. 7 LEVELLER STAND FOR DETERMINATION OF BULK DENSITY**

### 6.2.2 Procedure

Place the clean, dry volumetric cup on the extended beam of the balance and adjust the tare to zero. Select about 500 ml of the resin to be tested and place it on the feeder screen. Put the cup

in the cup stand and place the assembly such that the distance of free fall from the feeder outlet to the top rim of the cup is  $38.1 \pm 3.2$  mm. Increased fall causes packing in the cup and higher bulk-density values. Set the controller so that the cup is filled in 20 s to 30 s. Pour the sample on to the vibrating screen and fill the cup so that the resin forms a mound and overflows. Let the resin settle for about 15 s and then gently push the cup and its stand beneath the leveller. Exercise care to avoid agitation of the resin and cup before levelling. Weigh the resin to the nearest 0.1 g.

### 6.2.3 *Expression of Result*

Calculate the bulk density, in gram per litre, as follows:

$$\text{Bulk density} = \text{Mass of resin in cup} \times 4$$

#### NOTES

1. A laboratory-sized vibrating feeder has been found satisfactory for this purpose. Originally used was a “Vibra-Flow” feeder, Type F-T01A, with trough, which may still be available. This information is given for the convenience of users of this standard and does not constitute an endorsement. Equivalent products may be used if they can be shown to lead to the same results.
2. A suitable controller for the feeder should be used. Originally used was a “Syntron” controller, Type CSCRB1, which may still be available. This information is given for the convenience of users of this document and does not constitute an endorsement. Equivalent products may be used if they can be shown to lead to the same results.

## 6.3 Particle Size and Size Distribution

The wet and dry-sieve procedures of 6.3.1 and 6.3.2 are widely used with PTFE. The resistance-variation test procedure in 6.3.3 (the Coulter principle) is often used with PTFE fine-cut suspension powders, PVDF, PFA and filled PTFE resins. The light-scattering procedures in 6.3.4 are becoming more widely used with all the fluoropolymers. The use of automatic or other instruments that have been shown to provide equivalent results is an acceptable alternative.

### 6.3.1 *Wet-sieve Analysis*

**WARNING** — Perchloroethylene is under investigation by government agencies and industry for its carcinogenic effects. Establish protective measures in accordance with applicable health and safety requirements to prevent skin contact and provide ventilation of vapours. The supplier’s SDS sheet should be consulted for more information about required safety measures.

#### 6.3.1.1 *Apparatus and materials*

6.3.1.1.1 *Balance*, capable of weighing to  $\pm 0.1$  g.

6.3.1.1.2 *Standard sieves*, 203 mm diameter, conforming to ISO 565. It is suggested that the following sieve openings (sieve numbers) be used: 1.4 mm (No. 14), 1 mm (No. 18), 710  $\mu\text{m}$  (No. 25), 500  $\mu\text{m}$  (No. 35), 355  $\mu\text{m}$  (No. 45), 250  $\mu\text{m}$  (No. 60) and 180  $\mu\text{m}$  (No. 80). The equivalent sieve numbers, given for information, are those defined in ASTM E11. Other sieve



configurations may be used provided they give equivalent results. It is desirable to use a set of sieves that have openings that are uniformly related on a logarithmic scale.

#### **6.3.1.1.3** *Ventilated hood*

**6.3.1.1.4** *Six tared beakers*, capacity 150 ml. As an alternative, the sieves can be tared, dried and weighed on a balance to avoid errors that can be introduced during transfer of fractionated samples to the tared beakers.

**6.3.1.1.5** *Sieving and solvent-spraying apparatus*, a suggested arrangement for an apparatus with recirculating spray liquid is shown in Fig. 8. The apparatus shall be located, and the operations carried out, in a ventilated hood or adequately ventilated area.

**6.3.1.1.6** *Spray liquid, 20 litre*, see the comments and warning in **6.3.1**. Although perchloroethylene has been the usual choice, an alternative liquid may be used after its applicability and any hazards associated with its use should be investigated thoroughly and the use of the liquid may be satisfactory.

#### **6.3.1.2** *Procedure*

**6.3.1.2.1** Weigh out a 10 g test sample for powders with a particle size less than 100  $\mu\text{m}$  or a 50 g test sample for powders with larger particle size. Adjust the rate of flow of the spray liquid to  $6 \pm 0.5$  litre/min.

**6.3.1.2.2** Place the weighed resin on the top sieve and spray it with the organic spray liquid for  $1 \pm 0.2$  min. The shower head shall be about level with the top of the sieve and be moved in a circular fashion. Take care to break up all of the lumps and to wash the material from the sides of the sieve.

**6.3.1.2.3** Remove the top sieve and place it in the hood to dry until all the sieves are ready for oven drying as described in **6.3.1.2.4**.

**6.3.1.2.4** Repeat the procedure specified in **6.3.1.2.2** and **6.3.1.2.3** until all the sieves have been sprayed. Dry the sieves in a ventilated oven at a temperature of at least 90 °C up to a maximum of 130 °C for at least 15 min up to a maximum of 30 min and then cool to room temperature. Remove the resin from each sieve by tapping on a piece of paper as shown in the insert in Fig 8. Pour each fraction into a tared beaker and weigh to  $\pm 0.1$  g.

**6.3.1.2.5** Record the mass of resin on each sieve.

**6.3.1.2.6** Clean the sieve by inverting it over filter paper and spraying it with spray liquid. Take care to prevent the resin from getting into the spray liquid.

#### **6.3.1.3** *Expression of Results*

**6.3.1.3.1** Calculate the net percentage of resin on each sieve as follows:

$$\text{Net percentage of resin on sieve } Y = F \times m$$

where

$F = 2$  for a 50 g test sample or 10 for 10 g test sample; and  
 $m$  = mass of resin on sieve  $Y$ , in g.

**6.3.1.3.2** Calculate the cumulative percentage of resin on each sieve as follows:

Cumulative percentage of resin on sieve  $Y$  = sum of net percentage on sieve  $Y$  and on sieves having sizes greater (that is numbers smaller) than sieve  $Y$ .

Example — Cumulative percentage on 500  $\mu\text{m}$  (No. 35) sieve equals net percentage on 1,4 mm (No. 14) plus net percentage on 1.0 mm (No. 18) plus net percentage on 710  $\mu\text{m}$  (No. 25) plus net percentage on 500  $\mu\text{m}$  (No. 35) sieve.

**6.3.1.3.3** Plot the cumulative percentage versus the sieve opening size (or sieve number) on log/log paper as shown in the sample plot in Fig. 9. The sieve numbers and sieve opening sizes in micrometres are indicated below the figure. Draw the best straight line through the points and read the particle size at the 50 percent cumulative percentage point ( $d_{50}$ ). Take this value as the average particle size. It is permissible to carry out the calculation of  $d_{50}$  by use of computer programmes that provide ‘best-fit’ analysis using linear regression procedures involving a log-normal model.

## **6.3.2** *Dry-sieve analysis*

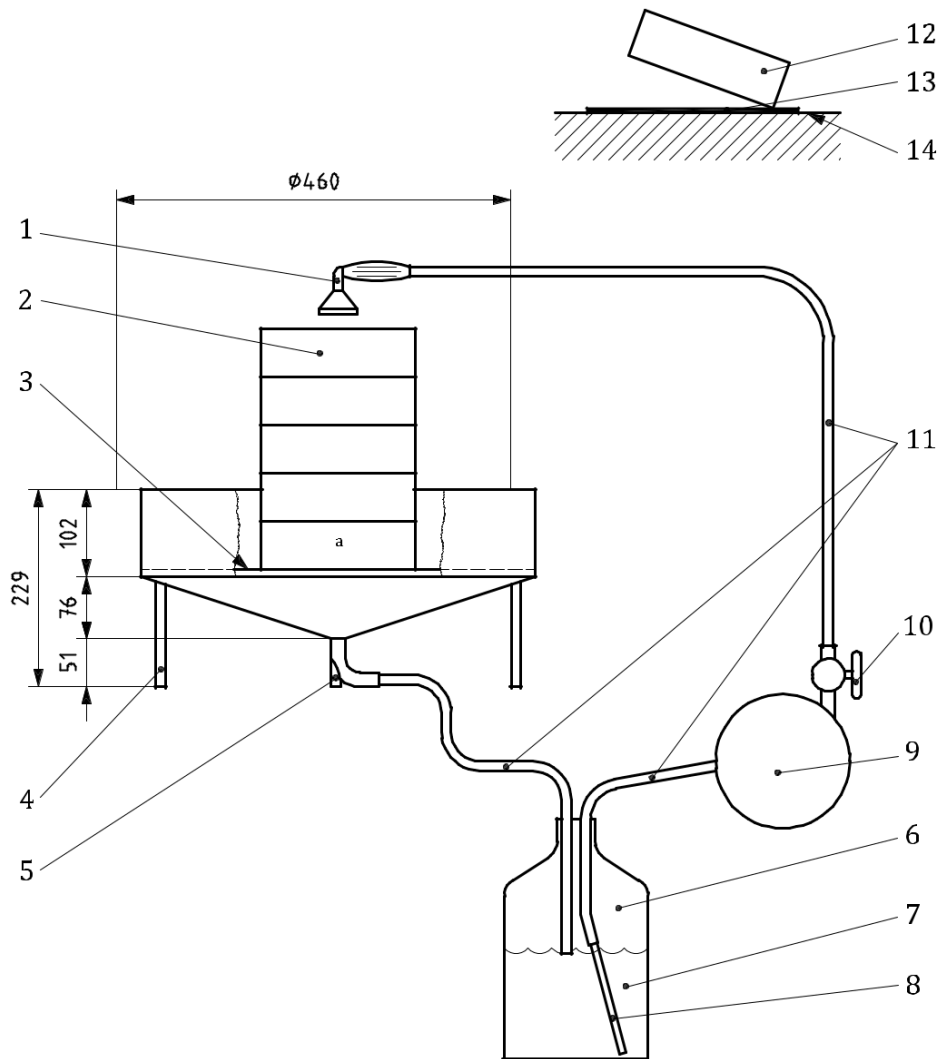
### **6.3.2.1** *Apparatus*

**6.3.2.1.1** *Balance*, capable of weighing to  $\pm 0.1$  g.

**6.3.2.1.2** *Standard sieves*, 203 mm diameter, conforming to ISO 565. It is suggested that the following sieve openings (sieve numbers) be used: 1.4 mm (No. 14), 1 mm (No. 18), 710  $\mu\text{m}$  (No. 25), 500  $\mu\text{m}$  (No. 35), 355  $\mu\text{m}$  (No. 45), 250  $\mu\text{m}$  (No. 60) and 180  $\mu\text{m}$  (No. 80). The equivalent sieve numbers, given for information, are those defined in ASTM E11. Other sieve configurations may be used provided they give equivalent results. It is desirable to use a set of sieves that have openings that are uniformly related on a logarithmic scale.

**6.3.2.1.3** *Mechanical sieve shaker*, capable of imparting a uniform rotary and tapping action.

**6.3.2.1.4** *Freezer*, any commercial ice freezer (a dry-ice chest may be used).

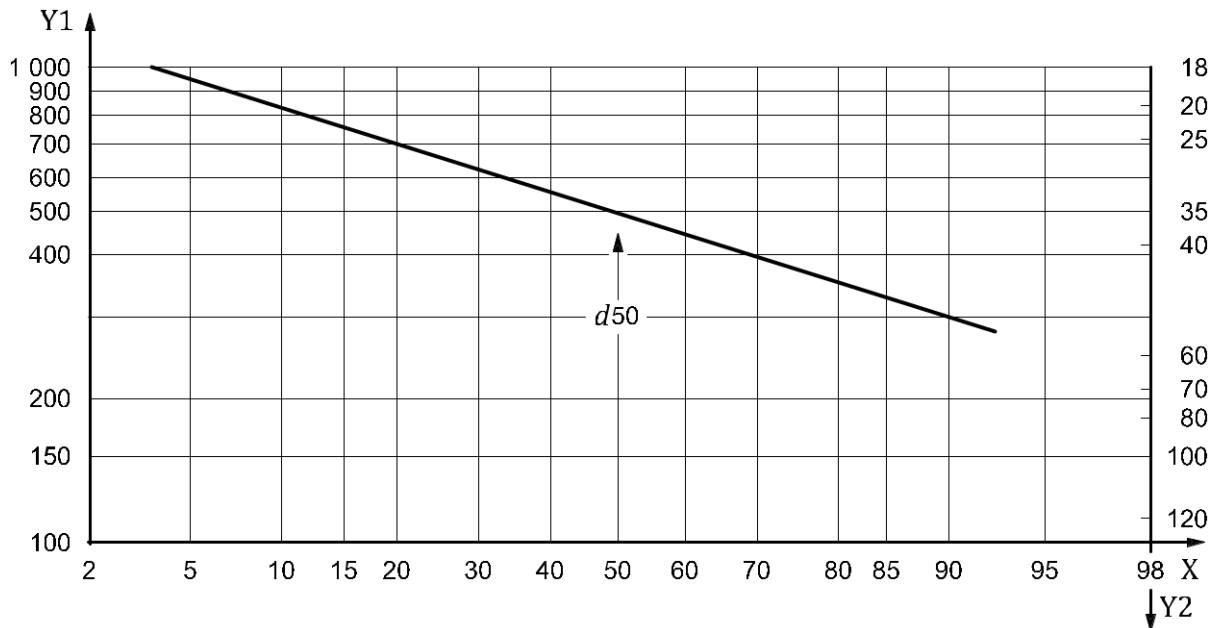


1	portable all-purpose shower head	8	13 mm ext. diam. glass tubing
2	stacked sieves	9	centrifugal pump capable of delivering 6 l/min at shower head
3	grating	10	clamp to adjust flow rate
4	support	11	all-plastic tubing, int. diam. 13 mm
5	13 mm diam. drain	12	sieve
6	20 l carboy	13	sheet of paper
7	perchloroethylene (or alternative solvent)	14	table top

a Use a fine sieve at the bottom of the stack to prevent material from going into the reservoir. A standard 38  $\mu$ m sieve has been found to be convenient.

All dimensions in millimetre.

**FIG. 8 APPARATUS FOR WET-SIEVE ANALYSIS**



X Cumulative percentage, percent

Y1 Sieve opening size,  $\mu\text{m}$

Y2 Sieve No.

Sieve No.	Sieve Opening Size ( $\mu\text{m}$ )
14	1 400
18	1 000
25	710
35	500
45	355
60	250
80	180

**FIG. 9 TYPICAL LOG/LOG PROBABILITY PLOT FOR SIEVE ANALYSIS**

### 6.3.2.2 Procedure

**6.3.2.2.1** Place  $50 \pm 0.1$  g of the sample in an aluminium pan, and cool the pan and contents to less than  $10^\circ\text{C}$ .

**6.3.2.2.2** Measure the tare mass of each of the sieves listed in **6.3.2.1.2**. Place the conditioned test sample on the top sieve of the assembly and shake in the sieve shaker for  $10 \pm 0.5$  min. The dewpoint temperature of the sieving room shall be less than the temperature of the conditioned test sample so that water will not condense on the test sample during the test. Determine the mass of resin retained on each sieve.

### 6.3.2.3 Expression of results

**6.3.2.3.1** Calculate the net percentage of resin on each sieve as follows:

$$\text{Net percentage of resin on sieve } Y = 2 \times m$$

where

$m$  = mass of resin on sieve  $Y$ , in g.

**6.3.2.3.2** Calculate the cumulative percentage of resin on each sieve as follows:

Cumulative percentage of resin on sieve  $Y$  = sum of net percentage on sieve  $Y$  and on sieves having sizes greater (i.e. numbers smaller) than sieve  $Y$ .

Example — Cumulative percentage on 500  $\mu\text{m}$  (No. 35) sieve equals net percentage on 1.4 mm (No. 14) plus net percentage on 1.0 mm (No. 18) plus net percentage on 710  $\mu\text{m}$  (No. 25) plus net percentage on 500  $\mu\text{m}$  (No. 35) sieve.

**6.3.2.3.3** Plot the cumulative percentage versus the sieve opening size (or sieve number) on log/log paper as shown in the sample plot in Fig 9. The sieve numbers and sieve opening sizes in micrometres are indicated below the figure. Draw the best straight line through the points and read the particle size at the 50 percent cumulative percentage point ( $d_{50}$ ). Take this value as the average particle size. It is permissible to carry out the calculation of  $d_{50}$  by use of computer programmes that provide ‘best-fit’ analysis using linear regression procedures involving a log-normal model.

**6.3.3** *Particle Size and Size Distribution by the Coulter Principle Using a Resistance-Variation Tester*

**6.3.3.1** *Apparatus*

**6.3.3.1.1** *Electric sensing-zone particle counter*, with an orifice tube, such that most of the particles lie within its measurement range (2 % to 60 % of the orifice-tube diameter). Calibration of the instrument in absolute terms shall be done by the count-integration procedure on narrow distributions of standard latices (essentially monosized particles suspended in distilled water containing a surfactant) that are available from various sources. Poly (styrene-co-divinylbenzene) latices are particularly recommended.

**6.3.3.1.2** *Analytical balance*

**6.3.3.1.3** *Magnetic stirrer*

**6.3.3.1.4** *Ultrasonic tank*

**6.3.3.2** *Procedure*

**6.3.3.2.1** Prepare a solution of a non-ionic surfactant (*see* Note 1) (at a concentration of 0.2 g/l to 0.3 g/l in an aqueous electrolyte, such as a 1 percent (mass per unit volume) solution of sodium chloride or a special 1 percent (mass per unit volume) saline solution (*see* Note 2). For example, four drops of the surfactant in 500 ml of electrolyte solution is sufficient to obtain a satisfactory suspension of the powder with moderate foaming. Filter the solution using a 0.3  $\mu\text{m}$  barrier filter (*see* Note 3).

NOTES

**1** Triton X-10 0, a surfactant of the octylphenol series, has been found to be satisfactory. Other similar materials should be equally effective. This information is given for the convenience of users and does

not constitute an endorsement. Equivalent products may be used if they can be shown to lead to the same results.

2 A specially prepared solution, known as Isoton, is available from Coulter Counter Ltd. This information is given for the convenience of users and does not constitute an endorsement. Equivalent products may be used if they can be shown to lead to the same results.

3 Available from the Millipore Corporation. This information is given for the convenience of users and does not constitute an endorsement. Equivalent products may be used if they can be shown to lead to the same results.

**6.3.3.2.2** Weigh 100 mg of powder into a 100 ml beaker and add 40 ml of the surfactant solution.

**6.3.3.2.3** Stir the slurry of powder using the magnetic stirrer at a high speed to ensure good wetting and deagglomeration of the powder. The proper dispersion conditions for a given mixer should preferably be determined in advance by plotting the average powder-particle diameter versus the speed and time used for mixing. It is important to avoid grinding of the powder (decrease in size of the ultimate particles). A typical set of conditions might involve stirring the slurry for 2 min to 3 min followed by 15 min at 700 r/min to 750 r/min.

**6.3.3.2.4** Put the beaker into the ultrasonic tank, ensuring that the level of the suspension coincides with the level of water in the tank. The processing time and the frequency of the ultrasonic treatment are selected to avoid any undesirable effects of warming on the morphology of the suspended particles (e.g. fracture of the particles). An ultrasonic cell disrupter operated at 20 kHz for 4 min should be sufficient to obtain a uniform suspension of the fluoropolymer powder.

**6.3.3.2.5** Resume stirring with the magnetic stirrer just rapidly enough to maintain a uniform suspension while removing 10  $\mu$ l to 200  $\mu$ l of the suspension, depending on the concentration. The analysis is made in a special 200 ml round-bottom container filled with the special 1 percent (mass per unit volume) saline solution described in **6.3.3.2.1**. Be careful to avoid the formation of air bubbles when filling.

**6.3.3.2.6** Count particles in time mode using three measurements.

**6.3.3.2.7** Correct the diameter as read to take into account the fraction with a diameter smaller than the last channel.

### **6.3.3.3** *Expression of results*

Plot a volume or mass percent curve on suitable graph paper as described for sieve analysis in **6.3.2.3.3**.

## **6.3.4** *Particle Size and Size Distribution by Light Scattering of a Laser Beam*

### **6.3.4.1** *Apparatus*

The particle-size analyser shall be based on Fraunhofer diffraction, Mie scattering or a combination of both light-scattering analysis techniques. Care shall be taken to ensure that the analyser system or subsystem is optimum for the size range of the powder being tested.

#### 6.3.4.2 Procedure

Follow the instructions from the manufacturer of the instrument unless there is a standard method available for the material being tested.

### 6.4 Tensile Properties

#### 6.4.1 Non-Melt Fluoropolymers (Fluoropolymers for which Tensile Modulus is not to be Determined)

For testing tensile properties, PTFE test specimens are prepared by method as described in section 4.1 to make PTFE skived film (equal to less than 0.125 mm in thickness) or PTFE molded disc. From these skived film or molded discs, prepare a minimum of five specimens using the microtensile die shown in Fig 3, section 4.2.

#### 6.4.2 Melt Processable Fluoropolymers (Fluoropolymers for which Tensile Modulus is to be Determined)

**6.4.2.1** Determine the tensile properties in accordance with the procedures described in IS 13360 (Part 5/Sec 1) except that the specimens used shall be as detailed in 4.2 and cut using microtensile die as shown in Fig 3 with crosshead speed of  $50 \pm 5$  mm/min. For determination of tensile modulus, use a crosshead speed of 1 mm/min.

#### 6.4.3 Testing method for determination of tensile properties for specimens mentioned in 6.4.1 and 6.4.2

Determine the tensile properties in accordance with the procedures described in IS 13360 (Part 5/Sec 1) except that the specimens used shall be as detailed above, the initial jaw separation shall be  $22.0 \pm 0.13$  mm, and the speed of testing shall be  $50 \pm 5$  mm/min. Clamp the specimens with an essentially equal length in each jaw. Determine the elongation from the recorder chart, expressing it as a percentage of the initial jaw separation. In determining elongation from the chart, draw a perpendicular line from the break point to the time axis. Measure the distance along the time axis from the foot of this perpendicular line to the beginning of the load-time curve. Optionally, an extensometer may be used to determine the elongation.

Calculate the percentage elongation using the following equation:

$$\text{percentage elongation} = \frac{100 * d}{22 * m}$$

where

d = Distance, in mm, on the chart;

m = Chart-speed magnification [= chart speed/ crosshead speed (both in same units)];  
and

x22.0 = Factor allowing for the fact that d is in mm.

### 6.5 Powder-Flow Time

The powder-handling characteristics of powders of polytetrafluoroethylene are critical in many of the procedures used to process these materials. This method is a procedure for determining

the flow characteristics of granular PTFE powder by quantifying the time required for a given volume of material to pass through an orifice vibrated under specified conditions. The method is particularly applicable to PTFE-S and PTFE-SS as defined in IS 14635 (Part 1).

**6.5.1 Apparatus**

**6.5.2 Feeder**, complying with 6.2.1.2.

**6.5.3 Funnel**, complying with Fig.10, constructed of aluminium, and electroplated with a regular chromium coating as described in A-2.1 of BS 4641, to a thickness of 12  $\mu\text{m}$ .

**6.5.4 Controller**, complying with 6.2.1.3.

**6.5.5 Vibrator**, capable of vibrating the funnel at 50 cycles per second with an amplitude of  $0.75 \pm 0.25$  mm.

**6.5.6 Volumetric Cup and Cup Stand**, as Detailed in Fig 11. The top and bottom of both cup and stand shall be flat and parallel to within 0.05 mm. The inside bottom corner of the cup shall be square, as shown in the figure, and the bottom of the hole in the cup stand shall be square with the centreline. The cup shall be adjusted to a volume of  $100 \pm 1.0$  ml. All external sharp corners of the cup stand shall be removed.

**6.5.7 Work Surface**, which shall be free from vibration. The feeder shall be mounted on an adjoining table or wall bracket.

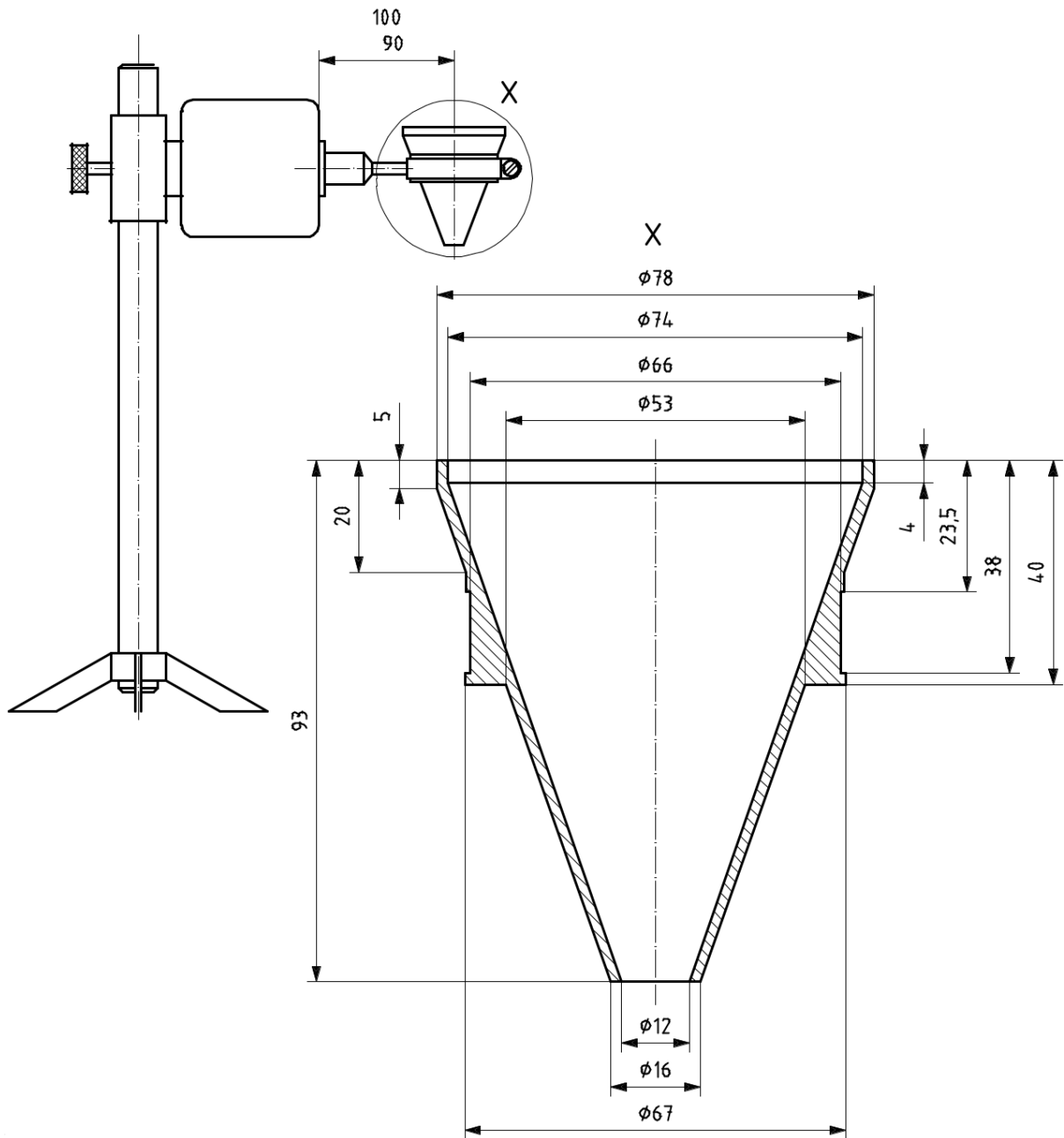
**6.5.8 Timing Device**, capable of recording to the nearest 0.1 s the time taken for the material to flow through the orifice.

**6.5.9 Levelling Device**, complying with 6.2.1.5.

**6.5.10 Conditioning**

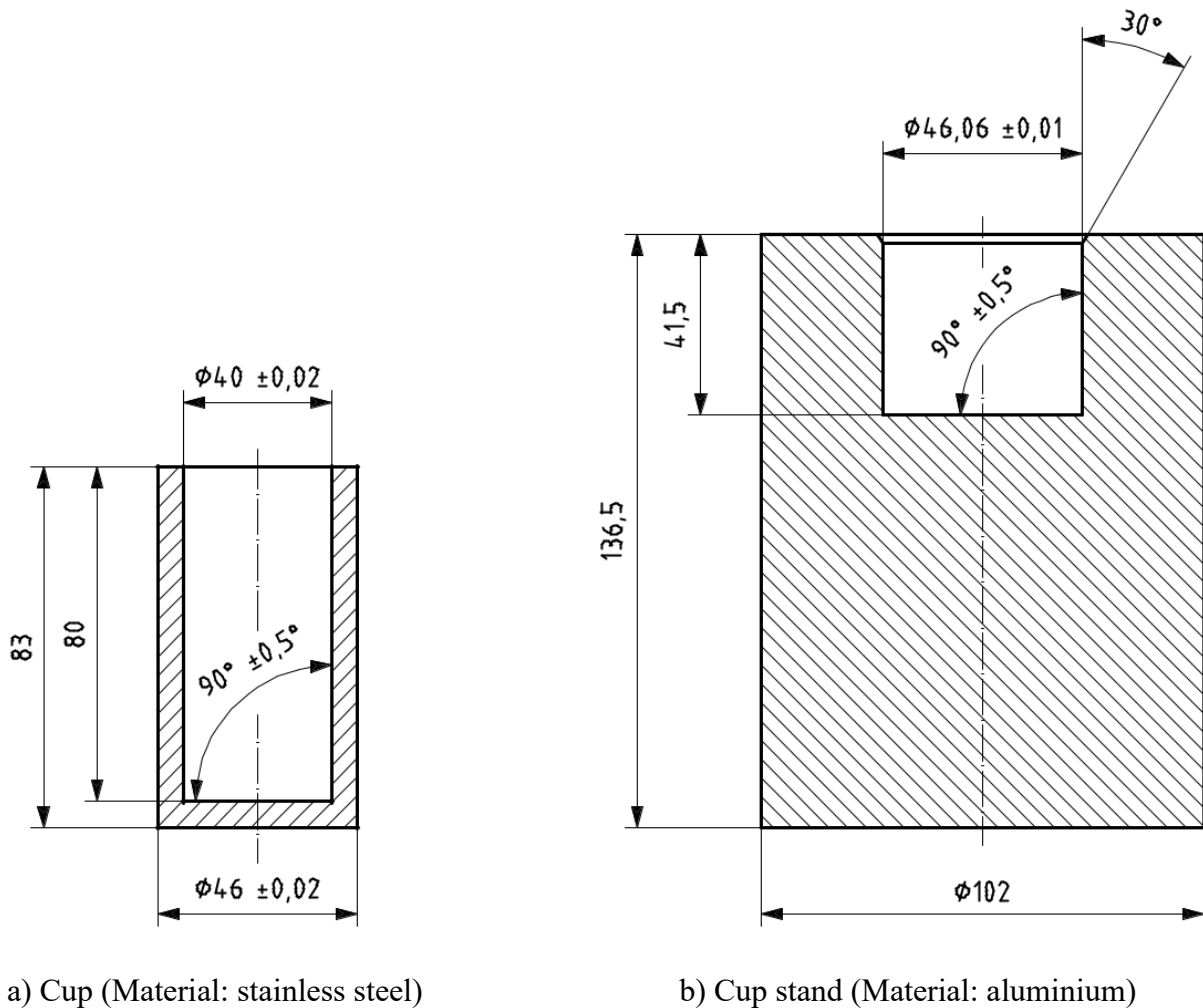
The material shall be conditioned for not less than 6 h at  $23 \pm 2$  °C and  $50 \pm 5$  percent R.





All dimensions in millimetres.

**FIG. 10 FUNNEL FOR DETERMINATION OF FLOW TIME**



All dimensions in millimetres.

**FIG.11 VOLUMETRIC CUP AND CUP STAND FOR DETERMINATION OF FLOW TIMES**

### 6.5.11 Procedure

**6.5.11.1** Select a test sample of material of about 200 ml. Place the sample on the feeder sieve (see 6.5.2). Vibrate all of the powder through the sieve and back into the sample container twice to break up any lumps. Put the cup in the stand (see 6.5.6) and place the assembly on the work surface (see 6.5.7) such that the distance the free powder falls from the feeder outlet to the top rim of the cup is  $31.8 \pm 3.2$  mm. Set the controller (see 6.5.4) so that the cup will be filled in 20 s to 30 s. Pour the test sample on to the vibrating sieve and fill the cup until the polymer forms a mound and overflows.

**6.5.11.2** Let the powder settle for 15 s and then gently push the cup and its stand beneath the leveller blade (see 6.5.9).

**6.5.11.3** Close the funnel orifice manually and insert the 100 ml of powder from the volumetric cup. Start the vibrator (see 6.5.5). Open the orifice and at the same time start the timing device (see 6.5.8). Record the time, to the nearest 0.1 s, for the material to flow through the orifice.

#### 6.5.12 Precision and Bias

The precision of this method has not yet been determined. There are no recognized standards on which to base an estimate of bias for this test procedure.

### 6.6 Melting-Peak Temperature

**6.6.1** Test samples/specimens for melting-peak temperature determination may be powder as received, dried polymer isolated from a dispersion, or the required amount cut from a pellet or fabricated piece of the resin as sold or received. The test shall be determined on a  $10 \pm 2$  mg specimen of dry polymer. It is desirable, but not essential, to test two specimens, each being run twice, using both a heating and a cooling cycle. Melting-peak temperature characteristics are specific for fluoropolymers and help identify a particular material. The procedures of ASTM D 4591 or ISO 11357-3 are appropriate for this determination.

**6.6.2** Use differential scanning calorimetry (DSC) as described in ISO 11357-3 and ASTM D 4591 for this determination. The heating rate shall be  $10 \pm 1$  °C/min. Two peaks during the initial melting test are observed occasionally. In this case, report the peak temperatures as  $T_l$  for the lower temperature and  $T_u$  for the upper temperature. Report the temperature corresponding to the peak largest in height as the melting point if a single value is required. If a peak temperature is difficult to discern from the curves — that is, if the peak is rounded rather than pointed — draw straight lines tangentially to the sides of the peak. Take the temperature corresponding to the point where these lines intersect beyond the peak as the peak temperature.

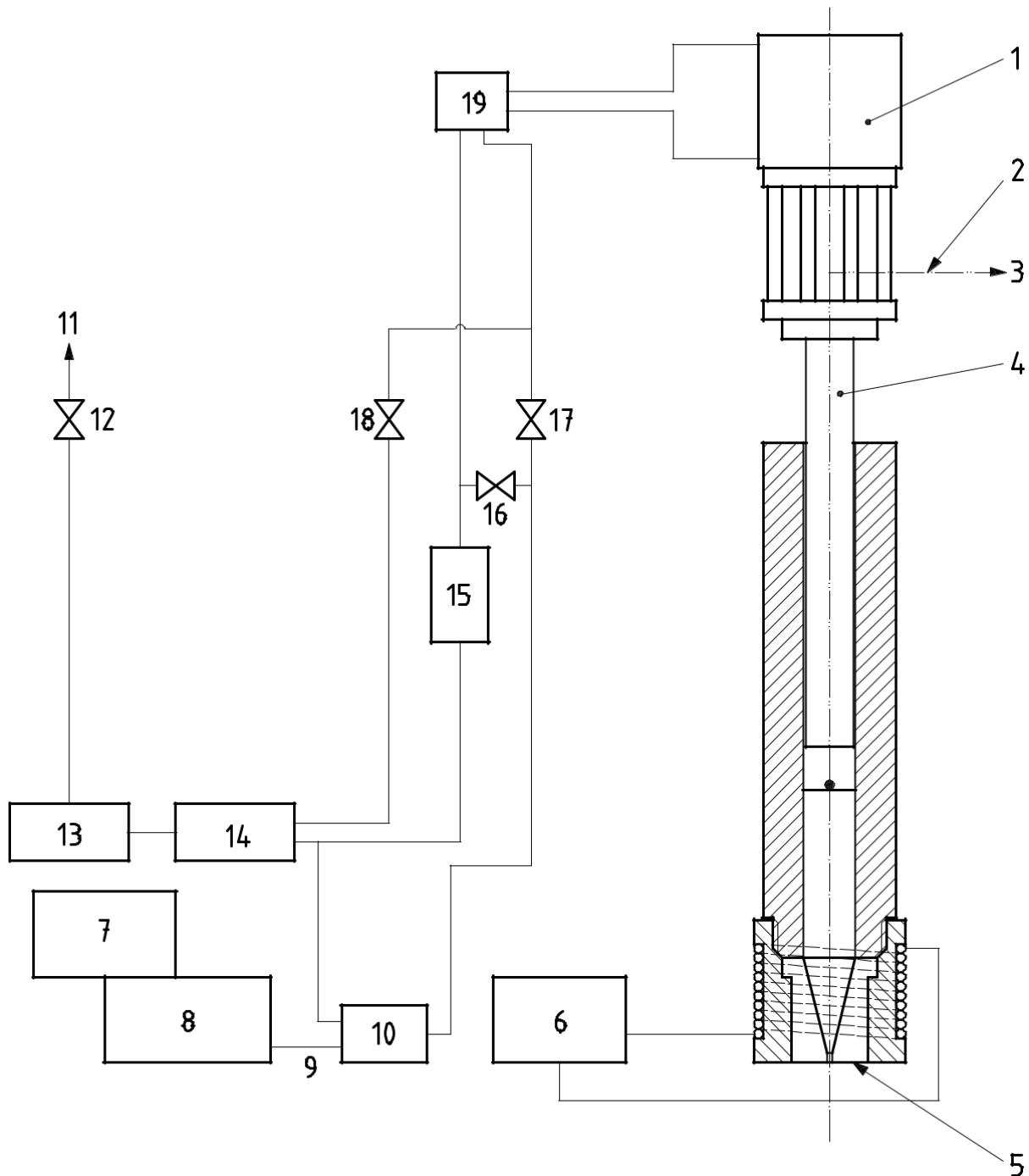
### 6.7 Extrusion Pressure

Processing of coagulated-dispersion PTFE resins normally involves ‘paste extrusion’ or ‘lubricated extrusion’ of a blend of the resin with a volatile liquid. The pressure that must be applied to such a blend to extrude it is affected by several processing conditions which include the nature and amount of deformation imparted to the blend during extrusion (usually characterized by the reduction ratio), the type and amount of liquid used, and the extrusion temperature. When such a blend is extruded under well-defined processing conditions, the pressure required to effect extrusion (the extrusion pressure) provides significant characteristic information about the resin itself that distinguishes among various, otherwise similar, materials.

#### 6.7.1 Apparatus (*Equivalent Apparatus may be Substituted*)

**6.7.1.1 Paste extruder** (*see Fig 12*), One paste extruder which may be used is a vertically disposed, breech-loading extruder with a 31.8 mm inside diameter extrusion cylinder. The barrel length is approximately 305 mm, which is not critical so long as it will hold enough lubricated resin to extrude for about 5 min. The ram is 31.6 mm in outside diameter, with a ring groove near its free end to hold an O-ring that makes a tight seal between the ram and the extruder cylinder. The extruder is equipped with devices for sensing and recording the pressure at the face of the ram. The range of the pressure transducer in the ram face is greater than 69 MPa. Temperature-controlling equipment maintains the extruder at  $30 \pm 1$  °C. A hydraulic system drives the ram at a speed of about 18 mm/min to give an output rate of 19 g/min on a dry-resin basis (about 23.5 g/min of lubricated resin) during the extrusion-pressure test. The

extruder also has a fast-speed drive (speed not precisely controlled) to run the ram rapidly into the cylinder cavity prior to the extrusion-pressure test. The extruder-die assembly slides on tracks from under the ram to allow easy access for cleaning the cylinder. An alternative muzzle-loaded paste extruder may be used which has a detachable die assembly. The die assembly is detached, a preformed charge of resin is inserted up into the cylinder and the die assembly is reattached.



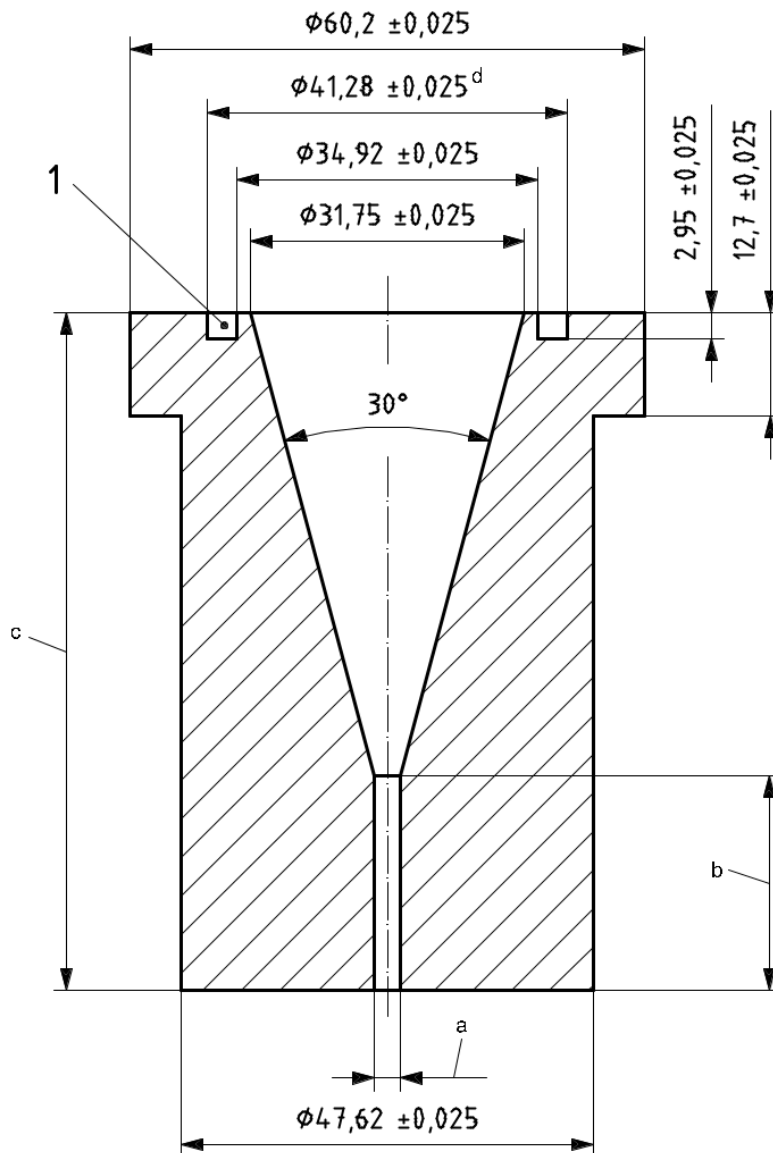
1	hydraulic cylinder	7	drive motor	13	air motor
2	pressure probe	8	link belt drive	14	air pump
3	to recorder	9	gear reducer	15	reservoir
4	ram	10	pump	16	safety valve

5	die	11	to air supply	17	slow speed
6	constant-temperature bath	12	air-motor valve	18	rapid speed
				19	Piston with two valve

**FIG. 12 PASTE EXTRUDER FOR DETERMINING EXTRUSION PRESSURE**

**6.7.1.2 Interchangeable Extrusion Dies** (see Fig 13), each having a 30° included angle and dimensions as indicated in Fig 12

NOTE — The reduction ratio in this specification is the ratio of the cross-sectional area of the extruder cylinder to the cross-sectional area of the die. This must not be confused with another definition wherein the reduction ratio is the ratio of the cross-sectional area of the extruder cylinder to the cross-sectional area of the sintered extrudate.



1	groove for O-ring
---	-------------------

a	Die orifice (see table)
b	Land length (see table)
c	Die length (see table)
d	Depending on O-ring dimensions

Reduction ratio	Die-orifice diameter	Land length	Die length
100 : 1	3.18	25.35	78.66
400 : 1	1.59	4.78	61.06
1 600 : 1	0.79	0.38	58.15

All dimensions in millimetres.

**FIG. 13 CROSS-SECTIONAL VIEW OF CYLINDRICAL DIE FOR EXTRUSION-PRESSURE APPARATUS**

**6.7.1.3** *Miscellaneous Apparatus*, for weighing, blending, conditioning (at 30 °C) and preforming, as well as for cleaning the extruder.

**6.7.2** *Procedure*

**6.7.2.1** Screen the dry resin through a 4.75 mm (No. 4) sieve onto a clean, dry, lint-free sheet of paper.

**6.7.2.2** Transfer  $200 \pm 0.5$  g of the screened resin to a clean, dry glass jar about 92 mm in diameter (approx. 1 litre capacity) having an airtight closure, or into a V-blender of laboratory size.

**6.7.2.3** Determine the density of the lubricant, a kerosene-type hydrocarbon liquid (*see* Note 1). The density shall be determined at 25 °C using ASTM D 4052 that calls for the use of a commercial density meter that will give the density to four significant figures (*see* Note 2), or a technically equivalent procedure. Calculate the mass of lubricant required by multiplying the density by 60.00. Add the calculated mass  $\pm 0.01$  g of the lubricant to the resin in the jar or blender. It is convenient to make this addition while the jar containing the powder is on a balance that has a sensitivity at least as good as the  $\pm 0.01$  g required for the test. Avoid wetting the walls of the blending vessel with the liquid as this impairs mixing. When a jar is used, tape the lid in place to prevent loss of lubricant. Shake the jar briefly to minimize the wetting of the jar wall with liquid.

**6.7.2.4** Blend the mixture by placing the jar on rubber-coated mill rolls and rolling it at 30 r/min for  $25 \pm 5$  min, by fastening the jar to a “windmill” type blender (*see* Note 3) and blending for  $20 \pm 1$  min, or by blending the mixture in the V-blender for  $15 \pm 5$  min. If a V-blender has been used, drop the resin from it into a jar of approximately 1 litre capacity and seal the jar.

**6.7.2.5** After blending, store the jar with its contents at  $30 \pm 1$  °C for a minimum of 2h. A water bath has been found to be satisfactory. This enables the lubricant to diffuse to the interior of individual particles and surfaces not reached during the blending process.

**6.7.2.6** Place the proper extrusion die for the desired reduction ratio (*see* Fig.13) in the paste extruder.

**6.7.2.7** To perform the resin for a breech-loading paste extruder (*see* 6.7.1.1), slide the extruder-die assembly forward and mount a 31.8 mm inside diameter extension tube about 610 mm in length at the breech end of the extruder cylinder. Quickly pour the lubricated resin through a funnel into the extension and force the resin into the extruder cylinder with a tamping rod. Apply the force with hand pressure and a very slow, even stroke. To perform the resin for a muzzle-loading paste extruder (*see* 6.7.1.1), mount a 31.8 mm inside diameter preforming tube about 610 mm in length with its cross-section resting against a flat, smooth surface. Quickly pour the lubricated resin through a funnel into the tube and force the resin down in the tube. The force may be applied with a hydraulically controlled tamping device to compact the resin with a slow, even stroke to a min of 690 kPa on the resin. Remove the preform from the preforming tube, insert the preform up into the cylinder of the extruder and attach the die assembly.

**6.7.2.8** Use the fast- speed drive to run the ram down into the cylinder cavity. When the first bit of beading emerges from the orifice, stop the descent of the ram.

**6.7.2.9** Immediately change to slow-speed drive, start the pressure-recording system and extrude the lubricated resin at a rate of  $19.0 \pm 1.0$  g/min (dry-resin basis).

**6.7.2.10** Record the pressure developed at the face of the ram in contact with the resin in the cylinder as a function of time. The extrusion pressure is the average pressure required to extrude the sample as measured between the third and fourth minutes of the extrusion.

#### NOTES

1 Isopar K, available from Exxon Co., has been found suitable for this purpose. This grade is used because its relatively low volatility prevents loss of lubricant during use and transfer of the lubricated powder. This information is given for the convenience of users and does not constitute an endorsement.

2 A Mettler/Paar density meter has been found suitable for determining density to the required precision. This information is given for the convenience of users and does not constitute an endorsement.

3 A spinning-wheel mixer has been found suitable for this purpose. Originally used was one from Gilson. This information is given for the convenience of users and does not constitute an endorsement.

## 6.8 Stretching-Void Index (SVI)

This test method compares the unstrained specific gravity (USG) of a resin to its strained specific gravity (strained SG). General procedures are given in 6.8.1. The method of calculation of the USG is given in 6.8.1.4 and that of the strained SG is given in 6.8.1.6. The SVI gives one indication of the potential for induced-void content of a solid fabricated resin product in use. Such void content may contribute to susceptibility to the formation of cracks and failures under extreme stretching and stress or, in some environments, when stressed. Similar failures have also been associated, at times, with improper processing techniques.

### 6.8.1 Procedure

**6.8.1.1** Prepare test discs using the general method described in 4.1.1 but modified to take into account the specific conditions given in 6.8.1.2.

**6.8.1.2** Screen 29 g of PTFE resin through a 2.00 mm (No. 10) sieve into the die. Adjust the lower plug so that the resin can be levelled by drawing a straight edge in contact with the top of the die across the top of the die cavity. Insert the die in the press and apply pressure gradually (*see* Note below 6.1.3) until a pressure of 7 MPa is attained. Hold this pressure for 2 min, then increase the pressure to 14 MPa and hold for an additional 2 min. Remove the disc from the die. A wax pencil may be used to write an identification marking on the disc at this time. Proceed as given in 6.1.4 for sintering preforms. Use the same sintering conditions as those specified for determining the SSG.

**6.8.1.3** Remove all flash from those portions of these specimens that will be used for determining specific gravities so that no air bubbles will cling to their edges when the specimens are immersed in liquid during these tests. It is recommended that cotton gloves be worn while handling test specimens.

**6.8.1.4** Determine the USG in accordance with 6.1.5, but using the specimen prepared as instructed in 6.8.1.1 and 6.8.1.2.

**6.8.1.5** Cut tensile specimens from the disc, using the microtensile die described in 6.4.1.2 and Fig 3. Clamp a specimen in a tensile-testing machine with essentially equal lengths in each jaw. The initial jaw separation shall be  $12.5 \pm 0.1$  mm. Strain the specimen at a constant rate of 5.0 mm/min until it breaks. This initial jaw separation and separation rate yield a strain rate of 40 percent/min, based on the original gauge length of the specimen. If elongation at break is less than 200 percent, discard the result and repeat with a fresh tensile specimen.

**6.8.1.6** Wait at least ten minutes after release of the stress in 6.8.1.5. Then cut off a portion of the stretched part of the specimen having a mass of at least 0.2 g. Determine, in accordance with 6.1.5, the specific gravity of this strained specimen (strained SG).

### 6.8.2 Expression of Results

Calculate the stretching-void index (SVI) from the equation:

$$\text{SVI} = (\text{USG} - \text{strained SG}) \times 1\,000$$

## 6.9 Testing of Fluoropolymer Dispersions

### 6.9.1 Preparation of Test Samples

Solids in the fluoropolymer dispersion tend to settle upon standing. Therefore, homogenize the dispersion by gentle mixing before sampling. Gentle mixing can be accomplished by rolling a



drum for 5 min at 3 r/min to 4 r/min, by stirring with a smooth rod for 3 min to 4 min, or by other types of gentle agitation.

**WARNING** — Excessive agitation can coagulate the dispersion.

### 6.9.2 Percentage fluoropolymer solids and surfactant in aqueous dispersion

#### 6.9.2.1 Fluoropolymer solids and surfactant content by mass loss

Percentage fluoropolymer solids and the percentage surfactant can be determined by successive evaporations of water and surfactant. The percentage surfactant is based on the mass of fluoropolymer present in the dispersion. All percentages are based on mass.

#### 6.9.2.2 Apparatus

An aluminium weighing dish, an oven capable of reaching  $120 \pm 5$  °C, an oven capable of reaching  $380 \pm 10$  °C, a desiccator and a balance capable of weighing to 0.1 mg.

#### 6.9.2.3 Procedure

Weigh the aluminium weighing dish to 0.1 mg ( $m_1$ ). Add 10 g of fluoropolymer dispersion and reweigh immediately to 0.1 mg ( $m_2$ ). Dry the test portion for 2 h at  $120 \pm 5$  °C. Reweigh the test portion to 0.1 mg ( $m_3$ ) after cooling to room temperature in the desiccator. After weighing, evaporate the surfactant by placing the test portion in an oven at  $380 \pm 10$  °C for  $35 \pm 1$  min. Allow the sample to cool in the desiccator to room temperature and weigh to 0.1 mg ( $m_4$ ).

#### 6.9.2.4 Expression of Results

For surfactants that are completely volatile, use Formulae (1) and (2)

$$\text{Fluoropolymer solids percent} = \frac{W_D - W_A}{W_B - W_A} \quad \dots (1)$$

$$\text{surfactant percent} = \frac{W_C - W_D}{W_D - W_A} \quad \dots (2)$$

For surfactants that are not completely volatile, use Formulae (3) and (4)

$$\text{Fluoropolymer solids percent} = \frac{(m_3 - m_1) - (m_3 - m_4)(1 + k)}{m_2 - m_1} * 100 \quad \dots (3)$$

$$\text{surfactant percent} = \frac{(m_3 - m_4)(1 + k)}{m_2 - m_1} * 100 \quad \dots (4)$$

where

$k$  = Mass of the non-volatile portion of the surfactant divided by the mass of the volatile portion of the surfactant.

NOTE — Upon request, the supplier shall inform the user whether the surfactant can be completely removed by the procedures of 6.9.2.3 and, if not, the manufacturer shall define the surfactant or the volatile and non-volatile portions of the surfactant.

## 6.10 Melt Mass-Flow Rate (MFR)

Melt mass-flow rate or melt volume-flow rate shall be determined in accordance with ISO 1133-1 as modified by details provided in this standard. Use of automated or other instruments that have been shown to provide equivalent results shall be an acceptable alternative to the detailed procedures given in this standard.

### 6.10.1 Test Conditions

The melt-flow rate is determined using the conditions for the fluoropolymer type shown in Table 3 and using a modification of the extrusion plastometer described in ISO 1133-1. The sample may be pellets or powder. For use with semi-finished forms or moulded articles, pieces of approximately the same size may be cut from a moulded or extruded form. Strips may also be handled conveniently.

### 6.10.2 Apparatus

The apparatus shall consist of an extrusion plastometer, as described in ISO 1133-1 but modified by use of corrosion-resistant alloy for the barrel lining, orifice and piston tip (*see Note*).

The usual orifice dimensions of 2.095 mm in diameter by 8.0 mm long are used except for some grades of ETFE when the orifice is 1.588 mm by 6.070 mm. Automated or other apparatus that have been shown to give equivalent results may be used in place of the apparatus described here.

NOTE — ‘Stellite’ Grade No. 18 and ‘Duranickel’ No. 301 have been found resistant to fluoropolymer resins for this application. This information is given for the convenience of users and does not constitute an endorsement. Equivalent products may be used if they can be shown to lead to the same results.

### 6.10.3 Procedure

#### 6.10.1.3.1 Calibration of instrument for temperature

The specified melt temperature is the temperature measured in the melt 12.7 mm above the orifice. This temperature may be obtained by controlling the temperature measured in the thermometer well at a temperature approximately 8 °C above the required temperature. Prior to making a test, set the plastometer temperature as follows. With the orifice in place, insert a standardized thermocouple (*see Note*) through the orifice from the bottom of the viscometer to a point 12.7 mm above the top of the orifice. Charge 5 g of resin granules into the plastometer, compact with the piston, and wait for  $10 \pm 0.5$  min for the melt temperature to reach equilibrium. Make the necessary adjustments in the temperature controller to bring the melt temperature to the level required for the particular polymer  $\pm 1$  °C. See Table 3 for the

conditions specified for each fluoropolymer. Repeat this calibration procedure and record temperature versus time at 1 min intervals for the first 10 min. The polymer should reach the required temperature within 5 min. With polymer in the plastometer for an elapsed time of 10 min for each point measured, determine the melt temperature at 6.4 mm intervals over the range from 6.4 mm to 51 mm above the orifice. The entire temperature profile shall be within a range of 2 °C. This precision is readily obtained by proper insulation of the sides, bottom and top of the plastometer.

**Table 3 Test Conditions for Melt Flow Rate Determinations**  
(Clause 6.10.1)

Sl No.	Fluoropolymer	Test Temperature (codes in brackets) °C	Load 1) (codes in brackets) kg	Orifice Diameter mm	Orifice Length mm
(1)	(2)	(3)	(4)	(5)	(6)
i)	FEP	372 (1)	2.16 or 5.0 (C or E)	2.095 ± 0.005	8.000 ± 0.025
ii)	PFA	372 (1)	5.0 (E)	2.095 ± 0.005	8.000 ± 0.025
iii)	ETFE	297 (2)	5.0 (E)	2.095 ± 0.005 or 1.588 ± 0.005	8.000 ± 0.025 or 6.070 ± 0.025
iv)	EFEP	265 (4)	5.0 (E)	2.095 ± 0.005	8.000 ± 0.025
v)	PVDF	230 (5)	2.16 or 5.0 (C or E)	2.095 ± 0.005	8.000 ± 0.025
vi)	VDF/CTFE	230 (5)	2.16 or 5.0 (C or E)	2.095 ± 0.005	8.000 ± 0.025
vii)	VDF/HFP	230 (5)	2.16 or 5.0 (C or E)	2.095 ± 0.005	8.000 ± 0.025
viii)	VDF/TFE	297 (2)	5.0 (E)	2.095 ± 0.005	8.000 ± 0.025
ix)	VDF/TFE/HFP	265 (4)	5.0 (E)	2.095 ± 0.005	8.000 ± 0.025
x)	PCTFE	265 (4)	21.6 or 31.6 (H or I)	2.095 ± 0.005	8.000 ± 0.025
xi)	CPT	297 (2)	5.0 (E)	2.095 ± 0.005	8.000 ± 0.025
xii)	ECTFE	271.5 (3)	2.16 (C)	2.095 ± 0.005	0.025

<sup>1)</sup> It is preferable to use the loads in this table. In some situations, however, it can be desirable to select a different load, chosen from Table 4, in order to comply with the recommendation of ISO 1133-1 that the measured melt flow rate should not be less than 0.1 g/10 min or in order to avoid having to follow the special procedure specified in ISO 1133-1 for melt flow rates greater than 100 g/10 min.

**Table 4**  
**Permissible Loads, in kilograms, for Melt Flow Rate Determinations (Preferred Loads in Heavy Type, Codes in Brackets)**  
(Table 3)

SI No.	Permissible Loads, in kg (Codes in Brackets)
(1)	(2)
i)	0.325 (A)
ii)	1.20 (B)
iii)	2.16 (C)
iv)	3.8 (D)
v)	5.0 (E)
vi)	10.0 (F)
vii)	12.5 (G)
viii)	21.6 (H)
ix)	31.6 (I)

NOTE — Suitable standards for calibrating thermocouples are: lead, m.p. 327 °C; potassium dichromate, m.p. 398.0 °C; and zinc, m.p. 419.4 °C.

#### 6.10.1.3.2 Measurement of melt mass-flow rate (MFR)

Make sure that the instrument is clean and level and that a clean orifice of the appropriate size is in place (*see* Table 3). Check, as described above, that the temperature of the plastometer is such that the melt temperature will be as specified. The controller settings shall be such that the heater is on and off for approximately equal periods. Charge  $5.0 \pm 0.5$  g of the sample. If the sample is in the form of granules, pour it into the plastometer through the funnel and push it down with the charging rod. As soon as the sample has been charged, wipe off the top of the instrument and place the piston in position by moving it downwards until resistance is met. This will compact the sample. Start a stopwatch. Allow the polymer to heat for exactly 5 min (by the stopwatch) to obtain equilibrium conditions. Stop and reset the stopwatch. Place suitable weights so that the total load on the plastometer meets the requirements of Table 3. Allow the polymer to extrude for 30 s by the stopwatch and then, without stopping the watch, cut off the extruded portion cleanly with a spatula at the exact moment that the second hand of the watch reaches 60 s. Discard this portion. In order to obtain a clean-cut, pass the tip of the spatula upwards along one side of the bevelled hole, then lightly across the bottom of the hole. This cutting should be done quickly and neatly in order to obtain the best precision. A light cutting force should be used to avoid excessive wear on the orifice opening. The neatness of the cut may be checked by observing the manner in which the succeeding portion is extruded. If the cut is clean and sharp, the succeeding portion will be extruded straight. If not, it will tend to curl and stick to one side or the other of the bevelled hole. Reshape the end of the spatula slightly to obtain the best results. Collect five successive cuts at half-minute intervals. After the extruded portions have cooled to room temperature, weigh the individual cuts to the nearest 1 mg. Compute the flow rate in grams per 10 min by multiplying the average mass of the five cuts by 20.

NOTES

- 1 The extrusion plastometer can be equipped with a device for automatically cutting off the extruded sample at pre-set time intervals.
- 2 The intervals between successive cut-offs can be chosen depending on the melt mass-flow rate. The footnotes to ISO 1133-1, Table 3 provide guidance on selecting test conditions.

## **6.11 Density**

**6.11.1** Cut two specimens from the moulding or other solid sample and determine the density in accordance with one of the methods described in IS 13360 (Part 3/Sec 10) or IS 13360 (Part 3/Sec 11). If IS 13360 (Part 3/Sec 11) is used, the liquid system used shall have a density gradient appropriate for the fluoropolymer being tested [*see* Table A.1 in IS 13360 (Part 3/Sec 11)]. The use of IS 13360 (Part 3/Sec 11) is discouraged, however, due to the carcinogenicity of the liquids used.

## **6.12 Dielectric Strength of Fluoropolymers**

Determine this property in accordance with the procedures of IEC 60243-1: 2013. Codes for values of the property are given in IS 14635 part 1.