INTERNATIONAL STANDARD

ISO 12086-2

Second edition
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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
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Foreword

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

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

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

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

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

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

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

— *Part 1: Designation system and basis for specifications*

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

Plastics — Fluoropolymer dispersions and moulding and extrusion materials —

Part 2: Preparation of test specimens and determination of properties

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

1 Scope

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

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

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

2 Normative references

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

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

ISO 178, *Plastics — Determination of flexural properties*

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

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

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

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

ISO 472, *Plastics — Vocabulary*

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

3 Terms and definitions

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

3.1
dispersion
heterogeneous system in which a finely divided material is distributed in another material

3.2
fluoroplastic
plastic based on polymers made with monomers containing one or more atoms of fluorine, or copolymers of such monomers with other monomers, the fluoromonomer being in the greatest amount by mass

3.3
latex
colloidal aqueous dispersion of a polymeric material

3.4
amorphous
noncrystalline, or devoid of regular structure

3.5
bulk density
mass (in grams) per litre of material, measured under the conditions of the test

3.6
copolymer
polymer formed from two or more types of monomer

3.7
emulsion polymer
⟨fluoropolymer materials⟩ material isolated from its polymerization medium as a colloidal aqueous dispersion of the polymer solids

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

3.8
fluorocarbon plastic
plastic based on polymers made from perfluoromonomers only

3.9
fluoropolymer
synonymous with fluoroplastic (see 3.2)

3.10
melt-processible
capable of being processed by, for example, injection moulding, screw extrusion and other operations typically used with thermoplastics

3.11
preforming
compacting powdered PTFE material under pressure in a mould to produce a solid object, called a preform, that is capable of being handled

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

3.12
sintering
⟨PTFE⟩ thermal treatment during which the material is melted and recrystallized by cooling, with coalescence occurring during the treatment

3.13
standard specific gravity
SSG
specific gravity of a specimen of PTFE material preformed, sintered and cooled through the crystallization point at a rate of 1 °C per minute in accordance with the appropriate sintering schedule as described in this part of ISO 12086

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

3.14
suspension polymer
polymer isolated from its liquid polymerization medium as a solid having a particle size well above colloidal dimensions

3.15
zero-strength time
ZST
measure of the relative molecular mass of PCTFE

4 Abbreviated terms and symbols

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

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

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

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

AF amorphous fluoropolymer

ESG extended specific gravity (see 10.6)

MFR melt mass-flow rate (see 11.2)

MVR melt volume-flow rate (see 11.2)

SSG standard specific gravity (see 10.6)

SVI stretching-void index (see 10.7)

TII thermal-instability index (see 10.6)

ZST zero-strength time (see 12.3)

5 Sampling

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

6 Preparation of test specimens

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

7 Conditioning and test conditions

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

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

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

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

8 General testing of fluoropolymers

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

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

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

8.1 Electrical properties

8.1.1 Dielectric constant and dissipation factor

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

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

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

Table 1 — Codes for test frequencies

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

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

Code	Dielectric constant	Code	Dissipation factor
A	< 1,6	**A**	< 0,000 1
B	1,6 to < 1,8	**B**	0,000 1 to < 0,000 2
C	1,8 to < 2,0	**C**	0,000 2 to < 0,000 4
D	2,0 to < 2,2	**D**	0,000 4 to < 0,000 6
E	2,2 to < 2,4	**E**	0,000 6 to < 0,000 8
F	2,4 to < 2,6	**F**	0,000 8 to < 0,001 0
G	2,6 to < 2,8	**G**	0,001 0 to < 0,001 2
H	2,8 to < 3,0	**H**	0,001 2 to < 0,001 4
I	3,0 to < 3,2	**I**	0,001 4 to < 0,001 6
J	3,2 to < 3,4	**J**	0,001 6 to < 0,001 8
K	3,4 to < 3,6	**K**	0,001 8 to < 0,002 0
L	3,6 to < 4,0	**L**	0,002 0 to < 0,002 2
M	4,0 to < 4,5	**M**	0,002 2 to < 0,002 4
N	4,5 to < 5,0	**N**	0,002 4 to < 0,002 6
O	5,0 to < 5,5	**O**	0,002 6 to < 0,002 8
P	5,5 to < 6.0	**P**	0,002 8 to < 0,003 0
R	6,0 to < 6,5	**Q**	0,003 0 to < 0,003 5
S	6,5 to < 7,0	**R**	0,003 5 to < 0,004 0
T	7,0 to < 8.0	**S**	0,004 0 to < 0,006 0
U	8,0 to < 9,0	**T**	0,006 0 to < 0,008 0
V	9,0 to < 10,0	**U**	0,008 0 to < 0,010
W	10,0 to < 11,0	**W**	0,010 to < 0,030
X	11,0 to < 12,0	**X**	0,030 to < 0,10
Y	12,0 to < 14,0	**Y**	⩽ 0,1
Z	⩾ 14,0		

8.1.2 Dielectric strength (electric strength)

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

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

Table 3 — Codes and ranges for dielectric strength

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

8.1.3 Surface resistivity

Determine this property in accordance with IEC 60093.

Codes and ranges are listed in Table 4.

Table 4 — Codes and ranges for surface resistivity

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

8.2 Mechanical properties

8.2.1 Impact properties

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

Table 5 — Codes and ranges for impact properties

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

8.2.2 Tensile properties

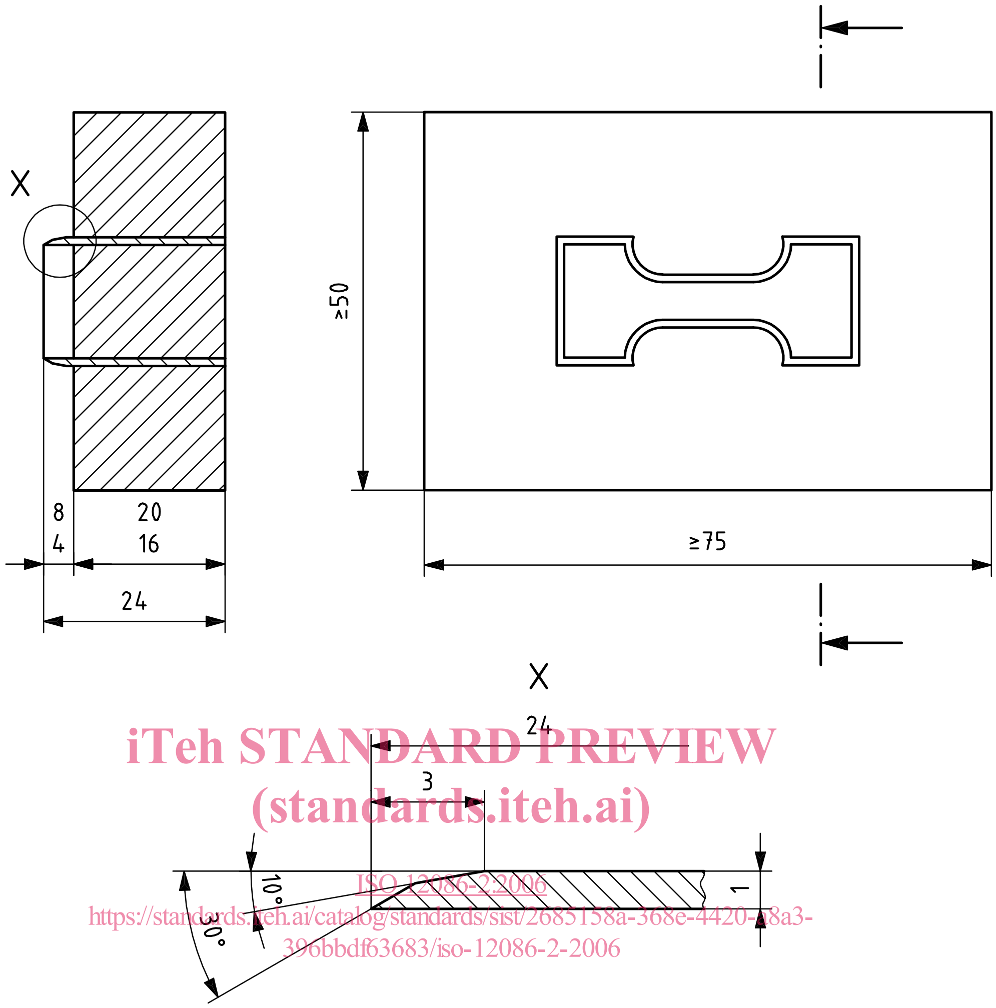
8.2.2.1 Fluoropolymers for which tensile modulus is not to be determined

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

8.2.2.1.2 For test specimens other than the skived film referred to in 8.2.2.1.1 (equal to or less than 0,125 mm in thickness), prepare five specimens using the microtensile die described in Figure 1. The die shall be of the steel-rule type with a curvature of 5 mm ± 0,5 mm [1]. Determine the tensile properties in accordance with the procedures described in ISO 527-1 except that the specimens used shall be as detailed above, the initial jaw separation shall be 22,0 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.

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

Dimensions in millimetres



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

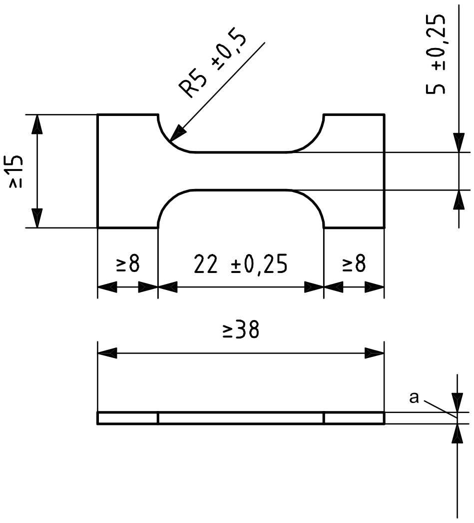
Die to be sharpened on outside of knife edge only (as shown in figure).

Rockwell C hardness of die: 45 to 50.

a) Steel-rule die

Figure 1 (*continued on next page*)

Dimensions in millimetres



b) Micro-tensile specimen

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

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

Calculate the percentage elongation using the following equation:

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

where

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

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

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

8.2.2.2 Fluoropolymers for which tensile modulus is to be determined

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

8.2.3 Modulus in flexure

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

8.3 Thermal-transition temperatures

8.3.1 Deformation temperature under load

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

8.3.2 Glass-transition temperature(s)

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

8.3.3 Melting-peak temperature

8.3.3.1 Test samples/specimens for melting-peak temperature determination may be powder as received, dried polymer isolated from a dispersion, or the required amount cut from a pellet or fabricated piece of the resin as sold or received. The test shall be determined on a 10 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. Melting-peak temperature characteristics are specific for fluoropolymers and help identify a particular material. The procedures of ASTM D 4591 or ISO 11357-3 supplemented by ASTM D 3418 are appropriate for this determination. Some fluoropolymers such as PTFE show different melting behaviour the first time a virgin powder is melted compared to the second and subsequent determinations that have lower melting-peak temperatures. Both the first and second melting points shall be measured. With PTFE, the second melting point usually is 327 °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, ISO 11357-3 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 can demonstrate that they are capable of measuring the melting-peak temperature and give results of equivalent significance.

8.4 Density

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

8.5 Flammability by oxygen index

Use the procedure in the appropriate part of ISO 4589.

8.6 Particle size and size distribution

8.6.1 General

The wet and dry-sieve procedures of 8.6.2 and 8.6.3 are widely used with PTFE and closely related materials. The resistance-variation test procedure in 8.6.4 (the Coulter principle) is often used with PVDF, PTFE filler resin, and fine-cut suspension powders. The light-scattering procedures in 8.6.5 are becoming more widely used with all the fluoropolymers. The use of automatic or other instruments that have been shown to provide equivalent results is an acceptable alternative to the detailed procedures given in this part of ISO 12086. ASTM F 660 (see the Bibliography) provides a standard practice for comparing particle size determined with different types of automatic particle counter.