
**Plastics — Fluoropolymer dispersions
and moulding and extrusion materials —**

**Part 2:
Preparation of test specimens
and determination of properties**

*Plastiques — Polymères fluorés: dispersions et matériaux pour
moulage et extrusion —*

Partie 2: Préparation des éprouvettes et détermination des propriétés

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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12086-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 12086-2:1995), which has been technically revised.

ISO 12086 consists of the following parts, under the general title *Plastics — Fluoropolymer dispersions and moulding and extrusion materials*:

- *Part 1: Designation system and basis for specifications*
- *Part 2: Preparation of test specimens and determination of properties*

Plastics — Fluoropolymer dispersions and moulding and extrusion materials —

Part 2: Preparation of test specimens and determination of properties

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory requirements. The warnings in Subclauses 8.6.2.1, 9.7 and 10.6.1.4 point out specific hazards.

1 Scope

1.1 This part of ISO 12086 describes the preparation of test specimens and provides test methods to define characteristics of thermoplastic fluoropolymer resins. Results from the testing may be used as the basis for designation, material specifications or both. This part of ISO 12086 describes the conditions of test for determining both designatory and other properties of the homopolymers and various copolymers of fluoromonomers, as dispersions or powders for moulding, extrusion and other uses. The test procedures included are appropriate for, but are not restricted to, the fluoropolymers listed in Clause 4 and for which designatory properties are specified in ISO 12086-1.

1.2 The properties of semi-finished and finished products made from fluoropolymer resins depend on the material used, the shape of the product, the physical and morphological state of the material resulting from the processing operations, and on the test conditions. Therefore, to obtain reproducible test results, the defined methods of preparation of test specimens and defined test conditions given in this part of ISO 12086 must be applied.

1.3 Agreements between vendor and purchaser should preferably be based on properties measured using the specimens and test conditions described in this part of ISO 12086.

2 Normative references

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

ISO 75-2, *Plastics — Determination of temperature of deflection under load — Part 2: Plastics and ebonite*

ISO 178, *Plastics — Determination of flexural properties*

ISO 179-1, *Plastics — Determination of Charpy impact properties — Part 1: Non-instrumented impact test*

ISO 180, *Plastics — Determination of Izod impact strength*

ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 293, *Plastics — Compression moulding of test specimens of thermoplastic materials*

ISO 472, *Plastics — Vocabulary*

ISO 527-1, *Plastics — Determination of tensile properties — Part 1: General principles*

ISO 527-2, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics*

ISO 527-3, *Plastics — Determination of tensile properties — Part 3: Test conditions for films and sheets*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 976, *Rubber and plastics — Polymer dispersions and rubber latices — Determination of pH*

ISO 1043-1, *Plastics — Symbols and abbreviated terms — Part 1: Basic polymers and their special characteristics*

ISO 1043-2, *Plastics — Symbols and abbreviated terms — Part 2: Fillers and reinforcing materials*

ISO 1133:2005, *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics*

ISO 1183-1, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pycnometer method and titration method*

ISO 1183-2, *Plastics — Methods for determining the density of non-cellular plastics — Part 2: Density gradient column method*

ISO 4589 (all parts), *Plastics — Determination of burning behaviour by oxygen index*

ISO 11357-2, *Plastics — Differential scanning calorimetry (DSC) — Part 2: Determination of glass transition temperature*

ISO 11357-3, *Plastics — Differential scanning calorimetry (DSC) — Determination of temperature and enthalpy of melting and crystallization*

ISO 12086-1, *Plastics — Fluoropolymer dispersions and moulding and extrusion materials — Part 1: Designation system and basis for specifications*

ISO 13320-1, *Particle size analysis — Laser diffraction methods — General principles*

IEC 60093, *Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials*

IEC 60243-1, *Electrical strength of insulating materials — Test methods — Part 1: Tests at power frequencies*

IEC 60250, *Recommended methods for the determination of the permittivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies including metre wavelengths*

ASTM D 746, *Standard Test Method for Brittleness Temperature of Plastics and Elastomers by Impact*

ASTM D 1430, *Standard Classification System for Polychlorotrifluoroethylene (PCTFE) Plastics*

ASTM D 1894, *Standard Test Method for Static and Kinetic Coefficients of Friction of Plastic Film and Sheeting*

ASTM D 3418, *Standard Test Method for Transition Temperatures of Polymers by Differential Scanning Calorimetry*

ASTM D 4052, *Standard Test method for Density and Relative Density of Liquids by Digital Density Meter*

ASTM D 4591, *Standard Test Method for Determining Temperatures and Heats of Transitions of Fluoropolymers by Differential Scanning Calorimetry*

ASTM D 4894, *Standard Specification for Polytetrafluoroethylene (PTFE) Granular Molding and Ram Extrusion Materials*

ASTM D 4895, *Standard Specification for Polytetrafluoroethylene (PTFE) Resin Produced from Dispersion*

BS 4641:1986, *Method for specifying electroplated coatings of chromium for engineering purposes*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following terms and definitions apply. The terms listed in 3.1 to 3.3 are repeated from ISO 472 to be sure there is no misunderstanding.

3.1

dispersion

heterogeneous system in which a finely divided material is distributed in another material

3.2

fluoroplastic

plastic based on polymers made with monomers containing one or more atoms of fluorine, or copolymers of such monomers with other monomers, the fluoromonomer being in the greatest amount by mass

3.3

latex

colloidal aqueous dispersion of a polymeric material

3.4

amorphous

noncrystalline, or devoid of regular structure

3.5

bulk density

mass (in grams) per litre of material, measured under the conditions of the test

3.6

copolymer

polymer formed from two or more types of monomer

3.7

emulsion polymer

(fluoropolymer materials) material isolated from its polymerization medium as a colloidal aqueous dispersion of the polymer solids

NOTE This definition, used in the fluoropolymer industry, is similar to that for "latex" in ISO 472 and is quite different from the definition for "emulsion" in ISO 472.

3.8

fluorocarbon plastic

plastic based on polymers made from perfluoromonomers only

3.9

fluoropolymer

synonymous with fluoroplastic (see 3.2)

3.10

melt-processible

capable of being processed by, for example, injection moulding, screw extrusion and other operations typically used with thermoplastics

3.11

preforming

compacting powdered PTFE material under pressure in a mould to produce a solid object, called a preform, that is capable of being handled

NOTE With PTFE, "moulding" and "compaction" are terms used interchangeably with "preforming".

3.12
sintering

(PTFE) thermal treatment during which the material is melted and recrystallized by cooling, with coalescence occurring during the treatment

3.13
standard specific gravity
SSG

specific gravity of a specimen of PTFE material preformed, sintered and cooled through the crystallization point at a rate of 1 °C per minute in accordance with the appropriate sintering schedule as described in this part of ISO 12086

NOTE The SSG of unmodified PTFE is inversely related to its molecular mass.

3.14
suspension polymer

polymer isolated from its liquid polymerization medium as a solid having a particle size well above colloidal dimensions

3.15
zero-strength time
ZST

measure of the relative molecular mass of PCTFE

4 Abbreviated terms and symbols

4.1 The abbreviated terms given in ISO 1043-1 and ISO 1043-2 are applicable to this part of ISO 12086.

4.2 This part of ISO 12086 is particularly concerned with, but is not limited to, test methods for the materials listed below:

ECTFE	ethylene-chlorotrifluoroethylene copolymer
EFEP	ethylene-tetrafluoroethylene-hexafluoropropene copolymer
ETFE	ethylene-tetrafluoroethylene copolymer
FEP	perfluoro(ethylene-propene) copolymer
PCTFE	polychlorotrifluoroethylene
PFA	perfluoro(alkoxy alkane)
PTFE	polytetrafluoroethylene
PVDF	poly(vinylidene fluoride)
PVF	poly(vinyl fluoride)
TFE/PDD	tetrafluoroethylene-perfluorodioxole copolymer
VDF/CTFE	vinylidene fluoride-chlorotrifluoroethylene copolymer
VDF/HFP	vinylidene fluoride-hexafluoropropene copolymer
VDF/TFE	vinylidene fluoride-tetrafluoroethylene copolymer
VDF/TFE/HFP	vinylidene fluoride-tetrafluoroethylene-hexafluoropropene copolymer

4.3 For the purposes of this part of ISO 12086, the following additional abbreviated terms apply.

AF	amorphous fluoropolymer
ESG	extended specific gravity (see 10.6)
MFR	melt mass-flow rate (see 11.2)
MVR	melt volume-flow rate (see 11.2)
SSG	standard specific gravity (see 10.6)
SVI	stretching-void index (see 10.7)
TII	thermal-instability index (see 10.6)
ZST	zero-strength time (see 12.3)

5 Sampling

Sampling shall be statistically adequate to satisfy the requirements of this part of ISO 12086.

6 Preparation of test specimens

Where applicable, 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.

7 Conditioning and test conditions

7.1 For determinations of specific gravity, tensile properties and electrical properties, condition the moulded test specimens in atmosphere 23 of ISO 291 for a period of at least 4 h prior to testing. The other determinations require no conditioning.

NOTE For PVDF, some producers recommend waiting one week after moulding before testing in order to minimize the effects of post-crystallization.

7.2 Conduct tests at a laboratory temperature of $23\text{ °C} \pm 2\text{ °C}$ for determining specific gravity, tensile properties and electrical properties only. (See the Note for comments related to PTFE.) Since the fluoropolymer resins do not absorb water, the maintenance of constant humidity during testing is not necessary. Conduct tests for melt flow rate and melting-peak temperature under ordinary laboratory conditions.

NOTE A minimum temperature of 22 °C should preferably be maintained with PTFE due to its first-order transition just below 22 °C that affects properties determined at slightly lower temperatures. This effect of temperature is especially important during the determination of density/specific gravity.

8 General testing of fluoropolymers

Properties required for designation or specification, or both, shall be determined in accordance with the international or national standards listed in Clause 2 or the procedures given in this part of ISO 12086.

Tables of values of the designatory properties and corresponding codes are included in ISO 12086-1.

Tables of values and codes are also included in this part of ISO 12086 for many of the other properties that are needed to supplement the designatory properties for specification and other purposes.

8.1 Electrical properties

8.1.1 Dielectric constant and dissipation factor

Determine these properties on three specimens, each 100 mm in diameter, in accordance with IEC 60250.

Typical frequencies used for testing are 100 Hz, 1 kHz, 1 MHz and 100 MHz. For some applications, it is important to know the values at subambient and elevated temperatures. Codes for test frequencies and values of the properties are given in Tables 1 and 2.

NOTE Electrical properties, like many other properties, vary with temperature.

Table 1 — Codes for test frequencies

Code	Test frequency
2	100 Hz
3	1 kHz
6	1 MHz
8	100 MHz

Table 2 — Codes and ranges for dielectric constant and dissipation factor

Code	Dielectric constant	Code	Dissipation factor
A	< 1,6	A	< 0,000 1
B	1,6 to < 1,8	B	0,000 1 to < 0,000 2
C	1,8 to < 2,0	C	0,000 2 to < 0,000 4
D	2,0 to < 2,2	D	0,000 4 to < 0,000 6
E	2,2 to < 2,4	E	0,000 6 to < 0,000 8
F	2,4 to < 2,6	F	0,000 8 to < 0,001 0
G	2,6 to < 2,8	G	0,001 0 to < 0,001 2
H	2,8 to < 3,0	H	0,001 2 to < 0,001 4
I	3,0 to < 3,2	I	0,001 4 to < 0,001 6
J	3,2 to < 3,4	J	0,001 6 to < 0,001 8
K	3,4 to < 3,6	K	0,001 8 to < 0,002 0
L	3,6 to < 4,0	L	0,002 0 to < 0,002 2
M	4,0 to < 4,5	M	0,002 2 to < 0,002 4
N	4,5 to < 5,0	N	0,002 4 to < 0,002 6
O	5,0 to < 5,5	O	0,002 6 to < 0,002 8
P	5,5 to < 6,0	P	0,002 8 to < 0,003 0
R	6,0 to < 6,5	Q	0,003 0 to < 0,003 5
S	6,5 to < 7,0	R	0,003 5 to < 0,004 0
T	7,0 to < 8,0	S	0,004 0 to < 0,006 0
U	8,0 to < 9,0	T	0,006 0 to < 0,008 0
V	9,0 to < 10,0	U	0,008 0 to < 0,010
W	10,0 to < 11,0	W	0,010 to < 0,030
X	11,0 to < 12,0	X	0,030 to < 0,10
Y	12,0 to < 14,0	Y	≤ 0,1
Z	≥ 14,0		

8.1.2 Dielectric strength (electric strength)

Determine this property in accordance with the procedures of IEC 60243-1. Codes for values of the property are given in Table 3.

NOTE Dielectric strength, which is expressed in kilovolts per millimetre, varies with the thickness of the test specimen.

Table 3 — Codes and ranges for dielectric strength

Code	Dielectric strength (kV/mm)
A	< 5
B	5 to < 10
C	10 to < 15
D	15 to < 20
E	20 to < 25
F	25 to < 30
G	30 to < 35
H	35 to < 40
I	40 to < 45
J	45 to < 50
K	50 to < 55
L	55 to < 60
M	60 to < 65
N	65 to < 70
O	70 to < 75
P	75 to < 80
Q	80 to < 85
R	85 to < 90
S	90 to < 95
T	95 to < 100
U	≥ 100

8.1.3 Surface resistivity

Determine this property in accordance with IEC 60093.

Codes and ranges are listed in Table 4.

Table 4 — Codes and ranges for surface resistivity

Code	Surface resistivity (Ω)
A	< 10^3
B	10^3 to 10^{12}
C	> 10^{12}

8.2 Mechanical properties

8.2.1 Impact properties

Determine impact properties using the procedures of ISO 180 for Izod impact strength and ISO 179-1 for Charpy impact strength. Codes and ranges are given in Table 5. The test used, the size of the test specimen and the type of notch shall be reported in addition to the code for impact strength.

Table 5 — Codes and ranges for impact properties

Code	Impact strength (J/m)
A	< 100
B	120 to < 140
C	140 to < 160
D	160 to < 180
E	180 to < 200
F	200 to < 300
G	300 to < 400
H	400 to < 500
I	500 to < 600
J	600 to < 700
K	700 to < 800
L	800 to < 900
M	≥ 900

8.2.2 Tensile properties

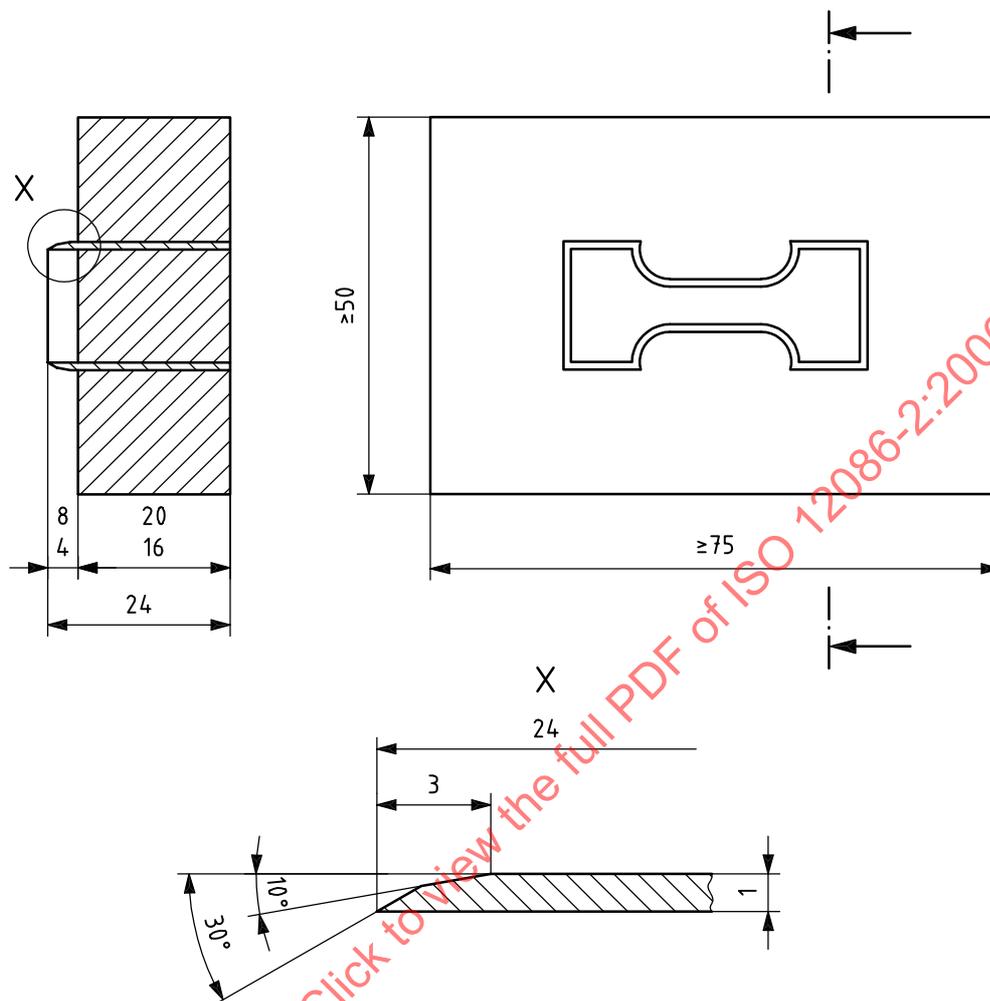
8.2.2.1 Fluoropolymers for which tensile modulus is not to be determined

8.2.2.1.1 PTFE skived film with a thickness equal to or less than 0,125 mm shall be tested in accordance with the procedure described in ISO 527-3, using test specimen type 2.

8.2.2.1.2 For test specimens other than the skived film referred to in 8.2.2.1.1 (equal to or less than 0,125 mm in thickness), prepare five specimens using the microtensile die described in Figure 1. The die shall be of the steel-rule type with a curvature of $5 \text{ mm} \pm 0,5 \text{ mm}^1$). Determine the tensile properties in accordance with the procedures described in ISO 527-1 except that the specimens used shall be as detailed above, the initial jaw separation shall be $22,0 \text{ mm} \pm 0,13 \text{ mm}$, and the speed of testing shall be $50 \text{ mm/min} \pm 5 \text{ mm/min}$. 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.

1) The steel-rule type of die has been found satisfactory for this purpose. Two sources for these steel-rule dies are: Stansvormenfabriek Vervloet B.V., Postbus 220, Gantelweg 15, 3350 AE Papendrecht, Netherlands, Tel.: +31 70 322 22 21, Fax: +31 70 322 22 24, and MS Laboratory Instruments, 28 Gateway Road, Fairport, NY 14450, USA, Tel: +1 585 377 2830, Fax: +1 585 388 1333. This information is given for the convenience of users of this part of ISO 12086 and does not constitute an endorsement by ISO of these products. Other sources may be available or a die may be constructed from details in Figure 1.

Dimensions in millimetres



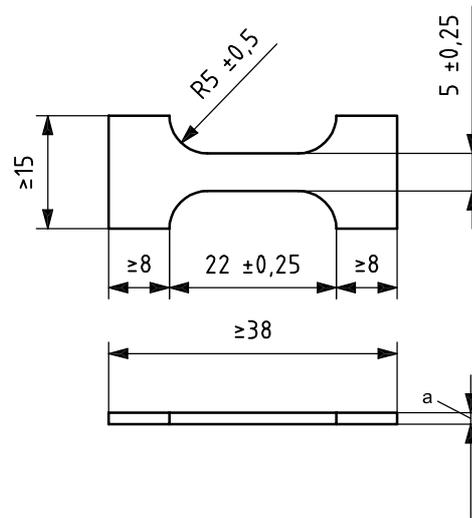
Inside dimensions of die are same as those of test specimen.

Die to be sharpened on outside of knife edge only (as shown in figure).

Rockwell C hardness of die: 45 to 50.

a) Steel-rule die

Figure 1 (continued on next page)



b) Micro-tensile specimen

- a Possible thicknesses:
- 1,5 ± 0,3
 - 0,8 ± 0,15
 - 0,5 ± 0,1
 - 0,125 ± 0,03.

Figure 1 — Knife-edged die for micro-tensile (type A) specimens, and punched-out specimen

Calculate the percentage elongation using the following equation:

$$\% \text{ elongation} = \frac{100d}{22,0m}$$

where

d is the distance, in millimetres, on the chart;

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

22,0 is a factor allowing for the fact that d is in millimetres.

8.2.2.2 Fluoropolymers for which tensile modulus is to be determined

Determine tensile properties in accordance with ISO 527-2, using test specimen 5A and a crosshead speed of 50 mm/min ± 5 mm/min. For determination of tensile modulus, use a crosshead speed of 1 mm/min.

8.2.3 Modulus in flexure

Determine this property in accordance with the procedures of ISO 178.

8.3 Thermal-transition temperatures

8.3.1 Deformation temperature under load

Determine this temperature in accordance with the procedures of ISO 75-2.

8.3.2 Glass-transition temperature(s)

Determine these temperatures in accordance with the procedures of ASTM D 3418 or ISO 11357-2.

8.3.3 Melting-peak temperature

8.3.3.1 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 $10 \text{ mg} \pm 2 \text{ mg}$ 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 supplemented by ASTM D 3418 are appropriate for this determination. Some fluoropolymers such as PTFE show different melting behaviour the first time a virgin powder is melted compared to the second and subsequent determinations that have lower melting-peak temperatures. Both the first and second melting points shall be measured. With PTFE, the second melting point usually is $327 \text{ }^\circ\text{C} \pm 10 \text{ }^\circ\text{C}$. The first melting point is normally at least $5 \text{ }^\circ\text{C}$ higher than the second melting point.

8.3.3.2 Use differential scanning calorimetry (DSC) as described in ASTM D 3418, ISO 11357-3 and ASTM D 4591 for this determination. The heating rate shall be $10 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$ per minute. 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.

8.3.3.3 Other thermal techniques may be used if the user can demonstrate that they are capable of measuring the melting-peak temperature and give results of equivalent significance.

8.4 Density

Cut two specimens from the moulding or other solid sample and determine the density in accordance with one of the methods described in ISO 1183-1 or ISO 1183-2. If ISO 1183-2 is used, the liquid system used shall have a density gradient appropriate for the fluoropolymer being tested (see Table A.1 in ISO 1183-2:2004). The use of ISO 1183-2 is discouraged, however, due to the carcinogenicity of the liquids used.

8.5 Flammability by oxygen index

Use the procedure in the appropriate part of ISO 4589.

8.6 Particle size and size distribution

8.6.1 General

The wet and dry-sieve procedures of 8.6.2 and 8.6.3 are widely used with PTFE and closely related materials. The resistance-variation test procedure in 8.6.4 (the Coulter principle) is often used with PVDF, PTFE filler resin, and fine-cut suspension powders. The light-scattering procedures in 8.6.5 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 to the detailed procedures given in this part of ISO 12086. ASTM F 660 (see the Bibliography) provides a standard practice for comparing particle size determined with different types of automatic particle counter.

8.6.2 Wet-sieve analysis

8.6.2.1 Significance and use

The fabrication of PTFE resins either by moulding or extrusion is affected significantly by particle (or agglomerate) size and size distribution. The average particle size of PTFE resins is determined by fractionation of the material with a series of sieves. Fractionation is facilitated by spraying the powder on a sieve with an organic liquid that wets the powder, breaks up lumps and prevents clogging of the sieve openings. In published test procedures, the liquid specified is perchloroethylene (see Warning). Use of isopropyl alcohol or ethyl alcohol instead of perchloroethylene has been reported as giving equivalent results.

WARNING — Perchloroethylene is under investigation by government agencies and industry for its carcinogenic effects. Protective nitrile or butyl gloves should be worn to prevent skin contact and adequate ventilation provided to remove the vapours. The supplier's MSDS sheet should be consulted for full safety measures.

8.6.2.2 Apparatus and materials

8.6.2.2.1 Balance, capable of weighing to $\pm 0,1$ g.

8.6.2.2.2 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 μm (No. 25), 500 μm (No. 35), 355 μm (No. 45), 250 μm (No. 60) and 180 μm (No. 80). The equivalent sieve numbers, given for information, are those defined in ASTM E 11 (see the Bibliography). 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.

8.6.2.2.3 Ventilated hood.

8.6.2.2.4 Six tared beakers, capacity 150 ml.

NOTE As an alternative, the sieves may be tared, dried and weighed on a balance to avoid errors that can be introduced during transfer of fractionated samples to the tared beakers.

8.6.2.2.5 Sieving and solvent-spraying apparatus: A suggested arrangement for an apparatus with recirculating spray liquid is shown in Figure 2. The apparatus shall be located, and the operations carried out, in a ventilated hood or adequately ventilated area.

8.6.2.2.6 Spray liquid, 20 litres. See the comments and Warning in 8.6.2.1. Although perchloroethylene has been the usual choice, an alternative liquid may be used after its applicability and any hazards associated with its use have been investigated thoroughly and use of the liquid shown to be satisfactory.

8.6.2.3 Procedure

8.6.2.3.1 Weigh out a 10 g test sample for powders with a particle size less than 100 μm or a 50 g test sample for powders with a larger particle size. Adjust the rate of flow of the spray liquid to 6 l/min \pm 0,5 l/min.

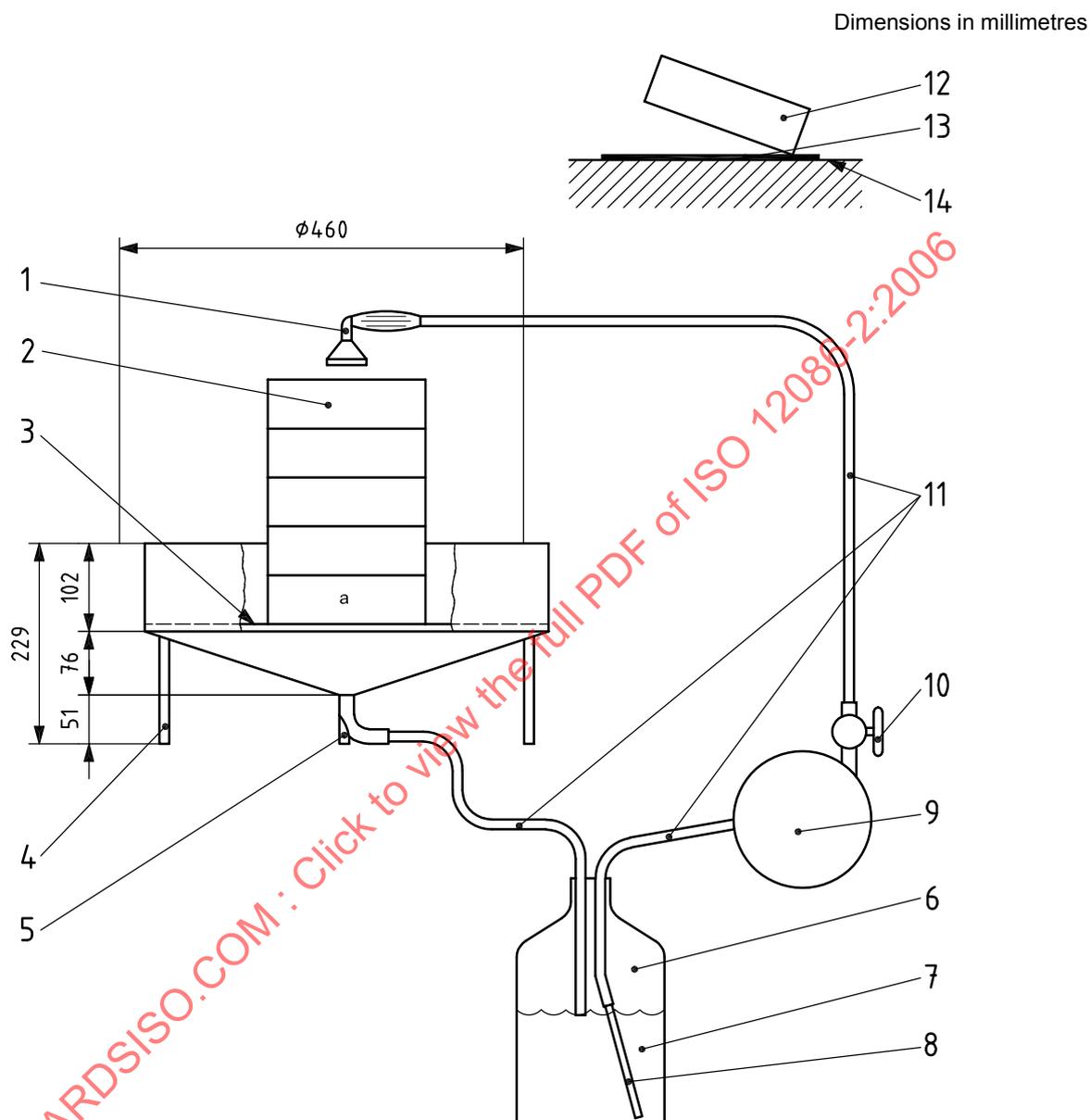
8.6.2.3.2 Place the weighed resin on the top sieve and spray it with the organic spray liquid for 1 min \pm 0,2 min. The shower head shall be about level with the top of the sieve and be moved in a circular fashion. Take care to break up all of the lumps and to wash the material from the sides of the sieve.

8.6.2.3.3 Remove the top sieve and place it in the hood to dry until all of the sieves are ready for oven drying as described in 8.6.2.3.4.

8.6.2.3.4 Repeat the procedure specified in 8.6.2.3.2 and 8.6.2.3.3 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 Figure 2. Pour each fraction into a tared beaker and weigh to $\pm 0,1$ g.

8.6.2.3.5 Record the mass of resin on each sieve.

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



Key

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

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

Figure 2 — Apparatus for wet-sieve analysis

8.6.2.4 Expression of results

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

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

where

$F = 2$ for a 50 g test sample;

$F = 10$ for 10 g test sample;

m is the mass, in grams, of resin on sieve Y .

8.6.2.4.2 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 μm (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 μm (No. 25) plus net percentage on 500 μm (No. 35) sieve.

8.6.2.4.3 Plot the cumulative percentage versus the sieve opening size (or sieve number) on log/log paper as shown in the sample plot in Figure 3. 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 % 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.

8.6.2.5 Precision and bias

Because the resin particles have complex shapes, and because on each sieve there is a distribution of particle sizes, the values for particle size and particle-size distribution obtained will only be relative numbers. The 95 % confidence limits based on a limited series of tests are $\pm 2,8$ % for the average particle size. Since there is no accepted reference material suitable for determining the bias for this test procedure, no statement on bias can be made.

8.6.3 Dry-sieve analysis

8.6.3.1 Significance and use

The fabrication of PTFE resins may be affected significantly by particle (or agglomerate) size and size distribution. The average particle size of PTFE resins is determined by fractionation of the material with a series of sieves. Fractionation is accomplished by mechanically shaking the material in an assembly of sieves for a specified period.

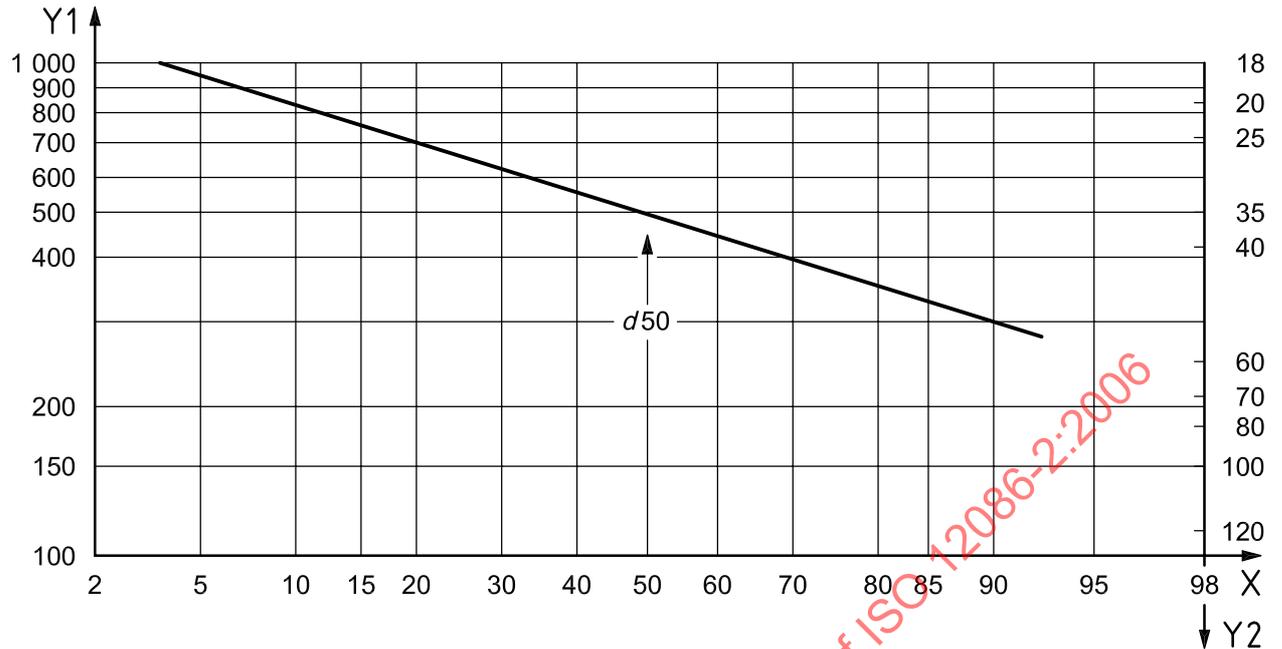
8.6.3.2 Apparatus

8.6.3.2.1 **Balance**, capable of weighing to $\pm 0,1$ g.

8.6.3.2.2 **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 μm (No. 25), 500 μm (No. 35), 355 μm (No. 45), 250 μm (No. 60) and 180 μm (No. 80). The equivalent sieve numbers, given for information, are those defined in ASTM E 11 (see the Bibliography). 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.

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

8.6.3.2.4 **Freezer**: Any commercial ice freezer (a dry-ice chest may be used).

**Key**

- X cumulative percentage, %
 Y1 sieve opening size, μm
 Y2 sieve No.

Sieve No.	Sieve opening size μm
14	1 400
18	1 000
25	710
35	500
45	355
60	250
80	180

Figure 3 — Typical log/log probability plot for sieve analysis

8.6.3.3 Procedure

8.6.3.3.1 Place $50 \text{ g} \pm 0,1 \text{ g}$ of the sample in an aluminium pan, and cool the pan and contents to less than 10°C .

8.6.3.3.2 Measure the tare mass of each of the sieves listed in 8.6.3.2.2. Place the conditioned test sample on the top sieve of the assembly and shake in the sieve shaker for $10 \text{ min} \pm 0,5 \text{ min}$. 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.

8.6.3.4 Expression of results

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

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

where m is the mass, in grams, of resin on sieve Y .

8.6.3.4.2 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 μm (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 μm (No. 25) plus net percentage on 500 μm (No. 35) sieve.

8.6.3.4.3 Plot the cumulative percentage versus the sieve opening size (or sieve number) on log/log paper as shown in the sample plot in Figure 3. 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 % 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.

8.6.3.5 Precision and bias

The test precision is $\pm 3,2\%$ (two sigma limits) for the combined sieve fractions for the 710 μm + 500 μm + 355 μm (No. 25 + 35 + 45) sieves for a resin where this combination of sieves retains, on the average, 78 % of the sample. Since there is no accepted reference material suitable for determining the bias for this test procedure, no statement on bias can be made.

8.6.4 Particle size and size distribution by the Coulter principle using a resistance-variation tester

8.6.4.1 Significance and use

Fluoropolymer powders have various uses that depend on their particle size and distribution. For some of these, such as the powder forms of PVDF, the sieve analysis procedures are not the method of choice.

8.6.4.2 Apparatus

8.6.4.2.1 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-divinyl benzene) latices are particularly recommended.

8.6.4.2.2 Analytical balance.

8.6.4.2.3 Magnetic stirrer.

8.6.4.2.4 Ultrasonic tank

8.6.4.3 Procedure

8.6.4.3.1 Prepare a solution of a non-ionic surfactant²⁾ (at a concentration of 0,2 g/l to 0,3 g/l in an aqueous electrolyte, such as a 1 % (mass per unit volume) solution of sodium chloride or a special 1 % (mass per unit volume) saline solution³⁾). 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 μm barrier filter^{4), 5)}.

2) Triton X-100, a surfactant of the octylphenol series, from Rohm & Haas, has been found to be satisfactory. Other, similar, materials should be equally effective.

3) A specially prepared solution, known as Isoton, is available from Coulter Counter Ltd.

4) Available from the Millipore Corporation.

5) This information is given for the convenience of users of this part of ISO 12086 and does not constitute an endorsement by ISO of these materials.

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

8.6.4.3.3 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 two to three minutes followed by fifteen minutes at 700 r/min to 750 r/min.

8.6.4.3.4 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 four minutes should be sufficient to obtain a uniform suspension of the fluoropolymer powder.

8.6.4.3.5 Resume stirring with the magnetic stirrer just rapidly enough to maintain a uniform suspension while removing 10 μ l to 200 μ 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 % (mass per unit volume) saline solution described in 8.6.4.3.1. Be careful to avoid the formation of air bubbles when filling.

8.6.4.3.6 Count particles in time mode using three measurements.

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

8.6.4.4 Expression of results

Plot a volume or mass percent curve on suitable graph paper as described for sieve analysis in 8.6.2.4.3.

8.6.5 Particle size and size distribution by light scattering of a laser beam

8.6.5.1 Significance and use

Light-scattering procedures for determining particle size and particle-size distribution for particulate materials have advanced a great deal in recent years and are increasingly being used with fluoropolymer powders. One procedure is in ISO 13320-1.

8.6.5.2 Apparatus

The particle-size analyser shall be based on Fraunhofer diffraction or 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.

8.6.5.3 Procedure

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

9 Testing of fluoropolymer dispersions

9.1 General

This part of ISO 12086 contains test methods used to define characteristics of the polymer and of the dispersion of the polymer. These methods supplement standards that have general application to polymer dispersions. The most commonly used fluoropolymer dispersion is that based on PTFE and the procedures in this part of ISO 12086 are limited to characteristics for PTFE dispersion and for the PTFE in the dispersion.

This clause identifies tests, the details of which are in Clause 10, that will (a) define the solids in the dispersion to be PTFE and (b) characterize the PTFE. In order to run these tests, dry, solid PTFE must be isolated from the dispersion by the procedure given below.

9.2 Preparation of test samples

9.2.1 PTFE solids in the 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 five minutes at 3 r/min to 4 r/min, by stirring with a smooth rod for three to four minutes, or by other types of gentle agitation.

WARNING — Excessive agitation can coagulate the dispersion.

9.2.2 After blending, take the sample by removing an aliquot. A suitable method is to insert a clean, smooth, dry glass tube, open at each end, until it reaches the bottom of the container. An internal diameter of 6 mm or 7 mm is suitable. The ends of the tube shall be smooth to prevent injury. Close the upper end of the tube, remove the tube from the container, and transfer the contents to a clean, dry glass jar. Repeat until the desired sample size is reached.

9.2.3 When samples are drawn from several containers, the individual samples may be combined and thoroughly mixed by gentle stirring when the samples are combined and again before the combined sample is tested.

9.3 Isolation of PTFE from dispersion

9.3.1 Apparatus

Required are a 475 ml wide-mouth bottle with sealable top, a 12,5 cm Buchner funnel, a 100 ml graduated cylinder, a watchglass or aluminium pan, a vacuum oven capable of operating at 150 °C and at an absolute pressure of 10 mmHg, a desiccator and a balance.

9.3.2 Materials

Required are methanol, acetone, filter fabric and deionized water.

9.3.3 Procedure for isolating PTFE as a powder

9.3.3.1 Filter the PTFE dispersion through a double layer of cheese cloth. A convenient amount is enough dispersion to isolate 35 g of solids. The test requires 12,6 g of solids. Add the appropriate amount of filtered dispersion to the 475 ml wide-mouth bottle.

9.3.3.2 Add to the filtered sample in the order indicated: 50 ml of acetone, 75 ml of deionized water and 75 ml of methanol.

9.3.3.3 Seal the bottle and shake until the sample is coagulated.

9.3.3.4 Place eight layers of filter fabric over the open end of the bottle and position the inverted bottle and filter fabric in the Buchner funnel.

9.3.3.5 Remove the bottle from the Buchner funnel before filtering the liquid portion by opening the vacuum valve.

9.3.3.6 Release the vacuum and return the resin to the 475 ml bottle. Add 200 ml of methanol. Shake for 120 s \pm 15 s. Remove the methanol by vacuum filtering, as in 9.3.3.4 and 9.3.3.5.

9.3.3.7 Repeat 9.3.3.6.

9.3.3.8 Repeat 9.3.3.6 twice more, using 200 ml of deionized or distilled water at 85 °C \pm 5 °C. Then repeat 9.3.3.6 once, using 150 ml of acetone.

9.3.3.9 Place the washed sample in the aluminium pan or watchglass and cover to prevent contamination.

9.3.3.10 Dry the PTFE powder to 0,04 %, or less, moisture. After drying, cool the powder to room temperature in the desiccator before weighing. Repeated drying, cooling and weighing may be necessary.

NOTE Use of a vacuum oven at an absolute pressure of 10 mmHg and a temperature of 150 °C is recommended to achieve dryness.

9.4 Coagulum in dispersions

9.4.1 General

Polymer that has coagulated in the dispersion may not be useful to the purchaser. This test will determine the percentage of coagulated polymer.

9.4.2 Apparatus

Required are a tared beaker to hold 1 000 g ± 1 g of dispersion, an 80-mesh filter screen, a funnel, an oven capable of operating at 120 °C ± 5 °C, a desiccator and a balance capable of weighing to ± 1 mg.

9.4.3 Materials

Required is distilled water.

9.4.4 Procedure

Weigh 1 000 g ± 1 g of dispersion (m_1) into the tared beaker. Weigh the screen to 1 mg and record the mass (m_2). Secure the screen to the funnel and filter the dispersion through the screen. Rinse the beaker with 25 ml of distilled water and use this rinse water to wash the coagulum on the screen. Gently wash the coagulated polymer on the screen with 25 ml of distilled water from a wash bottle. Carefully remove as much as possible of the coagulated polymer from the screen with 25 ml of distilled water from a wash bottle. Carefully remove the screen from the funnel and dry the screen and coagulum at 120 °C ± 5 °C for 2 h. Weigh the screen and coagulum to 1 mg after allowing to cool to room temperature in the desiccator. Record the mass as m_3 .

9.4.5 Expression of results

Calculate the coagulum content as follows:

$$\% \text{ coagulum content} = \frac{m_3 - m_2}{m_1 \times w(\text{PTFE})} \times 100$$

where

m_1 is the mass, in grams, of the test portion of dispersion;

m_2 is the mass, in grams, of the screen;

m_3 is the mass, in grams, of the screen and coagulated polymer;

$w(\text{PTFE})$ is the mass fraction of PTFE in the dispersion as determined in 9.5, expressed as a decimal fraction.

9.5 Percentage polymer and surfactant in aqueous dispersion

9.5.1 Polymer solids and surfactant content by mass loss

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

9.5.2 Apparatus

Required are an aluminium weighing dish, an oven capable of reaching $120\text{ °C} \pm 5\text{ °C}$, an oven capable of reaching $380\text{ °C} \pm 10\text{ °C}$, a desiccator and a balance capable of weighing to 0,1 mg.

9.5.3 Procedure

Weigh the aluminium weighing dish to 0,1 mg (m_1). Add 10 g of PTFE dispersion and reweigh immediately to 0,1 mg (m_2). Dry the test portion for 2 h at $120\text{ °C} \pm 5\text{ °C}$. 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 $380\text{ °C} \pm 10\text{ °C}$ for $35\text{ min} \pm 1\text{ min}$. Allow the sample to cool in the desiccator to room temperature and weigh to 0,1 mg (m_4).

9.5.4 Expression of results

For surfactants that are completely volatile, use the following equations:

$$\% \text{ PTFE} = \frac{m_4 - m_1}{m_2 - m_1} \times 100$$

$$\% \text{ surfactant} = \frac{m_3 - m_4}{m_4 - m_1} \times 100$$

For surfactants that are not completely volatile, use the following equations:

$$\% \text{ PTFE} = \frac{(m_3 - m_1) - (m_3 - m_4)(1 + k)}{m_2 - m_1} \times 100$$

$$\% \text{ surfactant} = \frac{(m_3 - m_4)(1 + k)}{(m_3 - m_1) - (m_3 - m_4)(1 + k)} \times 100$$

where k is the mass of the non-volatile portion of the surfactant divided by the mass of the volatile portion of the surfactant.

Upon request, the supplier shall inform the user whether the surfactant can be completely removed by the procedures of this part of ISO 12086 and, if not, the manufacturer shall define the surfactant or the volatile and non-volatile portions of the surfactant.

9.5.5 Precision and bias

No data are currently available.

9.6 PTFE solids content by hydrometer

9.6.1 Significance and use

An approximate solids content of a PTFE dispersion is commonly determined from the specific gravity of the dispersion. The hydrometer reading is a function of the solids content, the surfactant content and other parameters of the dispersion. Therefore any single conversion table has inherent error and cannot be universally applicable. If this method is to be used, a table relating specific gravity to solids content of the required precision shall be constructed for the system being used or, if available, obtained from the supplier.

NOTE Some of the additives permitted in fluoropolymer dispersions may increase the viscosity of the dispersion so much that it is unlikely that the hydrometer procedure can be used to determine the density in a reliable manner.

9.6.2 Apparatus

Required are a hydrometer or set of hydrometers capable of measuring specific gravity from 1,000 to 1,550 with a precision of $\pm 0,001$ and a graduated cylinder large enough to hold the hydrometer with reasonable clearance from the sidewalls of the hydrometer. Automatic apparatus to make this determination is an

acceptable alternative after equivalence of results has been determined. An example of such an apparatus is referred to in 10.4.3.3.

9.6.3 Procedure

Fill the graduated cylinder with enough PTFE dispersion to float the hydrometer. Place the hydrometer in the cylinder. Add dispersion until the cylinder is full and the meniscus is slightly convex. Read the hydrometer at the top of the dispersion. The reading shall be accurate to 0,001. Translate the hydrometer reading to solids content using the table.

9.6.4 Precision and bias

Duplicate results by the same operator should not be considered suspect unless they differ by more than 1 % relative.

9.7 pH of dispersions

The test method shall be in accordance with ISO 976. Although pH is not a designatory property in ISO 12086-1, the test method is included because some buyers or sellers attach importance to the pH of the dispersion.

NOTE PTFE dispersion can coat the electrode, so thorough cleaning is necessary. Cleaning with toluene on a soft cloth or a concentrated surfactant on a soft cloth is suggested.

WARNING — Toluene is hazardous. Consult appropriate safety information before using.

10 Testing of PTFE and closely related materials

10.1 General

The usual methods of processing thermoplastics are generally not applicable to these materials because of their viscoelastic properties at processing temperatures. The procedures needed to prepare test specimens of this group of fluoropolymers are close, in principle, to the procedures used for powdered metals, ceramics or lead in press operations. As a result, many of the usual procedures found in International Standards for thermoplastic materials are not appropriate. This part of ISO 12086 includes details, developed by the industry, that are required to prepare suitable specimens and to test for the properties critical in processing. Tests related to the nature of the particles are very important. They include particle size, bulk density and powder flow. The tests for SSG and ESG have been developed as a means of estimating relative molecular mass and thermal stability. In addition, extrusion pressure and stretching-void index (SVI) are important tests for appraising the suitability of the coagulated dispersion forms of PTFE for use in many applications.

10.2 Preparation of test specimens by moulding

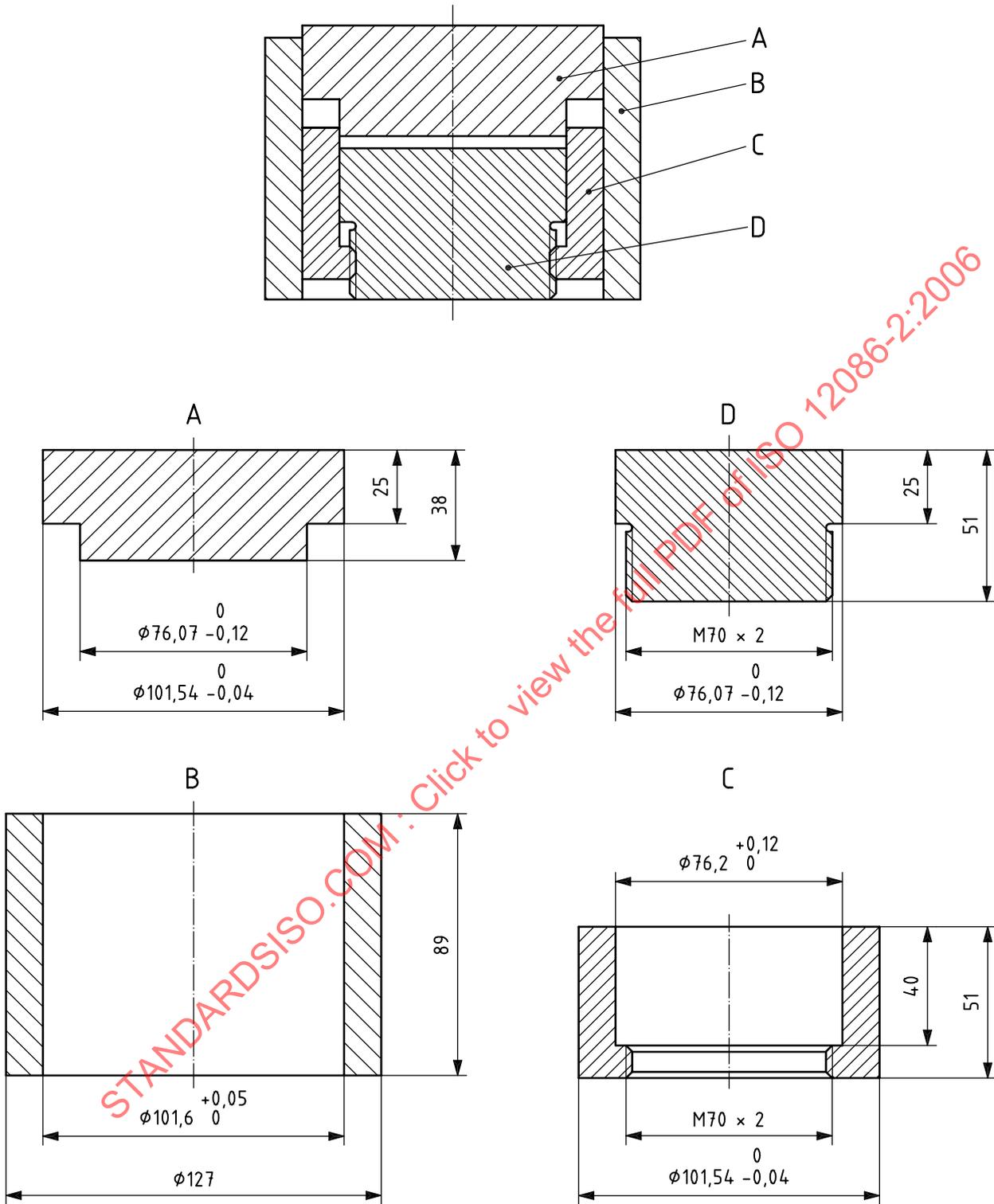
10.2.1 Test discs for tensile properties of PTFE and closely related materials

10.2.1.1 Apparatus

10.2.1.1.1 Mould assembly, as illustrated in Figure 4.

The mould shall be assembled as shown in Figure 4, leaving aside the upper pressing piece.

Dimensions in millimetres



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

Figure 4 — Mould assembly for preparation of specimens for the determination of tensile properties

10.2.1.1.2 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 preformed. 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.

10.2.1.2 Procedure

Condition the material to be preformed for at least 6 h in accordance with 7.1.

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.

Sieve a sample of approximately 20 g of the powder to be tested through a 2,0 mm aperture (No. 10) sieve. Weigh $14,5 \text{ g} \pm 0,1 \text{ g}$ 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.

NOTE 1 The adjustable cavity depth permits easier levelling of powders of different bulk densities.

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

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

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

NOTE 2 Small-particle-size, low-powder-flow forms of PTFE-S can usually be preformed 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.

Hold the assembly for 3 min while maintaining the pressure at the specified value.

Release the pressure slowly over a period of $10 \text{ s} \pm 2 \text{ s}$ 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 10.6.1.4 and Table 6.

10.2.2 Preparation of test billets for determining tensile properties of PTFE on skived specimens or specimens cut in other ways

Film or sheet skived or cut from small billets may be used as alternatives to the test discs described in 10.2.1. The apparatus for preforming a test billet is shown in Figure 5. The preforming and sectioning of the test billet shall be done as described in ASTM D 4894.

Preform pressures shall be:

15 MPa for small-particle-size, low-powder-flow granular and coagulated dispersion products;

35 MPa for PTFE-S (granular PTFE);

35 MPa to 70 MPa for filled PTFE.

The nominal thickness of unfilled skived film shall be at least 0,125 mm and may be as much as 0,5 mm.

The thickness of skived film from filled compositions shall be at least 0,5 mm and may be as much as 1,2 mm. If the billet is sliced to prepare the test specimen, the thickness shall be 0,8 mm.

10.3 Bulk density

10.3.1 Significance and use

Bulk density gives an indication of how a resin may 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 10.3.3 shall be used to measure this property.

This procedure may also be found in ASTM D 4894 and ASTM D 4895.

10.3.2 Apparatus

10.3.2.1 Funnel, as shown in Figure 6.

10.3.2.2 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 ⁶⁾.

10.3.2.3 Controller ⁷⁾.

10.3.2.4 Volumetric cup and cup stand, as shown in Figure 7. 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 $250 \text{ g} \pm 0,5 \text{ g}$.

10.3.2.5 Levelling device, as shown in Figure 8, affixed permanently to 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.

10.3.2.6 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.

10.3.2.7 Balance, having an extended beam, and with a capacity of 500 g and a sensitivity of 0,1 g or equivalent.

10.3.3 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 $38,1 \text{ mm} \pm 3,2 \text{ mm}$. 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.

10.3.4 Expression of results

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

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

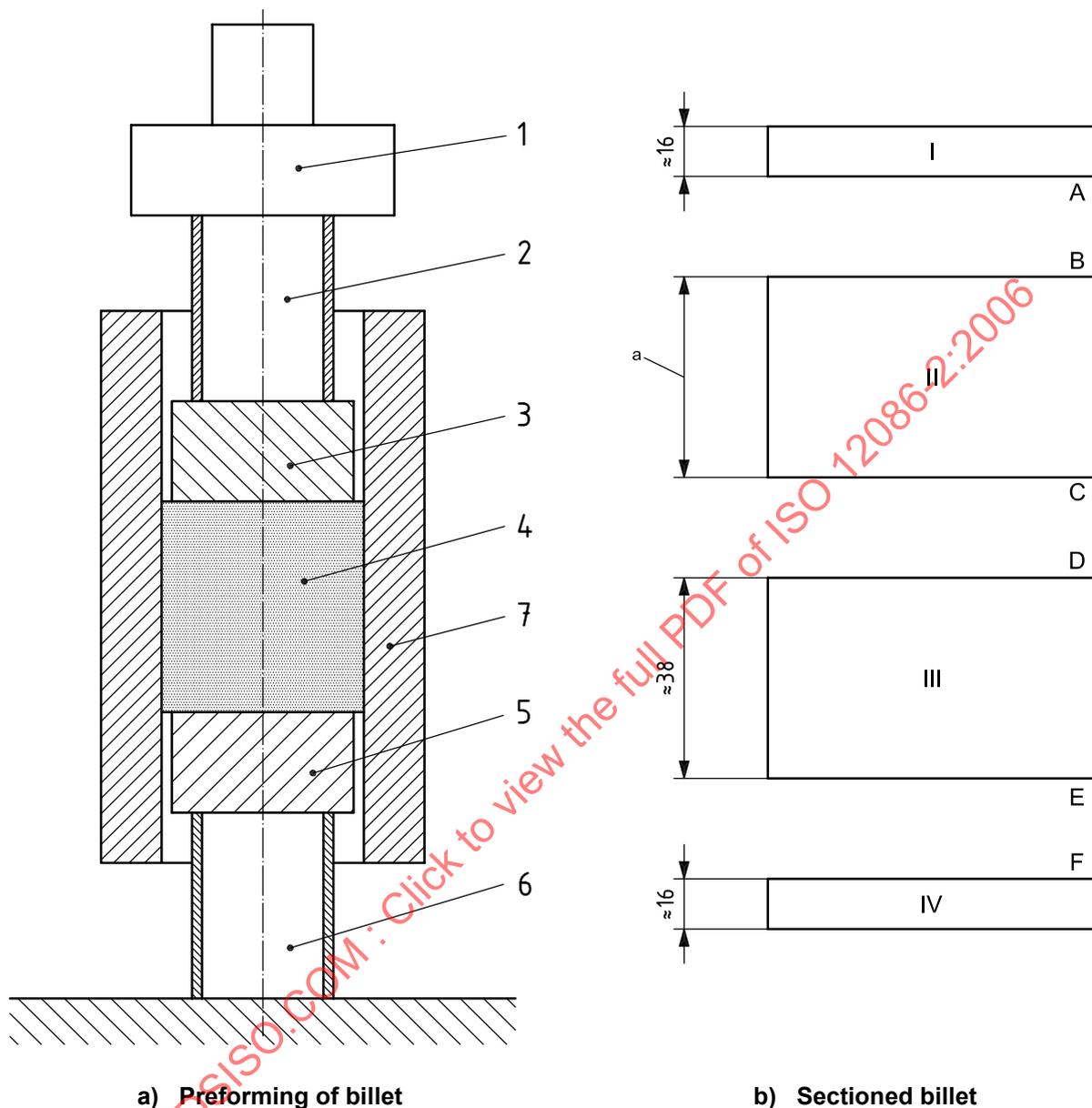
10.3.5 Precision and bias

No data are currently available.

6) A laboratory-sized vibrating feeder has been found satisfactory for this purpose. Originally used was a “Vibra-Flow” feeder, Type F-T01A, with trough, available from FMC, Material Handling Division, FMC Building, Homer City, PA 15748, USA, Tel.: +1 412 479-8011, Fax: +1 412 479-3400, which may still be available. This information is given for the convenience of users of this part of ISO 12086 and does not constitute an endorsement by ISO of this product. Other feeders may also be suitable.

7) A suitable controller for the feeder should be used. Originally used was a “Syntron” controller, Type CSCRB1, available from FMC, address as given in Footnote 6, which may still be available. This information is given for the convenience of users of this part of ISO 12086 and does not constitute an endorsement by ISO of this product. Other sources may be available.

Dimensions in millimetres



Key

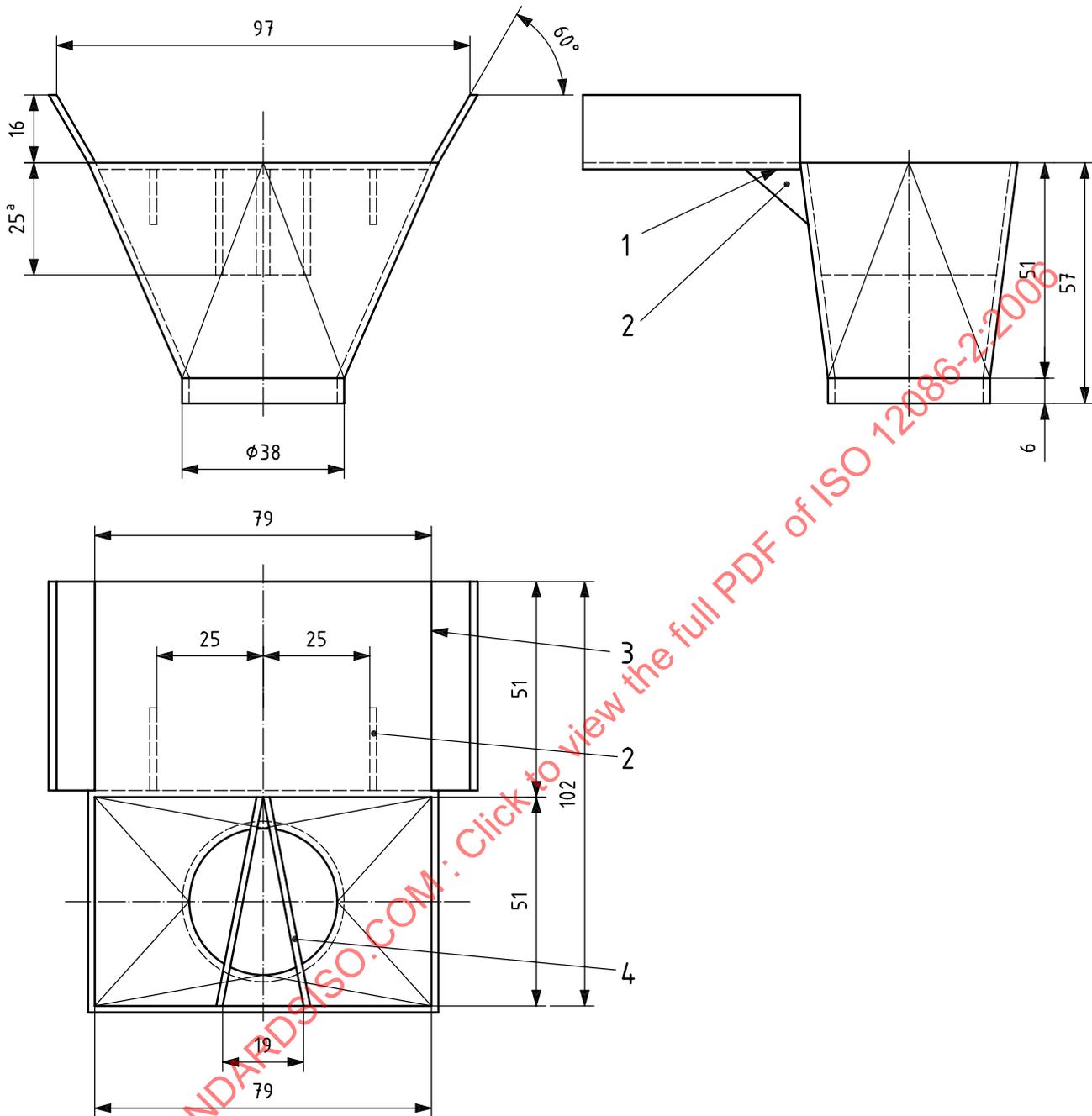
- 1 ram
- 2 steel pusher
- 3 brass plug
- 4 PTFE resin
- 5 plug
- 6 pusher
- 7 steel mould, int. diam. \approx 57 mm, length 305 mm to 380 mm

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

^a Variable between 24 mm and 40 mm, depending on the total height of the billet after sintering.

Figure 5 — Apparatus for preforming PTFE test billet

Dimensions in millimetres



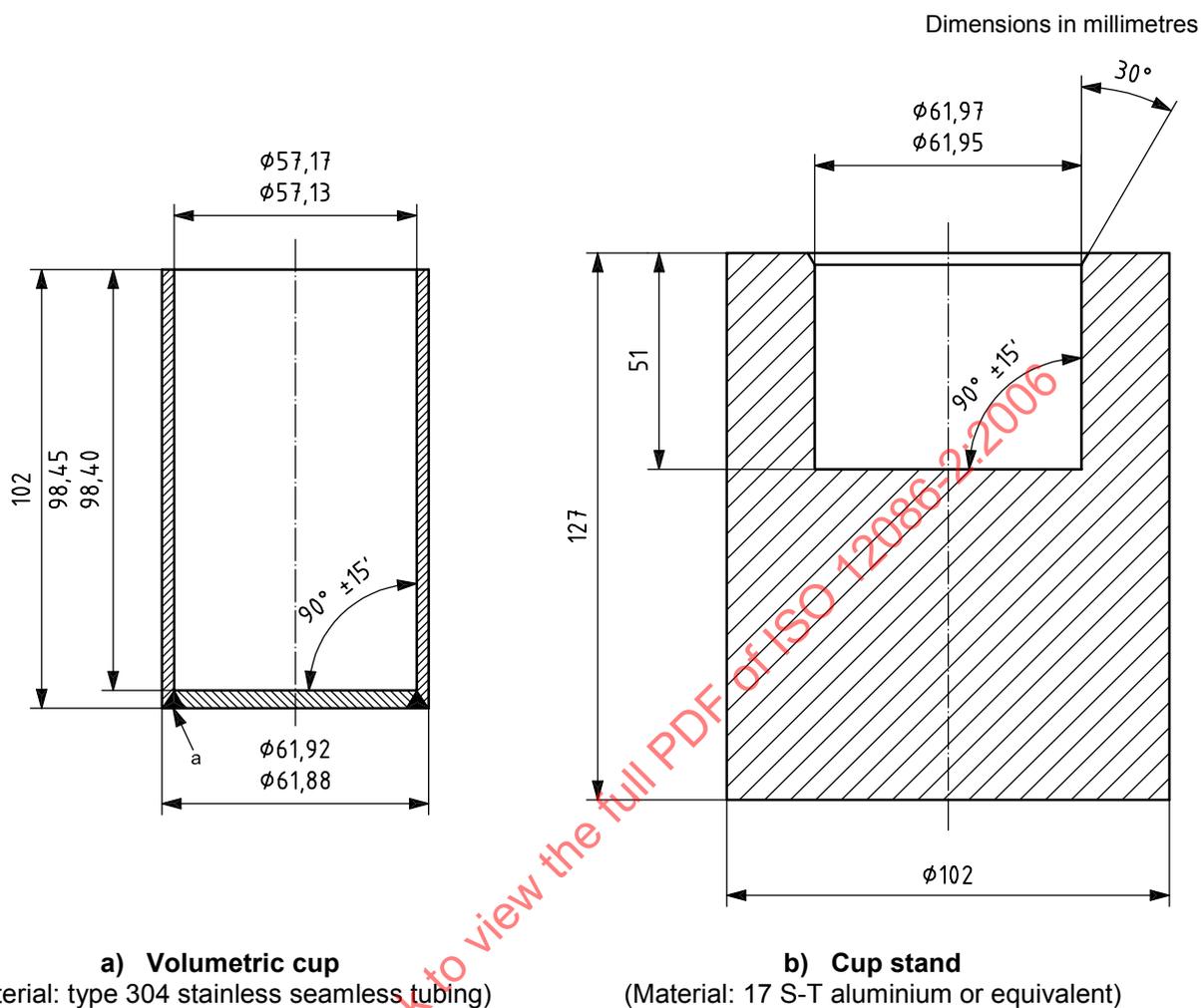
Key

- 1 weld
- 2 two support gussets, approx. 13 mm × 13 mm × 1,6 mm thick, located in positions shown
- 3 bend
- 4 straightening vanes (locate two partitions as shown)

Funnel material: type 304 stainless steel, 16 gauge (1,6 mm thickness).

^a Depth of partitions.

Figure 6 — Details of funnel used for determination of bulk density



^a Weld all round and grind smooth.

Figure 7 — Volumetric cup and cup stand for determination of bulk density

10.4 Extrusion pressure

10.4.1 Significance and use

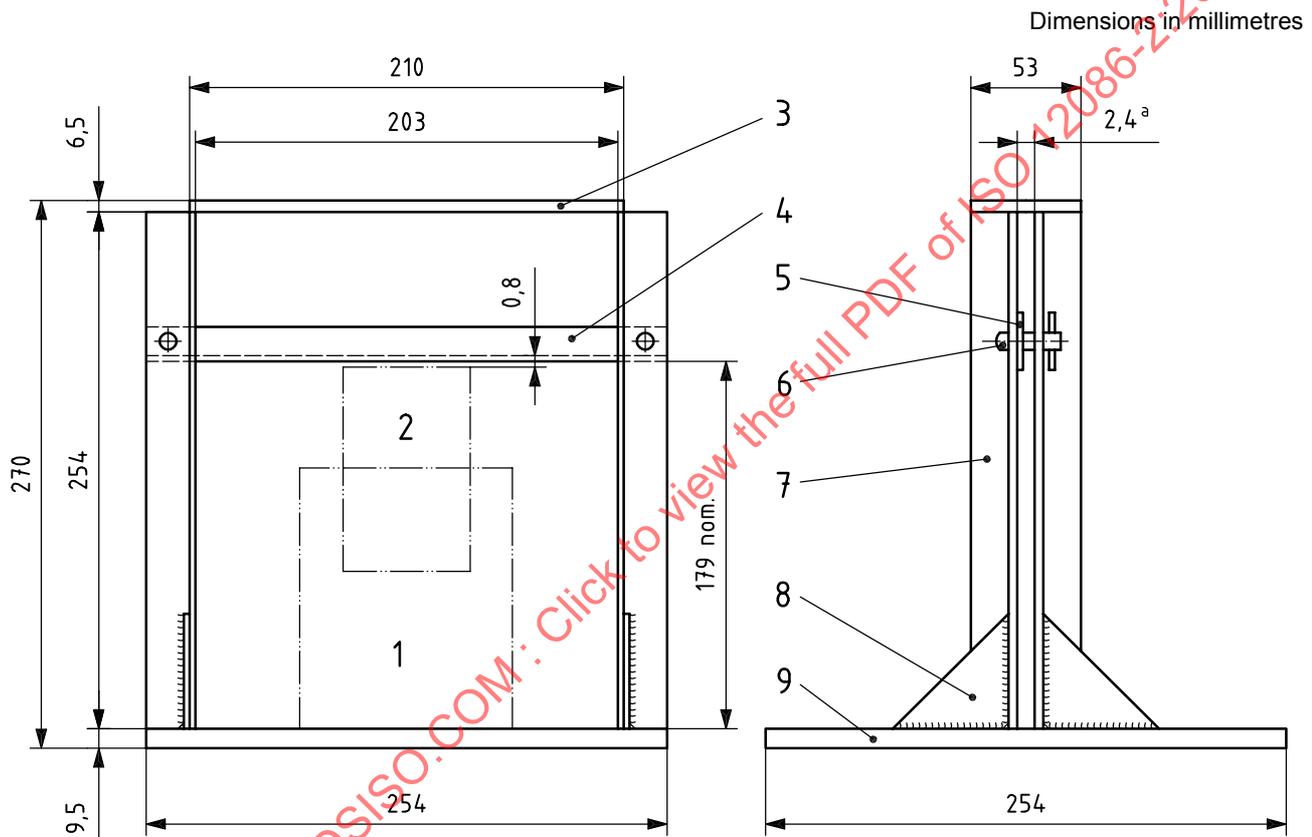
Processing of coagulated-dispersion PTFE resins normally involves “paste extrusion” or “lubricated extrusion” of a blend of the resin with a volatile liquid. The pressure that must be applied to such a blend to extrude it is affected by several processing conditions which include the nature and amount 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.

10.4.2 Apparatus (equivalent apparatus may be substituted)

10.4.2.1 Paste extruder (see Figure 9): 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 $30\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$. 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.

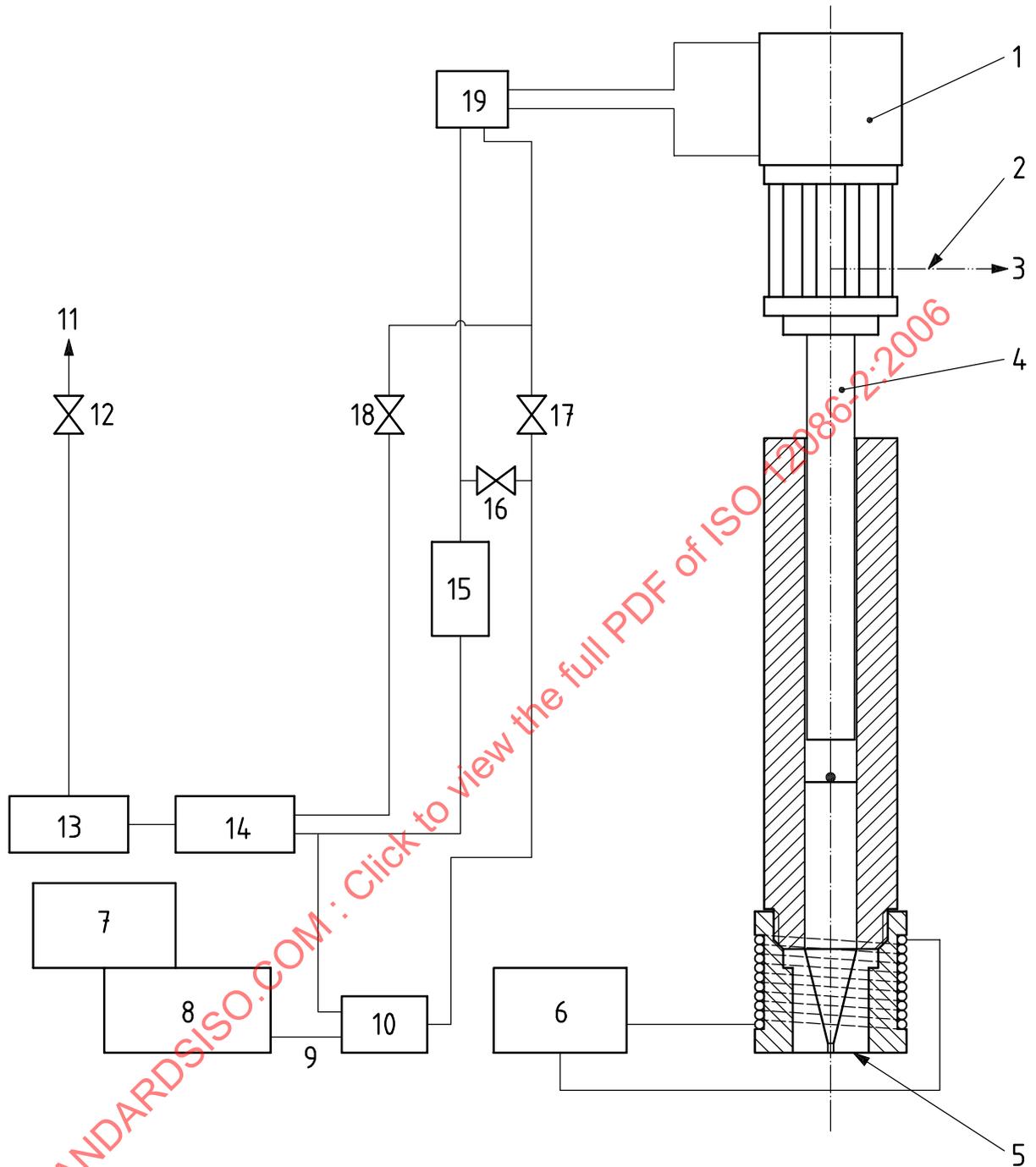


Key

- 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 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

^a Gap left between angles for mounting saw blade.

Figure 8 — Leveller stand for determination of bulk density



Key

- | | |
|-----------------------------|------------------------------|
| 1 hydraulic cylinder | 11 to air supply |
| 2 pressure probe | 12 air-motor valve |
| 3 to recorder | 13 air motor |
| 4 ram | 14 air pump |
| 5 die | 15 reservoir |
| 6 constant-temperature bath | 16 safety valve |
| 7 drive motor | 17 slow speed |
| 8 link belt drive | 18 rapid speed |
| 9 gear reducer | 19 piston with two-way valve |
| 10 pump | |

Figure 9 — Paste extruder for determining extrusion pressure

10.4.2.2 Interchangeable extrusion dies (see Figure 10), each having a 30° included angle and dimensions as indicated in the table in Figure 10.

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.

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

10.4.3 Procedure

10.4.3.1 Screen the dry resin through a 4,75 mm (No. 4) sieve onto a clean, dry, lint-free sheet of paper.

10.4.3.2 Transfer 200 g ± 0,5 g of the screened resin to a clean, dry glass jar about 92 mm in diameter (approximately 1 litre capacity) having an airtight closure, or into a V-blender of laboratory size.

10.4.3.3 Determine the density of the lubricant, a kerosene-type hydrocarbon liquid⁸⁾. 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 figures⁹⁾, or a technically equivalent procedure. Calculate the mass of lubricant required by multiplying the density by 60,00. Add the calculated mass ± 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 ± 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.

10.4.3.4 Blend the mixture by placing the jar on rubber-coated mill rolls and rolling it at 30 r/min for 25 min ± 5 min, by fastening the jar to a “windmill” type blender¹⁰⁾ and blending for 20 min ± 1 min, or by blending the mixture in the V-blender for 15 min ± 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.

10.4.3.5 After blending, store the jar with its contents at 30 °C ± 1 °C for a minimum of 2 h. 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.

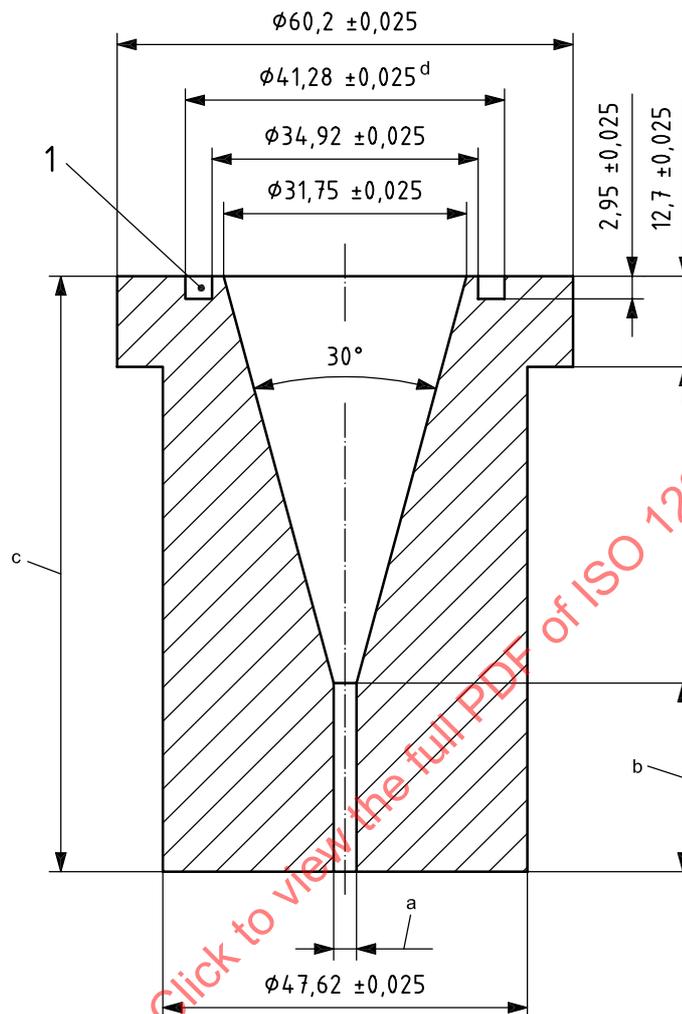
10.4.3.6 Place the proper extrusion die for the desired reduction ratio (given in the table in Figure 10) in the paste extruder.

10.4.3.7 To preform the resin for a breech-loading paste extruder (see 10.4.2.1), 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 preform the resin for a muzzle-loading paste extruder (see 10.4.2.1), 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 minimum 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.

8) 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 of this part of ISO 12086 and does not constitute an endorsement by ISO of this product.

9) 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 of this part of ISO 12086 and does not constitute an endorsement by ISO of this equipment.

10) 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 of this part of ISO 12086 and does not constitute an endorsement by ISO of this equipment.

**Key**

- 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

Figure 10 — Cross-sectional view of cylindrical die for extrusion-pressure apparatus

10.4.3.8 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.

10.4.3.9 Immediately change to slow-speed drive, start the pressure-recording system and extrude the lubricated resin at a rate of $19,0 \text{ g/min} \pm 1,0 \text{ g/min}$ (dry-resin basis).

10.4.3.10 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.

10.4.4 Precision and bias

No precision data are currently available.

10.5 Powder-flow time

10.5.1 Significance and use

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 ISO 12086-1.

10.5.2 Apparatus

10.5.2.1 Feeder, complying with 10.3.2.2.

10.5.2.2 Funnel, complying with Figure 11, constructed of aluminium and electroplated with a regular chromium coating as described in A.2.1 of BS 4641:1986, to a thickness of 12 µm.

10.5.2.3 Controller, complying with 10.3.2.3.

10.5.2.4 Vibrator, capable of vibrating the funnel at 50 cycles per second with an amplitude of 0,75 mm ± 0,25 mm.

10.5.2.5 Volumetric cup and cup stand, as detailed in Figure 12. 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 ml ± 1,0 ml. All external sharp corners of the cup stand shall be removed.

10.5.2.6 Work surface, which shall be free from vibration. The feeder shall be mounted on an adjoining table or wall bracket.

10.5.2.7 Timing device, capable of recording to the nearest 0,1 s the time taken for the material to flow through the orifice.

10.5.2.8 Levelling device, complying with 10.3.2.5.

10.5.3 Conditioning

The material shall be conditioned for not less than 6 h at 23 °C ± 2 °C and (50 ± 5) % R.H.

10.5.4 Procedure

Select a test sample of material of about 200 ml. Place the sample on the feeder sieve (10.5.2.1). 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 (10.5.2.5) and place the assembly on the work surface (10.5.2.6) such that the distance the free powder falls from the feeder outlet to the top rim of the cup is 31,8 mm ± 3,2 mm. Set the controller (10.5.2.3) 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.

Let the powder settle for 15 s and then gently push the cup and its stand beneath the leveller blade (see 10.5.2.8).

Close the funnel orifice manually and insert the 100 ml of powder from the volumetric cup. Start the vibrator (10.5.2.4). Open the orifice and at the same time start the timing device (10.5.2.7). Record the time, to the nearest 0,1 s, for the material to flow through the orifice.

10.5.5 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.

Dimensions in millimetres

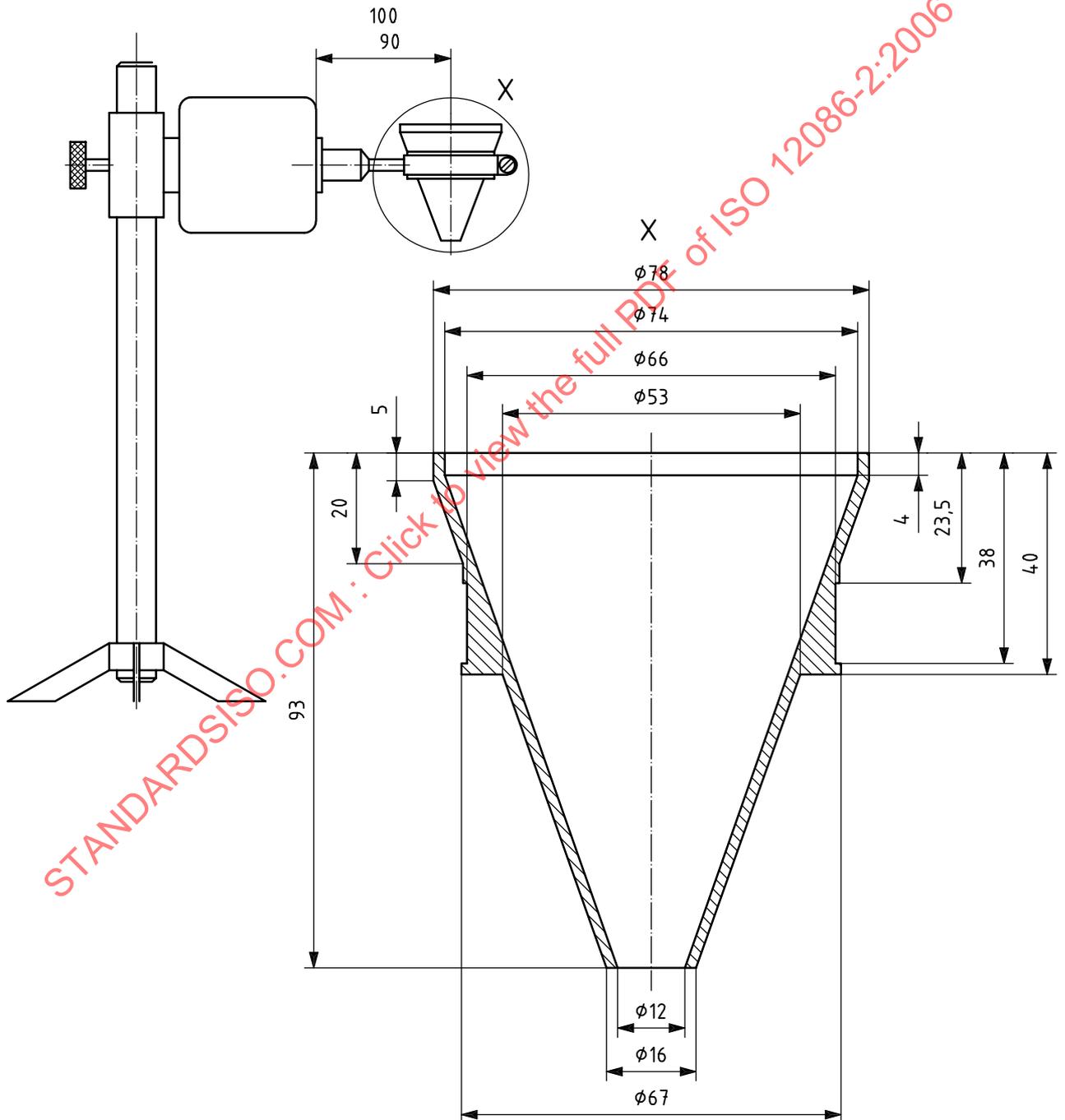


Figure 11 — Funnel for determination of flow time

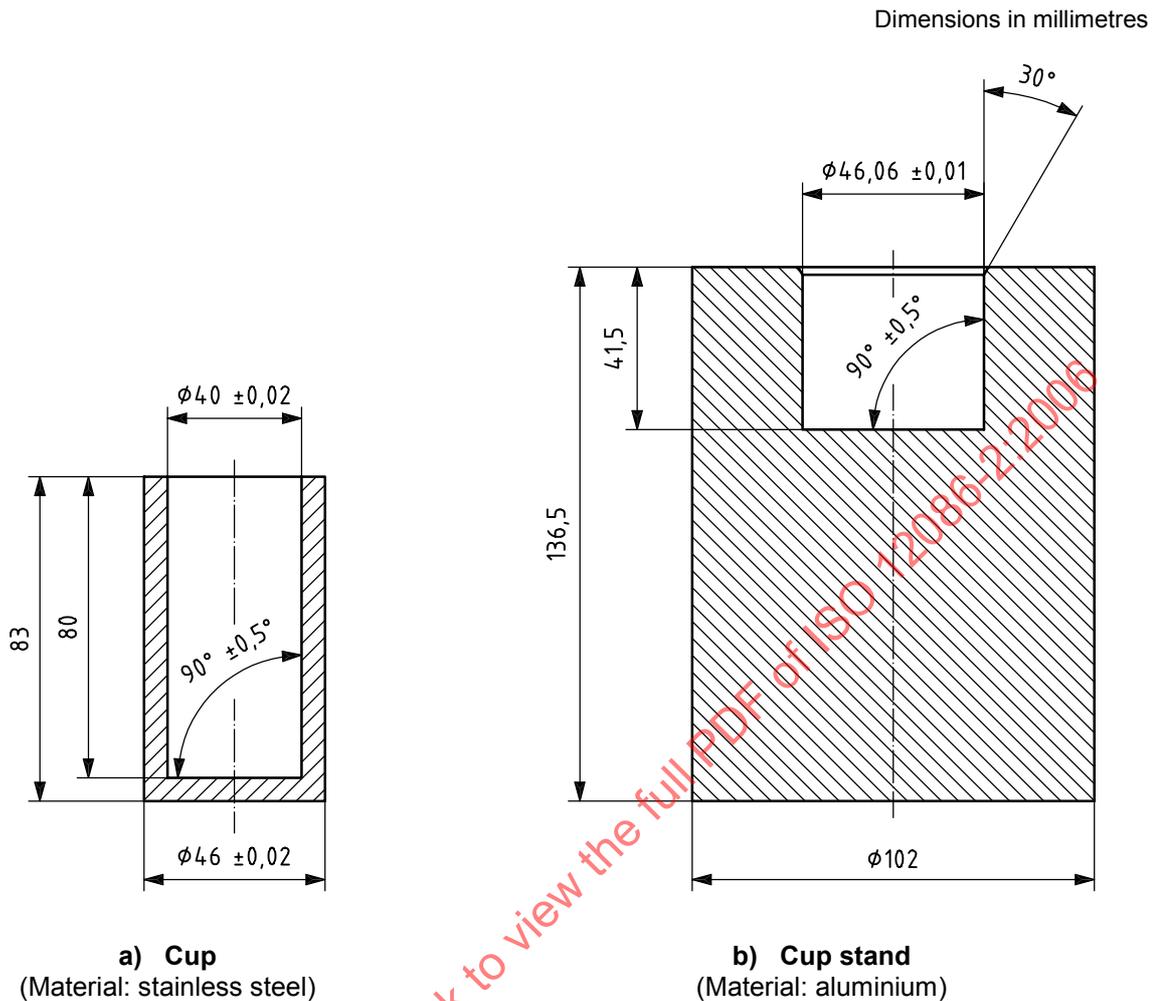


Figure 12 — Volumetric cup and cup stand for determination of flow time

10.6 Standard specific gravity (SSG), extended specific gravity (ESG) and thermal-instability index (TII)

10.6.1 Preparation of test specimens

10.6.1.1 For dispersions of PTFE, use the dried PTFE from 9.3.3.10 to prepare the test specimens. With other forms of PTFE, use the powder as received.

10.6.1.2 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 $23\text{ °C} \pm 1\text{ °C}$. 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.

10.6.1.3 Weigh out $12,0 \text{ g} \pm 0,1 \text{ g}$ of resin and place it in the preforming mould. Screen non-free-flowing resins through a 2,00 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 the $12,0 \text{ g} \pm 0,1 \text{ g}$ test sample. Insert the mould in a suitable hydraulic press and apply pressure gradually (see the Note) until the desired pressure is attained. As specified in 10.2.1.2, this pressure shall be between 15 MPa and 70 MPa, depending on the particular type of PTFE being used. 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.

10.6.1.4 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 in Table 6.

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. Since a burning cigarette would exceed 260 °C, those working with PTFE resins should ensure that tobacco is not contaminated.

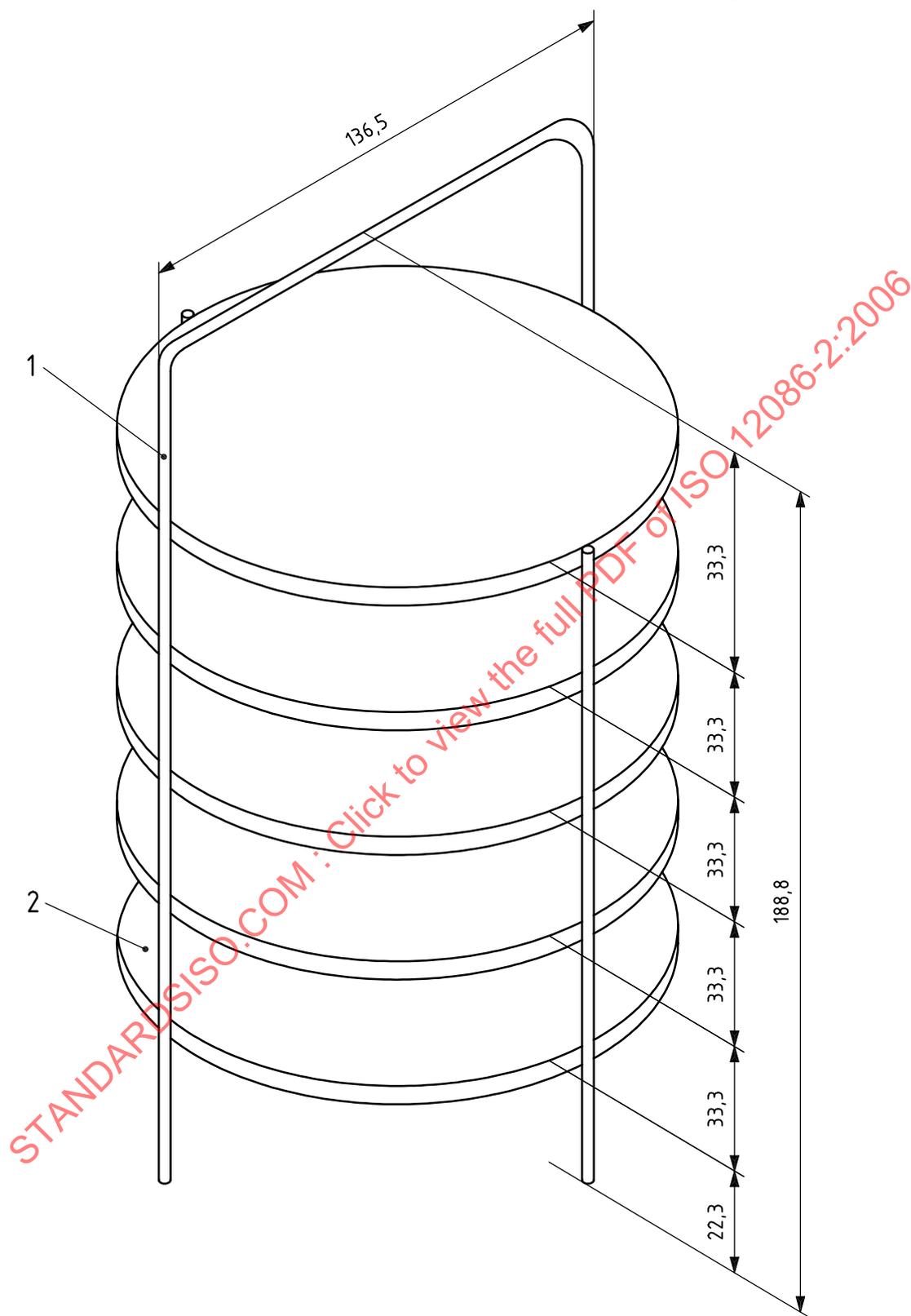
Table 6 — Sintering conditions for preparing SSG or ESG test specimens

Initial temperature	290
Rate of heating, °C/h	120 ± 10
Hold temperature, °C	380 ± 6
Hold time, minutes	
for SSG specimens	$30 \begin{smallmatrix} +2 \\ 0 \end{smallmatrix}$
for ESG specimens	360 ± 5
Cooling rate to 294 °C, °C/h	60 ± 5
Second hold temperature, °C	294 ± 6
Second hold time, minutes	$24 \begin{smallmatrix} +0,5 \\ 0 \end{smallmatrix}$
Time to room temperature, minutes	> 30

Although the rate of heat application while sintering a preform is not critical, the cooling cycle during sintering is most important and the conditions cited in this procedure shall be followed very closely. If they are not followed, the crystallinity of the discs and the resulting physical properties will be markedly changed. Therefore, the use of a cam-controlled, automatically timed, programmed oven is recommended for the most precise sintering-cycle control. This automatic control also permits the hood window to be left down during the entire sintering procedure, an important safety consideration.

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 6. The rack, as shown in Figure 13, 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 less 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.) Consult ASTM D 4894 or ASTM D 4895 for additional details.

Dimensions in millimetres



Key

- 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.

Figure 13 — Rack for sintering oven