INTERNATIONAL
STANDARD

ISO
12086-2

First edition
1995-12-15

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

Reference number
ISO 12086-2:1995(E)

Contents

Page

1 Scope 1

2 Normative references 1

3 Definitions 3

4 Symbols and abbreviations 3

5 Sampling 4

6 Preparation of test specimens 4

7 Conditioning and test conditions 4

8 General testing of fluoropolymers 4

8.1 Electrical properties 4

8.2 Mechanical properties 5

8.3 Thermal-transition temperatures 6

8.4 Density 8

8.5 Flammability by oxygen index 8

8.6 Particle size and size distribution 8

9 Testing of fluoropolymer dispersions 13

9.1 General 13

9.2 Preparation of test samples 13

9.3 Isolation of PTFE from dispersion 14

9.4 Coagulum in dispersions 14

9.5 Percentage polymer and surfactant in aqueous dispersions 15

9.6 PTFE solids content by hydrometer 15

9.7 pH of dispersions 16

10 Testing of PTFE and closely related materials 16

10.1 General 16

10.2 Preparation of test specimens by moulding 16

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

Printed in Switzerland

10.3 Bulk density **18**

10.4 Extrusion pressure **23**

10.5 Powder-flow time **27**

10.6 Standard specific gravity (SSG), extended specific gravity (ESG), and thermal-instability index (TII) **27**

10.7 Stretching-void index (SVI) **30**

11 Testing of conventionally melt-processible fluoropolymers .. **32**

11.1 Preparation of test specimens by moulding **32**

11.2 Melt mass-flow rate (MFR) and melt volume-flow rate (MVR) **33**

12 Other test methods used with fluoropolymers **35**

12.1 Brittleness temperature of plastics and elastomers by impact **35**

12.2 Coefficients of static and kinetic friction **35**

12.3 Zero-strength time **35**

Annexes

A Listing of test methods (alphabetical order) **36**

B Designatory properties for common fluoropolymer types with cross-reference listing to the tables for codes in ISO 12086-1 and the test methods in ISO 12086-2 .... **37**

C Bibliography **40**

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.

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

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

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*

Annexes A, B and C of this part of ISO 12086 are for information only.

Plastics — Fluoropolymer dispersions and moulding and extrusion materials —

Part 2:

Preparation of test specimens and determination of properties

WARNING — This part of ISO 12086 may involve hazardous materials, operations and equipment. It does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 12086 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. The warnings in subclauses 8.6.1.1, 9.7 and 10.6.1.3 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 standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 12086. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 12086 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 75-2:1993, *Plastics — Determination of temperature of deflection under load — Part 2: Plastics and ebonite.*

ISO 178:1993, *Plastics — Determination of flexural properties.*

ISO 179:1993, *Plastics — Determination of Charpy impact strength.*

ISO 180:1993, *Plastics — Determination of Izod impact strength.*

ISO 291:1977, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 293:1986, *Plastics — Compression moulding test specimens of thermoplastic materials.*

ISO 472:1988, *Plastics — Vocabulary.*

ISO 527-1:1993, *Plastics — Determination of tensile properties — Part 1: General principles.*

ISO 527-2:1993, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics.*

ISO 527-3:1995, *Plastics — Determination of tensile properties — Part 3: Test conditions for films and sheets.*

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

ISO 1043-1:1987, *Plastics — Symbols — Part 1: Basic polymers and their special characteristics.*

ISO 1043-2:1988, *Plastics — Symbols — Part 2: Fillers and reinforcing materials.*

ISO 1133:1991, *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics.*

ISO 1148:1980, *Plastics — Aqueous dispersions of polymers and copolymers — Determination of pH.*

ISO 1183:1987, *Plastics — Methods for determining the density and relative density of non-cellular plastics.*

ISO 4589:1984, *Plastics — Determination of flammability by oxygen index.*

ISO 8962:1987, *Plastics — Polymer dispersions — Determination of density.*

ISO 12000:—[1], *Plastics/rubber — Polymer dispersions and rubber latices (natural and synthetic) — Definitions and review of test methods.*

ISO 12086-1:1995, *Plastics — Fluoropolymer dispersions and moulding and extrusion materials — Part 1: Designation system and basis for specifications.*

IEC 93:1980, *Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials.*

IEC 243-1:1988, *Methods of test for electric strength of solid insulating materials — Part 1: Tests at power frequencies.*

IEC 250:1969, *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 746-79(1987), *Test method for brittleness temperature of plastics and elastomers by impact.*

ASTM D 1430-91a, *Specification for polychlorotrifluoroethylene (PCTFE) plastics.*

ASTM D 1457-91a, *Specification for PTFE molding and extrusion materials.*

ASTM D 1894-93, *Test method for static and kinetic coefficients of friction of plastic film and sheeting.*

ASTM D 3418-83(1988), *Test method for transition temperatures of polymers by thermal analysis.*

ASTM D 4052-91, *Test method for density and relative density of liquids by digital density meter.*

ASTM D 4591-93a, *Test method for determining temperatures and heats of transitions of fluoropolymers by differential scanning calorimetry.*

ASTM D 4894-91a, *Specification for polytetrafluoroethylene (PTFE) granular molding and ram extrusion materials.*

ASTM D 4895-91a, *Specification for polytetrafluoroethylene (PTFE) resins produced from dispersion.*

BS 3406:Part 5:1983, *Methods for determination of particle size distribution — Part 5: Recommendations for electrical sensing zone method (the Coulter principle).*

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

---

[1] To be published.

3 Definitions

3.1 The terminology given in ISO 472 is applicable to this part of ISO 12086, except for terms defined in 3.2. The terms listed in 3.1.1 to 3.1.3 are repeated from ISO 472 to be sure there is no misunderstanding.

3.1.1 dispersion: A heterogeneous system in which a finely divided material is distributed in another material.

3.1.2 fluoroplastic: A plastic based on polymers made with monomers containing one or more atoms of fluorine, or copolymers of such monomers with other monomers, the fluoromonomer(s) being in the greatest amount by mass.

3.1.3 latex: A colloidal aqueous dispersion of a polymeric material.

3.2 For the purposes of this part of ISO 12086, the following additional definitions apply.

3.2.1 amorphous: Noncrystalline, or devoid of regular structure.

3.2.2 bulk density: The mass (in grams) per litre of material, measured under the conditions of the test.

3.2.3 copolymer: A polymer formed from two or more types of monomer.

3.2.4 emulsion polymer (as it applies to fluoropolymer materials)**:** Material isolated from its polymerization medium as a colloidal aqueous dispersion of the polymer solids.

NOTE 1 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.

3.2.5 fluorocarbon plastic: A plastic based on polymers made from perfluoromonomers only.

3.2.6 fluoropolymer: Synonymous with *fluoroplastic* (see 3.1.2).

3.2.7 melt-processible: Capable of being processed by, for example, injection moulding, screw extrusion, and other operations typically used with thermoplastics.

3.2.8 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 2 With PTFE, "moulding" and "compaction" are terms used interchangeably with "preforming".

3.2.9 sintering (as it applies to PTFE)**:** A thermal treatment during which the material is melted and recrystallized by cooling, with coalescence occurring during the treatment.

3.2.10 standard specific gravity (SSG): The 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 3 The SSG of unmodified PTFE is inversely related to its molecular mass.

3.2.11 suspension polymer: A polymer isolated from its liquid polymerization medium as a solid having a particle size well above colloidal dimensions.

3.2.12 zero-strength time (ZST): A measure of the relative molecular mass of PCTFE.

4 Symbols and abbreviations

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:

PTFE	polytetrafluoroethylene
PFA	perfluoro(alkoxy alkane)
FEP	perfluoro(ethylene-propene) copolymer
EFEP	ethylene-tetrafluoroethylene-hexafluoropropene copolymer
TFE/PDD	tetrafluoroethylene-perfluoro(dioxole) copolymer
VDF/HFP	vinylidene fluoride-hexafluoropropene copolymer
VDF/TFE	vinylidene fluoride-tetrafluoroethylene copolymer
VDF/TFE/HFP	vinylidene fluoride-tetrafluoroethylene-hexafluoropropene copolymer

ETFE	ethylene-tetrafluoroethylene copolymer
PVDF	poly(vinylidene fluoride)
VDF/CTFE	vinylidene fluoride-chlorotrifluoroethylene copolymer
PCTFE	polychlorotrifluoroethylene
PVF	poly(vinyl fluoride)
ECTFE	ethylene-chlorotrifluoroethylene copolymer

4.3 For the purposes of this part of ISO 12086, the following abbreviated terms apply in addition to those given in 3.2 and 4.2.

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

The materials should preferably be sampled in accordance with ISO 842. Adequate statistical sampling shall be considered an acceptable alternative. Special considerations are included in the pertinent clause.

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 tests 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 tests require no conditioning.

NOTE 4 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 °C ± 2 °C for determining specific gravity, tensile properties, and electrical properties only. (See note 5 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 5 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 250. 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 6 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
Q	6,0 to < 6,5	**R**	0,003 0 to < 0,003 5
S	6,5 to < 7,0	**S**	0,003 5 to < 0,004 0
T	7,0 to < 8,0	**T**	0,004 0 to < 0,006 0
U	8,0 to < 9,0	**U**	0,006 0 to < 0,008 0
V	9,0 to < 10,0	**V**	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 243-1. Codes for values of the property are given in table 3.

NOTE 7 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 93. 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 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 527-3:1995, 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 mm ± 0,5 mm (see note 8). 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 mm ± 0,13 mm, and the speed of testing shall be 50 mm/min ± 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 extensiometer may be used to determine the elongation.

NOTE 8 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.: (078) 15.10.77, Fax: (078) 41.05.45, and Accurate Steel Rule Die Co., 22 West 21st Street, New York, NY 10010, USA, Tel.: (212) 242-3606. 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.

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.

8.2.2.1.3 ASTM D 1457 includes a summary of the precision for tensile strength and percentage elongation at break for PTFE, FEP, and PFA. Additional round-robin testing on use of the microtensile die is in progress within ASTM Committee D-20.

Bias is a systematic error that contributes to the difference between a test result and a true (or reference) value. There are no recognized standards on which to base an estimate of bias for this test procedure.

8.2.2.2 Fluoropolymers for which tensile modulus is to be determined

Determine tensile properties in accordance with ISO 527-2:1993 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.

Dimensions in millimetres

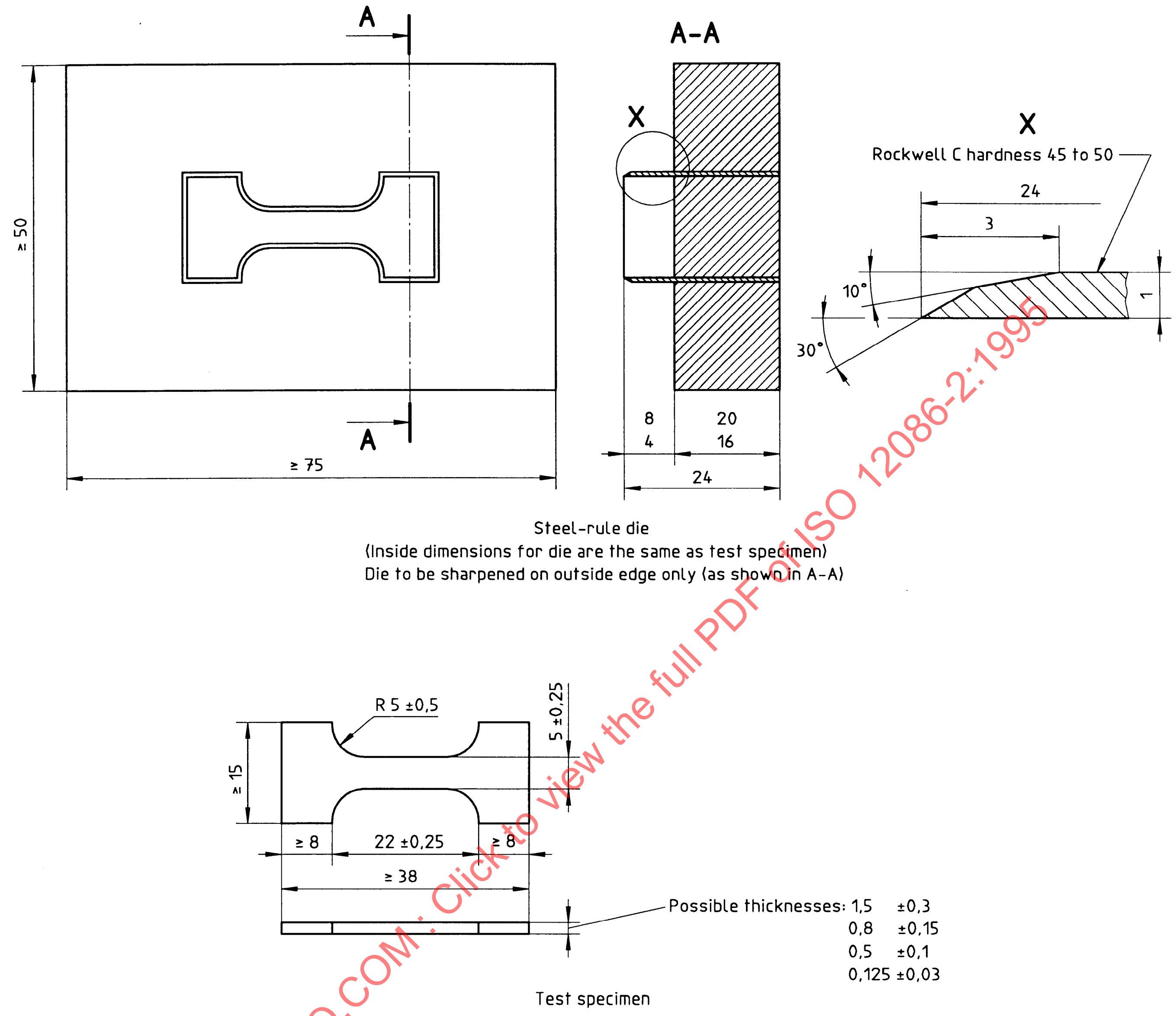


Figure 1 — Microtensile die

8.3.2 Glass-transition temperature(s)

Determine these temperatures in accordance with the procedures of ASTM D 3418.

8.3.3 Melting-peak temperature

Melting-peak temperature characteristics are specific for fluoropolymers and help identify a particular material. The procedures of ASTM D 4591 supplemented by ASTM D 3418 are appropriate for this determination.

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 mg ± 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.

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 °C ± 10 °C. The first melting point is normally at least 5 °C higher than the second melting point.

8.3.3.2 Use differential scanning calorimetry (DSC) as described in ASTM D 3418 and ASTM D 4591 for this determination. The heating rate shall be 10 °C ± 1 °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 demonstrates 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 test in accordance with ISO 1183. If method D is used, the solution in the tube shall have a linear density gradient as specified in the table appropriate for the fluoropolymer being tested.

8.5 Flammability by oxygen index

Use the procedure of ISO 4589.

8.6 Particle size and size distribution

The wet- and dry-sieve procedures of 8.6.1 and 8.6.2 are widely used with PTFE and closely related materials. The resistance-variation test procedure in 8.6.3 (the Coulter principle) is often used with PVDF, PTFE filler resin, and fine-cut suspension powders. The light-scattering procedures in 8.6.4 are becoming more widely used with all the fluoropolymers. 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 part of ISO 12086. ASTM F 660 (see annex C) provides a standard practice for comparing particle size determined with different types of automatic particle counter.

8.6.1 Wet-sieve analysis

8.6.1.1 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 has been reported as giving equivalent results when used as a replacement for perchloroethylene.

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.

8.6.1.2 Apparatus and materials

8.6.1.2.1 Balance, capable of weighing to ± 0,1 g.

8.6.1.2.2 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 annex C). 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.

8.6.1.2.3 Ventilated hood.

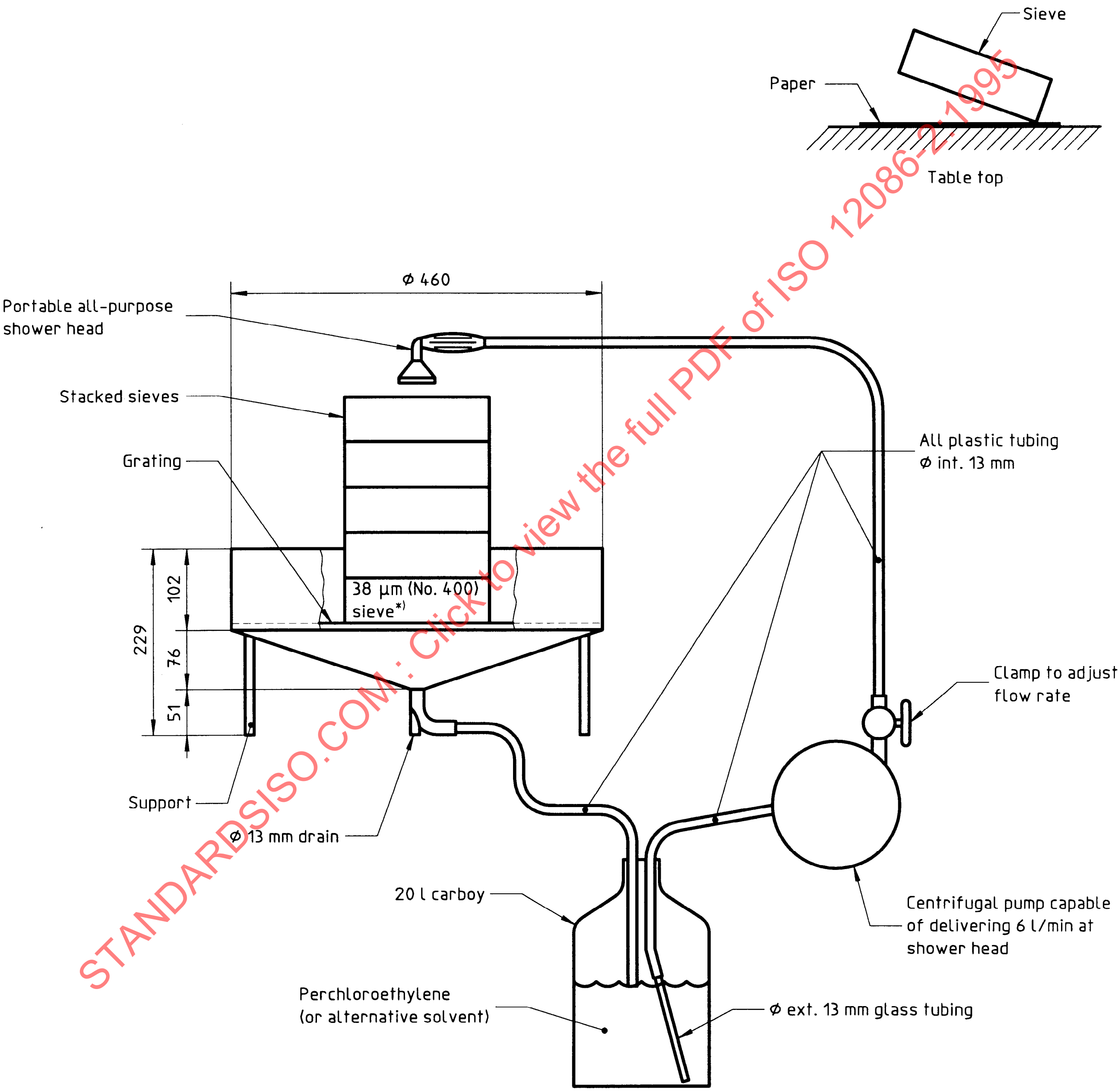
8.6.1.2.4 Six tared beakers, capacity 150 ml.

NOTE 9 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.

8.6.1.2.5 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.

Dimensions in millimetres



*) Use a fine sieve to prevent material from going into the reservoir. A standard 38 µm sieve has been found to be convenient.

Figure 2 — Apparatus for wet-sieve analysis

8.6.1.3 Spray liquid, 20 litres.

See the comments and warning in 8.6.1.1. Although perchloroethylene has been the usual choice, an alternative liquid may be used after its applicability and hazards associated with its use have been investigated thoroughly and use of the liquid shown to be satisfactory.

8.6.1.4 Procedure

8.6.1.4.1 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 ± 0,5 l/min.

8.6.1.4.2 Place the weighed resin on the top sieve and spray it with the organic spray liquid for 1 min ± 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.

8.6.1.4.3 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 8.6.1.4.4.

8.6.1.4.4 Repeat the procedure specified in 8.6.1.4.2 and 8.6.1.4.3 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 ± 0,1 g.

8.6.1.4.5 Record the mass of resin on each sieve.

8.6.1.4.6 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.

8.6.1.5 Expression of results

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

> Net percentage of resin on sieve $Y = F \times$ mass, in grams, on sieve Y

where

> $F = 2$ for a 50 g test sample;
>
> $F = 10$ for 10 g test sample.

8.6.1.5.2 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 Y

EXAMPLE

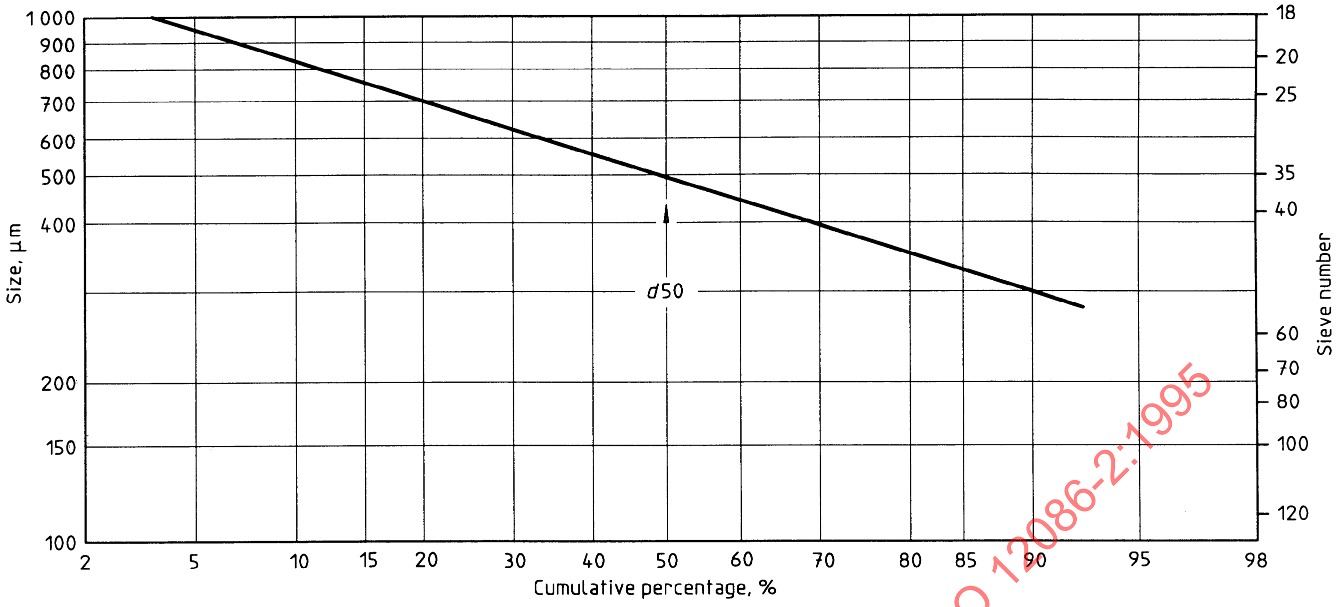
Cumulative percentage on 500 µm (No. 35) sieve equals net percentage on 1,4 mm (No. 14) plus net percentage on 1,00 mm (No. 18) plus net percentage on 710 µm (No. 25) plus net percentage on 500 µm (No. 35) sieve.

8.6.1.5.3 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 ($d50$). Take this value as the average particle size.

It is permissible to carry out the calculation of $d50$ by use of computer programmes that provide "best-fit" analysis using linear regression procedures involving a log-normal model.

8.6.1.6 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 be only relative numbers. The 95 % confidence limits based on a limited series of tests are ± 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 is being made.



Sieve No.	Sieve opening 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

8.6.2 Dry-sieve analysis

8.6.2.1 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.

8.6.2.2 Apparatus

8.6.2.2.1 Balance, capable of weighing to ± 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 annex C). 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 Mechanical sieve shaker, capable of imparting a uniform rotary and tapping action.

8.6.2.2.4 Freezer: any commercial ice freezer (a dry-ice chest may be used).

8.6.2.3 Procedure

8.6.2.3.1 Place 50 g ± 0,1 g of the sample in an aluminium pan, and cool the pan and contents to less than 10 °C.

8.6.2.3.2 Measure the tare mass of each of the sieves listed in 8.6.2.2.2. Place the conditioned test sample on the top sieve of the assembly and shake in the sieve shaker for 10 min ± 0,5 min. The dew-point 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.

8.6.2.4 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 $Y = 2 \times$ mass, in grams, 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 Y

EXAMPLE

Cumulative percentage on 500 µm (No. 35) sieve equals net percentage on 1,4 mm (No. 14) plus net percentage on 1 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 throught the points and read the particle size at the 50 % cumulative percentage point (d50). Take this value as the average particle size.

It is permissble to carry out the calculation of d50 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

The test precision is ± 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 is being made.

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

A test method for use of this equipment with powders is given in BS 3406:Part 5:1993.

8.6.3.1 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.

8.6.3.2 Apparatus

8.6.3.2.1 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.

8.6.3.2.2 Analytical balance.

8.6.3.2.3 Magnetic stirrer.

8.6.3.2.4 Ultrasonic tank.

8.6.3.3 Procedure

8.6.3.3.1 Prepare a solution of a non-ionic surfactant (see note 10) at a concentration of 0,2 g/l to 0,3 g/l in an aqueous electrolyte, such as a 1 % (m/V) solution of sodium chloride or a special 1 % (m/V) saline solution (see note 11). 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 (see note 12).

NOTES

10 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.

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

12 Available from the Millipore Corporation.

13 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.

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

8.6.3.3.3 Stir the slurry of powder using the magnetic stirrer at a high speed to obtain 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 rpm to 750 rpm.

8.6.3.3.4 Put the beaker into an 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 in order to avoid any undesirable effects of warming on the morphology or fracture of the suspended particles. An ultrasonic cell disrupter operated at 20 kHz for four minutes should be sufficient to obtain a uniform suspension of the fluoropolymer powder.

8.6.3.3.5 Resume stirring with the magnetic stirrer just rapidly enough to maintain a uniform suspension while removing 100 µl to 200 µl of the suspension depending on the concentration. The analysis is made in a special 200 ml round-bottom container filled with the special 1 % (m/V) saline solution described in 8.6.3.3.1. Be careful to avoid air bubbles when filling.

8.6.3.3.6 Count particles in time mode using three measurements.

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

8.6.3.4 Expression of results

Plot a volume or mass percent curve on suitable graph paper as described for sieve analysis in 8.6.1.5.3.

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

8.6.4.1 Significance and use

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

8.6.4.2 Apparatus

The particle-size analyzer 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 analyzer system or subsystem is optimum for the size range of the powder being tested.

8.6.4.3 Procedure

The instructions from the manufacturer of the instrument shall be followed 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 to define characteristics of the polymer and of the dispersion of the polymer. These methods supplement such standards as ISO 12000 and ISO 8962 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 rpm to 4 rpm, 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 by inserting 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

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 an absolute pressure of 10 mm of mercury; a desiccator; a balance.

9.3.2 Materials

Methanol, acetone, filter fabric, PTFE dispersion, and deionized water.

9.3.3 Procedure to isolate 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 a 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 ± 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, using 200 ml of deionized or distilled water at 85 °C ± 5 °C. Then repeat 9.3.3.6 once, using 150 ml of acetone.

9.3.3.9 Place the washed sample in an 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 a desiccator before weighing. Repeated drying, cooling, and weighing may be necessary.

NOTE 14 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

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

9.4.1 Apparatus

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; a balance capable of weighing to ± 1 mg.

9.4.2 Materials

Distilled water and PTFE dispersion.

9.4.3 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 the coagulated polymer on the screen with 25 ml of distilled water from a wash bottle. Carefully remove the screen from the funnel and dry at 120 °C ± 5 °C for 2 h. Weigh the screen and coagulum to 1 mg after allowing it to cool to room temperature in a desiccator. Record the mass as m_3.

9.4.4 Expression of results

Calculate the coagulum content as follows:

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

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(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 dispersions

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

An aluminium weighing dish, an oven capable of reaching 120 °C ± 5 °C, an oven capable of reaching 380 °C ± 10 °C, 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 weigh immediately to 0,1 mg (m_2). Dry the test portion for 2 h at 120 °C ± 5 °C. Reweigh the test portion to 0,1 mg (m_3) after cooling to room temperature in a desiccator. After weighing, evaporate the surfactant by placing the test portion in an oven at 380 °C ± 5 °C for 35 min ± 1 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 nonvolatile 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 nonvolatile portions of the surfactant.

9.5.5 Precision and bias

The precision and bias of this method are under investigation by a task group of ASTM Subcommittee D-20.15.12.

9.6 PTFE solids content by hydrometer

9.6.1 Significance and use

An approximate solids content in 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 15 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

A hydrometer or set of hydrometers capable of measuring specific gravity from 1,000 to 1,550 with a precision of ± 0,001; a graduated cylinder large enough to hold the hydrometer with reasonable clearance from the sidewalls of the hydrometer. Automated apparatus to make this determination shall be an acceptable alternative after equivalence of results has been determined. An example of such apparatus is mentioned 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

The precision and bias of this method are under investigation by a task group of ASTM Subcommittee D-20.15.12.

9.7 pH of dispersions

The test method shall be in accordance with ISO 1148. Although pH is not a designatory property in part 1 of this International Standard, the test method is included because some buyers or sellers attach importance to the pH of the dispersion.

NOTE 16 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 generally are 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 thermosplastic 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.

10.2.1.1.2 Hydraulic press, capable of 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 g ± 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 straightedge in contact with and across the top of the mould cavity.

NOTE 17 The adjustable cavity depth permits easier levelling for 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.

Dimensions in millimetres

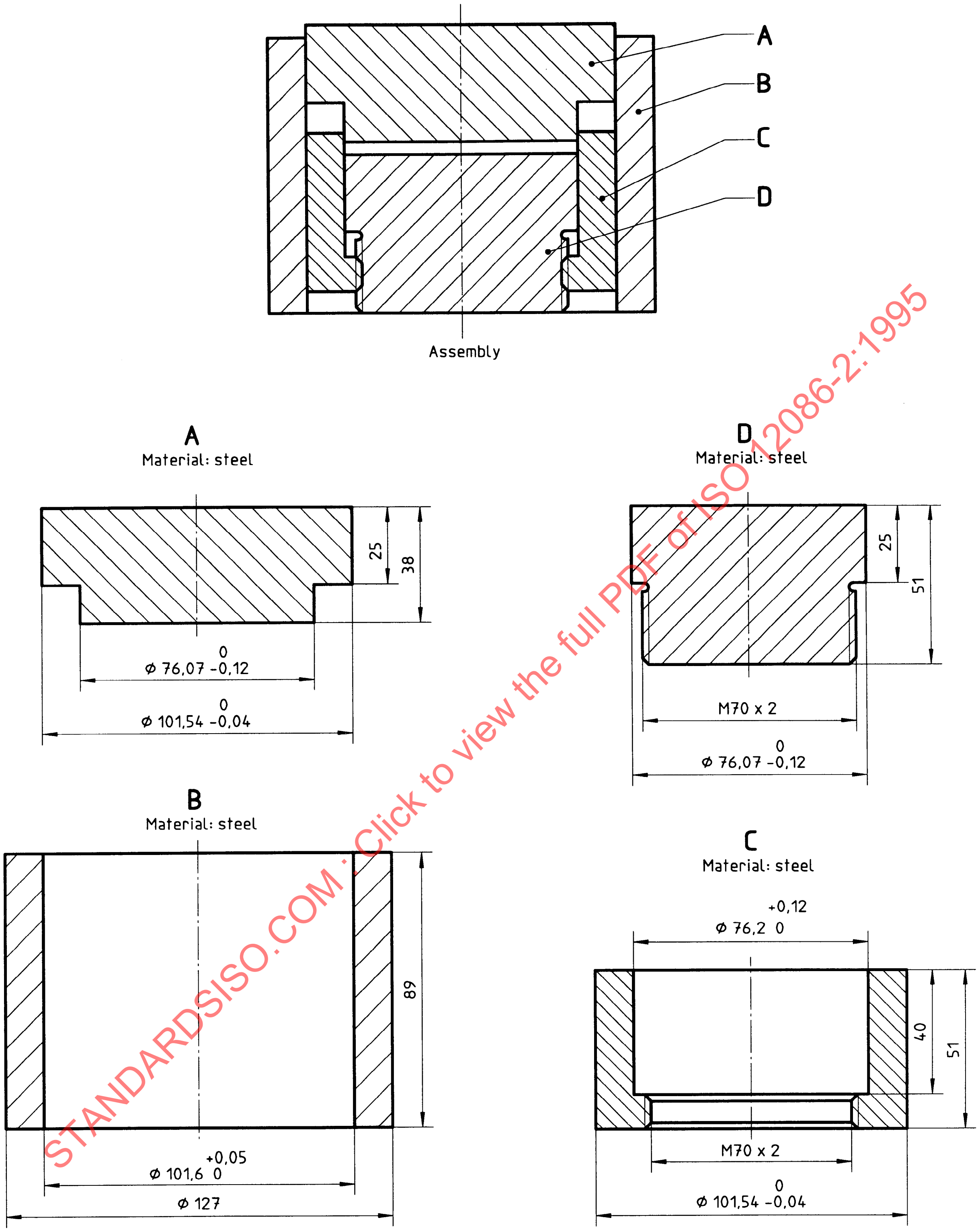


Figure 4 — Mould assembly for preparation of specimens for the determination of tensile properties

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 18 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 s ± 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 10.6.1.3 and table 6.

10.2.2 Test billets for 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 the following paragraphs shall be used to measure this property. This procedure also may be found in ASTM D 1457 and ASTM D 4894.

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.

NOTE 19 A "Vibra-Flow" feeder, Type F-T01A, with trough, available from FMC, Material Handling Division, FMC Building, Homer City, PA 15748, USA, Tel.: (412) 479-8011, Fax: (412) 479-3400, has been found satisfactory for this purpose. 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.

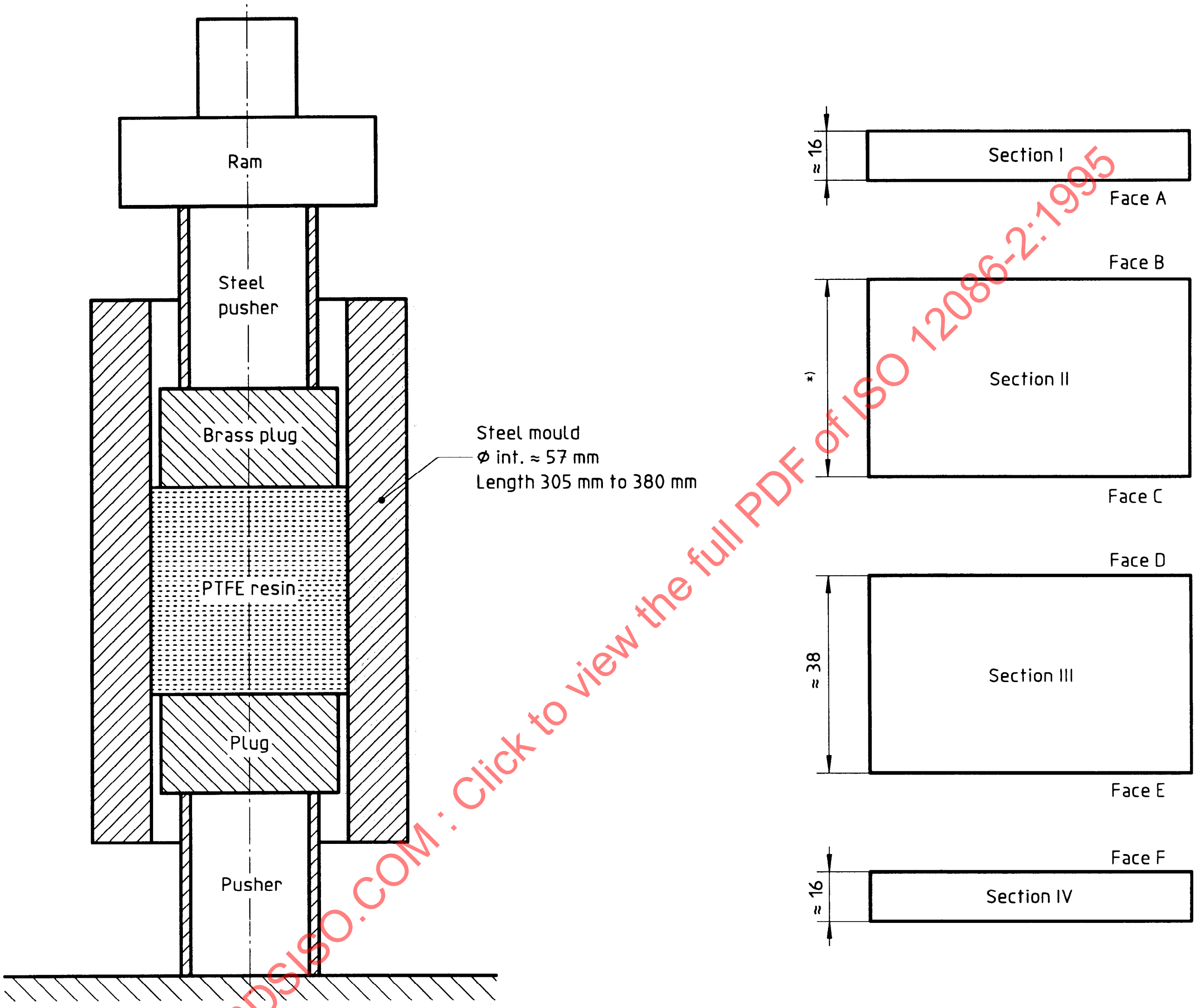
10.3.2.3 Controller.

NOTE 20 A "Syntron" controller, Type CSCRB1, available from FMC, address as given in note 19, has been found satisfactory for this purpose. 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.

10.3.2.4 Volumetric cup and cup stand, as shown in figure 7. 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 g ± 0,5 g. The top and bottom faces of the volumetric cup and the cup stand shall be machined plane and parallel.

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.

Dimensions in millimetres



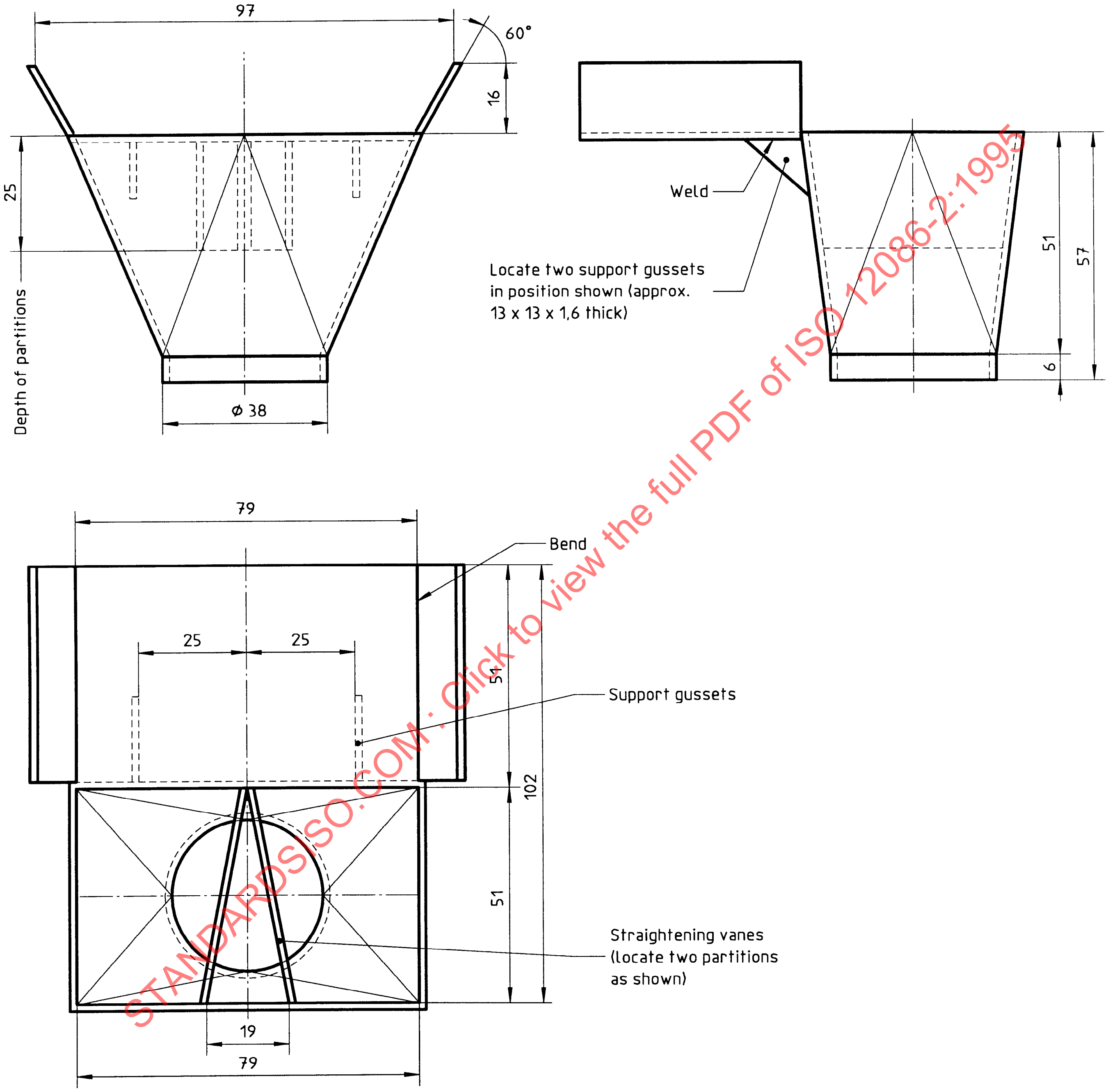
a) Preforming of billet

b) Sectioned billet

*) Variable, between 25 mm and 40 mm, depending on the total height of the billet after sintering.

Figure 5 — Apparatus for preforming PTFE test billet

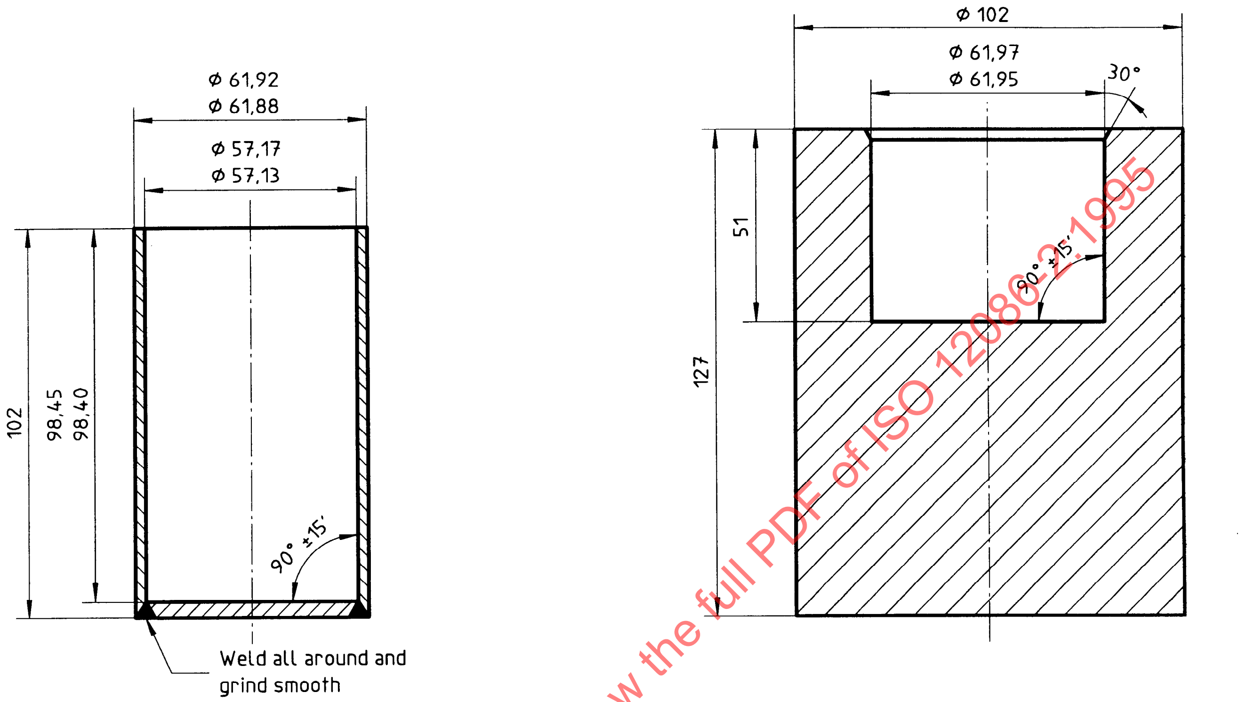
Dimensions in millimetres



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

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

Dimensions in millimetres



NOTE:
a) Top and bottom shall be flat and parallel within 0,05 mm.
b) Inside bottom corner shall be square as shown.

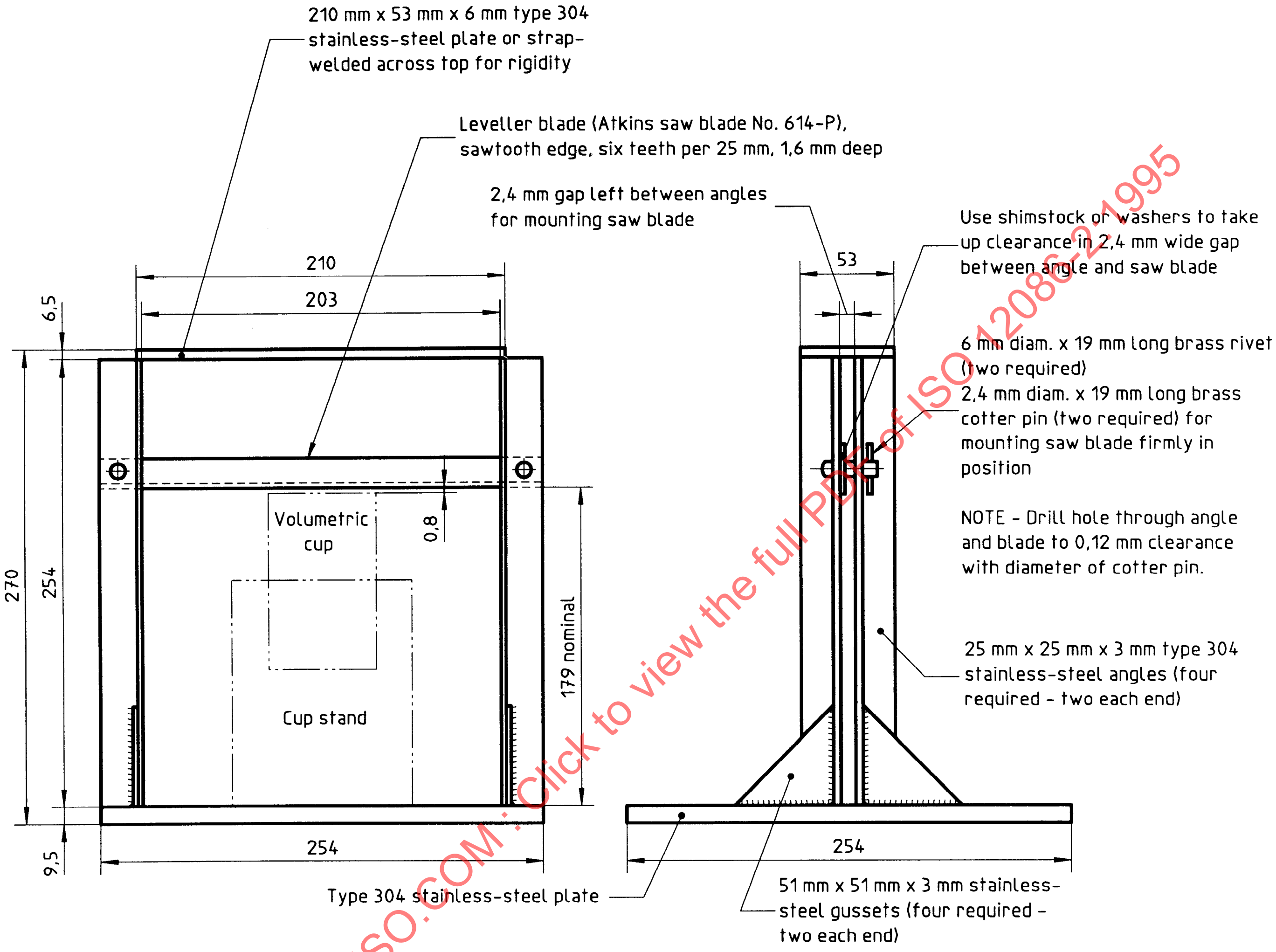
Volumetric cup
Material: type 304 stainless seamless tubing

NOTE:
a) Top and bottom shall be flat and parallel within 0,05 mm.
b) Remove all external sharp corners.
c) Bottom of hole shall be square with centreline and with top surface of stand.

Cup stand
Material: 17 S-T aluminium (or equivalent)

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

Dimensions in millimetres



NOTE — Base plate shall be flat and parallel. Saw blade, when mounted, shall be square to and parallel with base plate to within 0,12 mm from end to end.

Height of saw blade shall be adjusted to give 0,8 mm or less clearance between blade and assembled cup and cup stand (as indicated by phantom lines).

Welded construction where indicated.

Material: as noted.

Figure 8 — Leveller stand for the determination of bulk density

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 mm ± 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.

10.3.4 Expression of results

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

Mass of resin in cup × 4 = Bulk density

10.3.5 Precision and bias

A precision statement for use with this procedure is under development. The procedure in this test method has no bias because the value of bulk density can be defined only in terms of a test method.

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 °C ± 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.

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

NOTE 21 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.

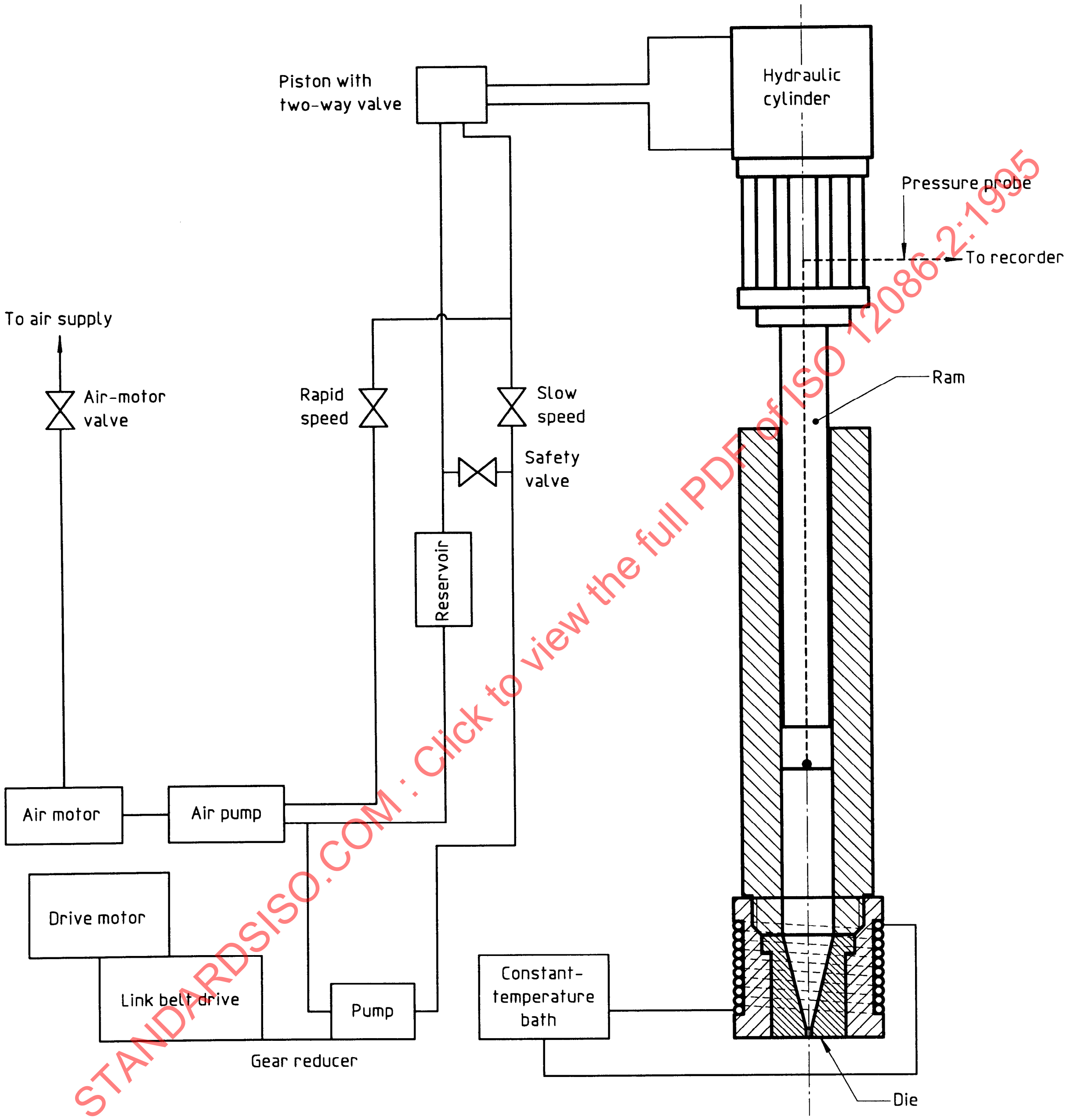
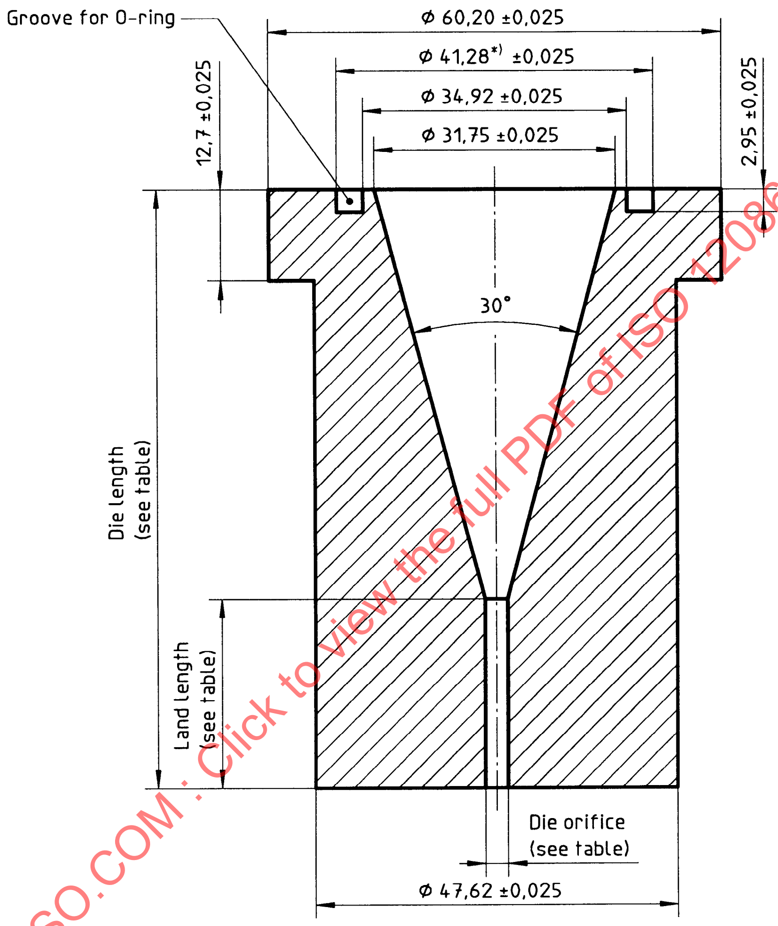


Figure 9 — Paste extruder for determining extrusion pressure

Dimensions in millimetres



*) 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 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 liquid (see note 22). The density shall be determined at 25 °C using method ASTM D 4052 that calls for the use of a commercial density meter that will give the density to four significant figures (see note 23), 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.

NOTES

22 Isopar K, available from Exxon Co., has been found satisfactory 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.

23 A Mettler/Paar DMA 40 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.

10.4.3.4 Blend the mixture by placing the jar on rubber-coated mill rolls and rolling it at 30 rpm for 25 min ± 5 min, by fastening the jar to a "windmill" type blender (see note 24) 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.

NOTE 24 A Gilson spinning-wheel mixer has been found suitable for this purpose. 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.

10.4.3.5 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.

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 g/min ± 1,0 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

The test precision and bias are to be determined by round-robin testing.

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 fom 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.

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

10.6.1 Preparation of test specimens

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.

Dimensions in millimetres

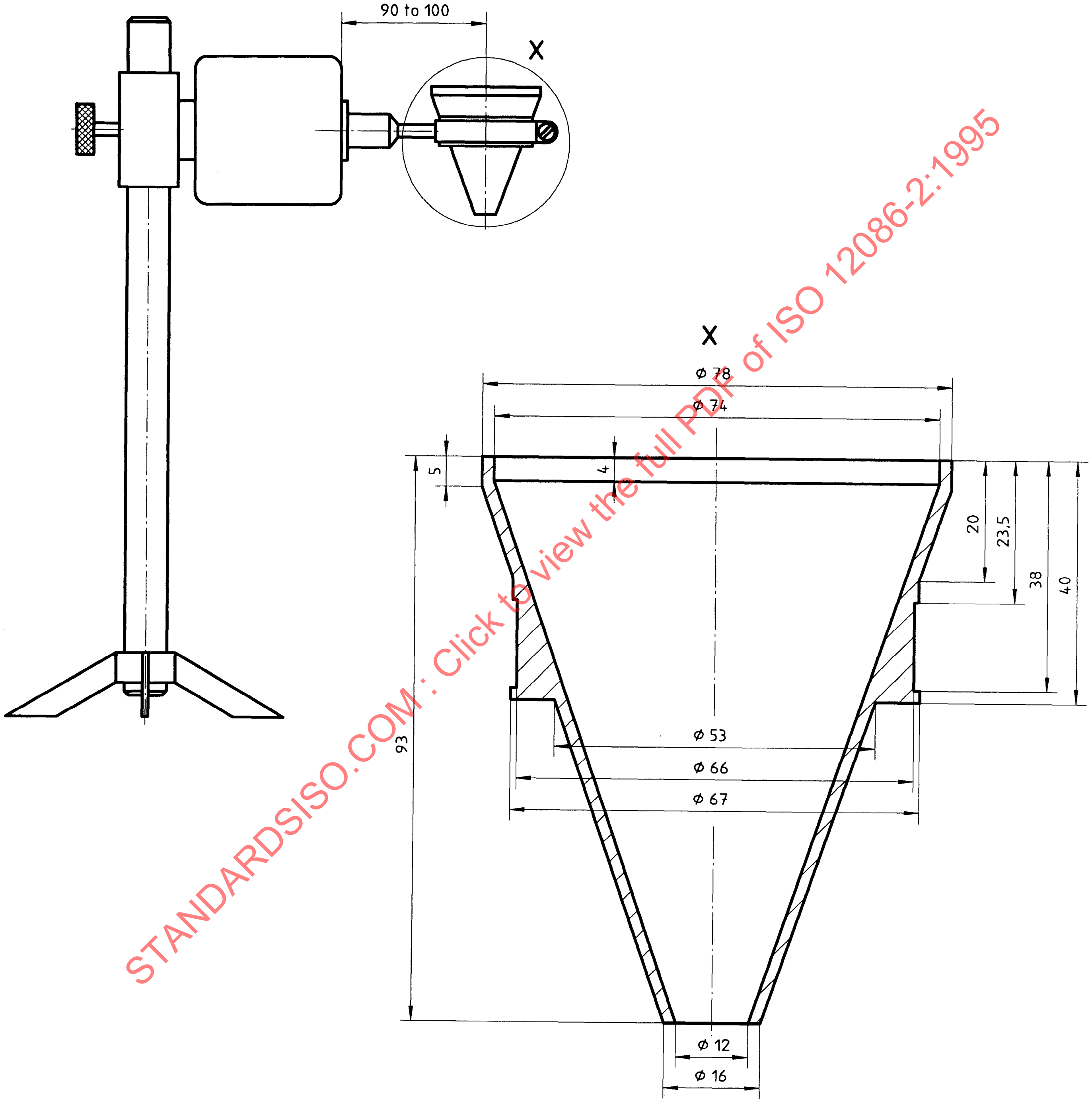
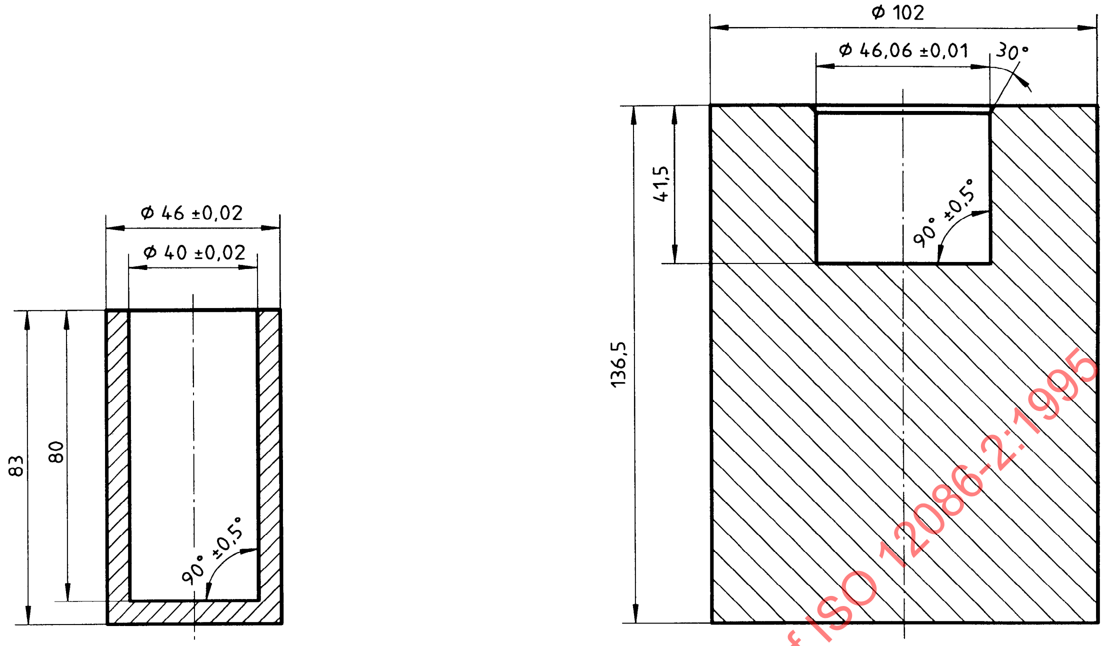


Figure 11 — Funnel for determination of flow time

Dimensions in millimetres



Cup
Material: stainless steel

Cup stand
Material: aluminium

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

10.6.1.1 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 pressing. Place flat aluminium foil discs, normally 0,013 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 preforming operations shall be carried out in a constant-temperature room at 23 °C ± 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.

10.6.1.2 Weigh out 12,0 g ± 0,1 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 g ± 0,1 g test sample. Insert the mould in a suitable hydraulic press and apply pressure gradually (see note 25) 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 25 As a guide, increasing the pressure at a rate of 3,5 MPa/min is suggested until the desired maximum is attained.

10.6.1.3 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.

Although the rate of pressure application while marking 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.)

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

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

10.6.1.4 After cooling and equilibrating the sintered pieces at room temperature, remove all flash from each specimen so that no air bubbles will cling to the edges when the specimen is immersed in the solution for weighing during determination of the specific gravity. It is recommended that clean cotton gloves be worn when test specimens are being handled.

10.6.2 Determination of SSG and 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 ISO 1183. Add two drops of a wetting agent (see 8.6.3.3.1) to the water in order to reduce the surface tension and ensure complete wetting of the specimen.

NOTE 26 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 (e.g. one reading to ±0,1 °C) is used in the liquid and the temperature is kept at at least 22 °C.

10.6.3 Determination of TII

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

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

10.6.4 Precision and bias

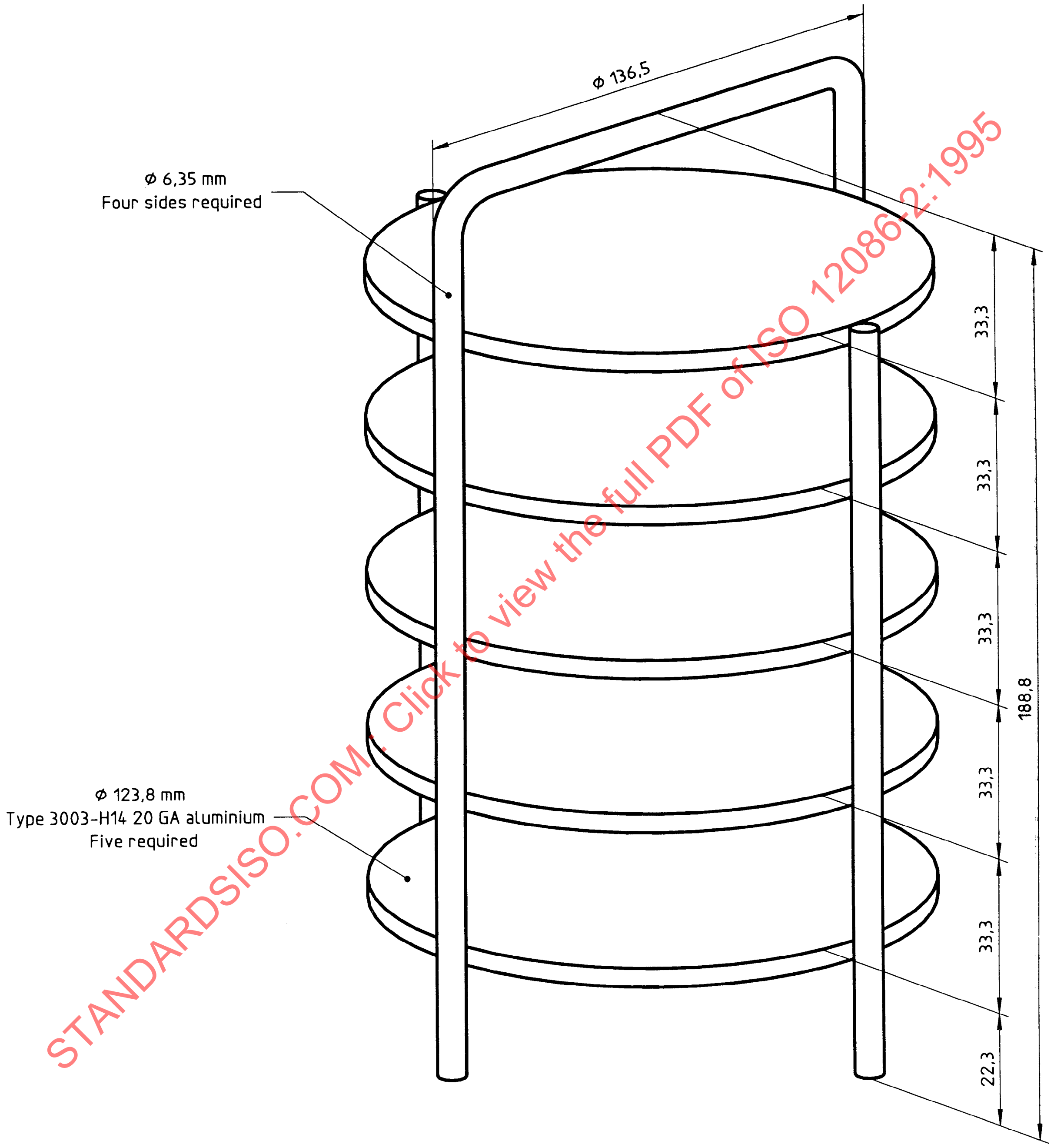
The precision of this method has not yet been determined. There are no recognized standards on which to base an estimate of bias for this test procedure.

10.7 Stretching-void index (SVI)

10.7.1 Significance and use

This test method compares the unstrained specific gravity (USG) of a resin to its strained specific gravity (strained SG). General procedures are given in 10.7.2. The method of calculation of the USG is given in 10.7.2.4 and that of the strained SG is given in 10.7.2.6. 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.

Dimensions in millimetres



NOTE — Aluminium plates tack-welded to rods.

Figure 13 — Apparatus for sintering oven

10.7.2 Procedure

10.7.2.1 Prepare test discs using the general method described in 10.2.1 but modified to take into account the specific conditions given in 10.7.2.2.

10.7.2.2 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 straightedge 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 25 in 10.6.1.2) 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 in 10.6.1.3 for sintering preforms. Use the same sintering conditions as those specified for determining the SSG.

10.7.2.3 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.

10.7.2.4 Determine the USG in accordance with 10.6.2, using the specimen prepared as instructed in 10.7.2.1 and 10.7.2.2.

10.7.2.5 Cut tensile specimens from the disc, using the microtensile die described in 8.2.2.1.2 and figure 1. Clamp the specimen in a tensile-testing machine with essentially equal lengths in each jaw. The initial jaw separation shall be 12,5 mm ± 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 %/min, based on the original gauge length of the specimen. If elongation at break is less than 200 %, discard the result and repeat with fresh tensile specimens.

10.7.2.6 Wait at least ten minutes after release of the stress in 10.7.2.5. Cut off a portion of the stretched part of the specimen having a mass of at least 0,2 g. Determine, in accordance with 10.6.2, the specific gravity of this strained specimen (strained SG).

10.7.3 Expression of results

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

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

10.7.4 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.

11 Testing of conventionally melt-processible fluoropolymers

11.1 Preparation of test specimens by moulding

For tests that require a compression-moulded sheet, the principles of ISO 293 shall be followed with modification of details as presented in 11.1.1. For tests that require an injection-moulded specimen, consult the manufacturer of the resin for suitable moulding conditions.

11.1.1 Prepare a moulded sheet 1,5 mm ± 0,25 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 27). Use steel moulding plates at least 1,0 mm thick and of an area adequate to cover the chase.

NOTE 27 Polyimide films have been found satisfactory for use with fluoropolymers melting above 200 °C. For lower-melting polymers, films of poly(ethylene terephthalate) (PET) work well. In some situations, aluminium foil 0,13 mm to 0,18 mm thick has been used satisfactorily alone or with a high-temperature mould-release agent sprayed on to the aluminium foil to help prevent the foil from sticking to the moulded sheet.

11.1.2 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 7 for the fluoropolymer being used.

Table 7 — Test conditions for moulding test sheets and determining the melt mass-flow rate of melt-processible fluoropolymers

Fluoropolymer	Moulding temperature (codes in brackets) °C	Melt mass-flow rate determination			
		Test temperature (codes in brackets) °C	Load (codes in brackets) kg	Orifice diameter mm	Orifice length mm
FEP	325 (D)	372 (A)	2,16 or 5,0 (3 or 5)	2,095 ± 0,005	8,000 ± 0,025
PFA	380 (E)	372 (A)	5,0 (5)	2,095 ± 0,005	8,000 ± 0,025
ETFE	300 (C)	297 (B)	5,0 (5)	2,095 ± 0,005 or 1,588 ± 0,005	8,000 ± 0,025 or 6,070 ± 0,025
PVDF	205 (A)	230 (E)	21,6 or 5,0 (8 or 5)	2,095 ± 0,005	8,000 ± 0,025
PCTFE	265 (B)	265 (D)	21,6 or 31,6 (8 or 9)	2,095 ± 0,005	8,000 ± 0,025
ECTFE	265 (B)	271,5 (C)	2,16 (5)	2,095 ± 0,005	8,000 ± 0,025

NOTE — It is preferable to use the loads given in this table. In some situations, however, it may be desirable to select a different load, chosen from table 8, in order to meet the requirement of ISO 1133 that the measured MFR shall not be less than 0,1 g/10 min or greater than 100 g/10 min.

Table 8 — Permissible loads, in kilograms, for MFR determinations (preferred loads in heavy type, codes in brackets)

0,325 (1)	3,8 (4)	12,5 (7)
1,20 (2)	**5,0** (5)	**21,6** (8)
2,16 (3)	10,0 (6)	**31,6** (9)

11.1.3 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 7 for the fluoropolymer being used. Remove the assembly from the press and place between two 20 mm ± 7 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.

11.1.4 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.)

11.2 Melt mass-flow rate (MFR) and melt volume-flow rate (MVR)

Melt mass-flow rate or melt volume-flow rate shall be determined in accordance with ISO 1133 as modified by details provided in this part of ISO 12086. 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 part of ISO 12086. If melt volume-flow rate is determined, the melt mass-flow rate may be calculated from the volume rate and the melt density of the polymer.

11.2.1 Principle

The melt-flow rate is determined using the conditions for the fluoropolymer type shown in table 7 and using a modification of the extrusion plastometer described in ISO 1133. The sample may be pellets or powder. For use with semifinished 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.

11.2.2 Apparatus

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

NOTE 28 "Stellite" Grade No. 18, Stellite Div. of Cabot Corp., Kokomo, IN 46901, USA, and "Duranickel" No. 301, Huntington Alloy Co., Huntington, WV 25720, USA, have been found resistant to fluoropolymer resins for this application. 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.

The usual orifice dimensions of 2,095 mm in diameter by 8,000 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 has been shown to give equivalent results may be used in place of the apparatus described here.

11.2.3 Procedure

11.2.3.1 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 29) 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 10 min ± 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 ± 1 °C. See table 7 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.

NOTE 29 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.

11.2.3.2 Measurement of melt mass-flow rate

Make sure that the instrument is clean and level and that a clean orifice of the appropriate size is in place (see table 7). 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 g ± 0,5 g of 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 7. 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. It may be necessary to 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 minutes by multiplying the average mass of the five cuts by 20.

NOTES

30 The extrusion plastometer may be equipped with a device for automatically cutting off the extruded sample at pre-set time-intervals.

31 The intervals between successive cut-offs can be chosen depending on the melt mass-flow rate. The note to table 1 in ISO 1133:1991 provides guidance on selecting test conditions.