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**Plastics — Polytetrafluoroethylene (PTFE)  
semi-finished products —**

**Part 2:**

Preparation of test specimens and  
determination of properties

*Plastiques — Semi-produits en polytétrafluoroéthylène (PTFE) —*

*Partie 2: Préparation des éprouvettes et détermination des propriétés*



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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.

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 13000-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

ISO 13000 consists of the following parts, under the general title *Plastics — Polytetrafluoroethylene (PTFE) semi-finished products*:

- *Part 1: Requirements and designation*
- *Part 2: Preparation of test specimens and determination of properties*

Annex A of this part of ISO 13000 is for information only.

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## Plastics – Polytetrafluoroethylene (PTFE) semi-finished products

### Part 2:

#### Preparation of test specimens and determination of properties

**WARNING – This part of ISO 13000 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 13000 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.**

#### 1. Scope

This part of ISO 13000 specifies the preparation of test specimens and gives the test methods for the requirements of semi-finished products of polytetrafluoroethylene (PTFE).

#### 2. Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 868:1985 *Plastics and ebonite — Determination of indentation hardness by means of a durometer (Shore hardness).*

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

- ISO 1923:1981 *Cellular plastics and rubbers — Determination of linear dimensions.*
- ISO 2039-1:1993 *Plastics — Determination of hardness — Part 1: Ball indentation method.*
- ISO 3611:1978 *Micrometer callipers for external measurement.*
- ISO 4599:1986 *Plastics — Determination of resistance to environmental stress cracking (ESC) — Bent strip method.*
- ISO 4600:1992 *Determination of environmental stress cracking (ESC) — Ball or pin impression method.*
- ISO 13000-1:1997 *Plastics — Polytetrafluoroethylene (PTFE) semi-finished products. Part 1: Requirements and designation.*
- IEC 243-1:1988 *Methods of test for electric strength of solid insulating materials — Part 1: Tests at power frequencies.*
- IEC 243-2:1990 *Method of test for dielectric strength of solid insulating materials — Part 2: Additional requirements for tests using direct voltage.*
- ASTM D621-64 (1988) *Test methods for Deformation of Plastics Under Load* (Withdrawn – see subclause 6.13 for details of availability).
- ASTM D1389-90 *Methods for dielectric proof-voltage testing of thin solid electrical insulating materials.*
- ASTM E94-93 *Guide for radiographic testing.*
- CIE Publication 15.2:1986, *Colorimetry.*

### 3. Definitions

The terminology given in ISO 472 and in ISO 13000-1 is applicable to this part of ISO 13000.

### 4. Sampling

Details of procedures for sampling semi-finished products depend to a large extent on the physical shape of the particular material. Whenever feasible, the materials shall be sampled in accordance with ISO 842. Adequate statistical sampling shall be considered an acceptable alternative.

### 5. Preparation of test specimens

The specimen used for testing shall be taken directly from or be machined from the semi-finished product without other treatment. Thus, conversion of a semi-finished product into a test specimen by any moulding procedure is not permitted. 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.

### 6. Testing of semi-finished PTFE products

#### 6.1 General

Properties required for specification shall be determined according to the International Standards as referenced in this part of ISO 13000 or the procedures in this part of ISO 13000. For the determination of density, tensile properties, hardness,

and electrical properties, condition the test specimens at  $23 \pm 2$  °C for a period of at least 4 hours prior to test. The other tests require no conditioning.

Informative annex A provides a list of other standards relating to testing semi-finished products of PTFE.

## 6.2 Linear dimensions

These shall be determined by the procedures provided in ISO 1923 for cellular plastics.

## 6.3 Tensile properties

### 6.3.1 Tensile specimens

#### 6.3.1.1 General

The appropriate type of test specimen shall be chosen from those given in 6.3.1.2 through 6.3.1.5. At least three test specimens shall be prepared from the sample and three specimens shall be tested.

NOTE—Test specimens prepared from the moulded basic shapes, rod, or tube and conforming to 6.3.1.2 are likely to give similar results to test specimens prepared from the same product but conforming to 6.3.1.3.

When testing extruded products, the test specimens shall be cut parallel to the axis of extrusion so that, as far as possible, each test specimen represents a different section along the axis and a different area of the cross section.

When testing skived tape, skived sheet, or skived film, the test specimens shall be cut perpendicular to the direction in which the pressure was applied when moulding the billet from which the product was skived. If this direction is not known, two sets of test specimens shall be prepared and tested, the test specimens in one set being cut at 90° to those in the other set and the mean results shall be calculated from the higher set of values (indicating, typically, the direction perpendicular to the axis of applied pressure). This procedure ensures a uniform way to report results from the testing. The micro tensile test specimen is used most commonly for PTFE products.

#### 6.3.1.2 Turned dumb-bell test specimens

The test specimens shall be turned so that their dimensions are in the ratios given in figure 1, with the shape shown in Figure 1. The value of the larger diameter (D) shall be  $7 \pm 0,5$  mm,  $10 \pm 0,5$  mm, or  $20 \pm 0,5$  mm.

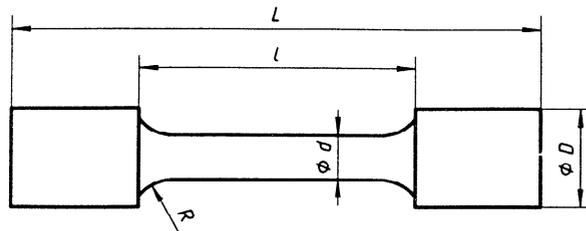


Table of ratios for turned dumb-bell specimens

$$d = 0,45 D \text{ to } 0,55 D$$

$$L = 5 D \text{ to } 6 D$$

$$l = 3 D \text{ to } 3,5 D$$

$$R = 0,2 D \text{ to } 0,3 D$$

**Figure 1 - Turned dumb-bell specimen for tensile testing**

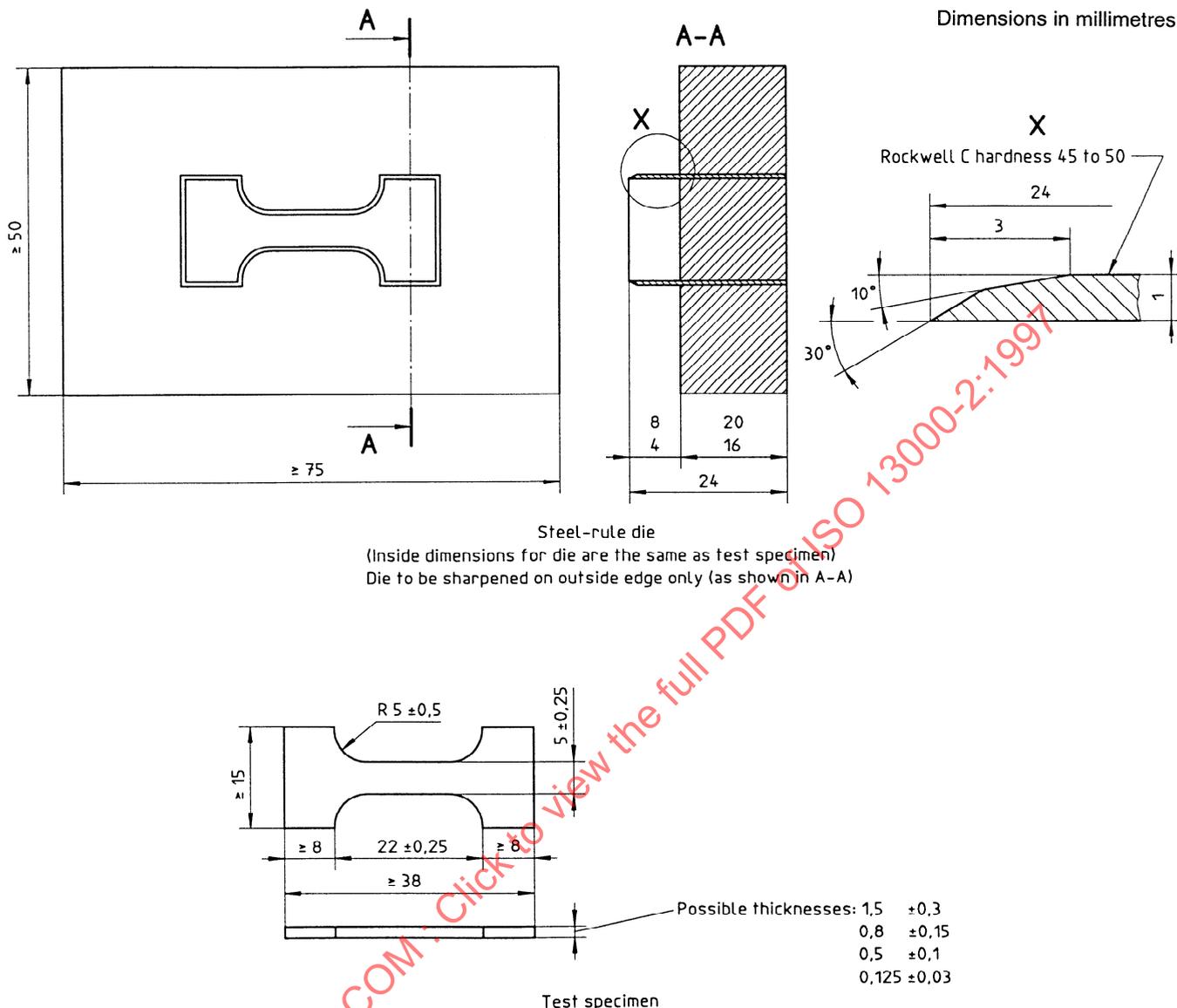
### 6.3.1.3 Micro tensile die (Small punched dumb-bell) test specimens

The sample shall be machined to produce a sheet or disc of thickness 1,5 to 2,5 mm, or turned to produce a rectangular sectioned ring which can be cut and flattened to give such a sheet.

Test specimens conforming to the shape shown in Figure 2 and the dimensions given in table 1 for the micro tensile die shall be punched from sheet or disc using a single stroke press and the appropriate knife-edged die. In any one test specimen, the thickness of the narrow parallel-sided portion shall nowhere deviate by more than two percent from the mean. The sheet shall be supported, while the test specimens are punched from it, on a slightly yielding material having a smooth surface (e. g. leather, rubber, or high quality cardboard) on a flat rigid base. The cutting edge of the die shall be sharp and free from notches or other visual defects. In the case of thin sheet or skived tape of thickness from 0,125 mm to 3,0 mm, the test specimens shall be punched from the material 'as received'.

Table 1 - Dimensions of dumb-bell test specimens

	Micro tensile die (Figure 2)	Large dumb-bells (Figure 3)
	mm	mm
A overall length, minimum	38	115
B width at ends, minimum	15	25 ± 1
C length of narrow parallel-sided portion	12 ± 0,5	33 ± 2
D width of narrow parallel-sided portion	5 ± 0,25	6 ± 0,4
E small radius	5 ± 0,5	14 ± 1
F large radius		25 ± 2

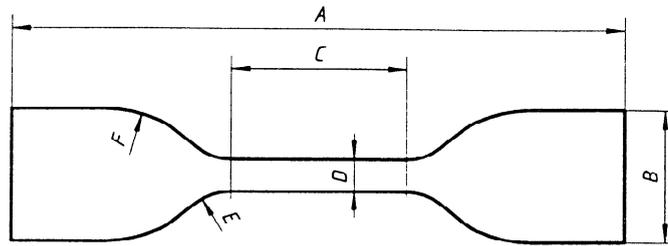


**Figure 2 – Micro tensile die and punched micro tensile (type A) specimen for tensile testing**

**6.3.1.4 Test specimens prepared from thin or narrow tape and small-diameter tubing less than 7,0 mm I.D.**

When testing tape of less than 0,1 mm in thickness that has a width  $\geq 25$  mm, the test specimens shall be parallel-sided strips 25 mm wide and of an appropriate length to suit the testing machine grips. Alternatively, test specimens conforming to the shape shown in figure 3 and the dimensions given in table 1 for large dumb-bells shall be punched from the tape, by the method described in 6.3.1.3. In any one test specimen, the thickness of the narrow parallel portion shall nowhere deviate by more than 2 percent from the mean.

In the case of tape of less than 25 mm in width, the full width shall be tested. Tubing less than 7 mm OD shall be tested as manufactured, unmachined, without slitting and flattening the slit tube.



A overall length, minimum	115 mm
B width at ends, minimum	$25 \pm 1$ mm
C length of narrow, parallel-sided portion	$33 \pm 2$ mm
D width of narrow parallel-side portion	$6 \pm 0,4$ mm
E small radius	$14 \pm 1$ mm
F large radius	$25 \pm 2$ mm

**Figure 3 – Punched large dumb-bell (type B) specimen for tensile testing**

#### 6.3.1.5 Test specimens prepared from small diameter rod

When testing rod of not more than 7 mm in diameter, the test specimens shall consist of 100 mm lengths cut from the rod either 'as received', or with a reduction in diameter of up to 15 percent over a test length of 25 to 30 mm. This reduction in diameter will facilitate gripping the specimen in the testing machine without breakage occurring within the grips.

#### 6.3.1.6 Marking of test specimens

If measurements of elongation are made visually, the gauge length shall be marked on the test specimens by means of two reference marks. The elongation is followed during the testing by holding a ruler by one of the marks and noting the amount of elongation at the break point.

Before marking test specimens of the types specified in 6.3.1.2, 6.3.1.3, or 6.3.1.5, any machining or punch marks may be removed by sanding lightly with fine abrasive paper.

Mark each test specimen for visual measurement of elongation with two reference marks 10 mm to 25 mm apart (15 mm to 20 mm for a small dumb-bell test specimen) and approximately equidistant from the midpoint, taking care to avoid damaging the test specimen. Ensure that the marking medium has no detrimental effect on the material being tested and that the marks are as narrow as possible. Do not scratch, punch, or impress the lines upon the test specimen.

#### 6.3.2 Procedure

Determine the tensile properties in accordance with the procedures described in the appropriate part of ISO 527 except that the specimens used shall be as detailed above.

The initial jaw separation for types 6.3.1.4 and 6.3.1.5 shall be at least twice the gauge length of 10 mm to 25 mm, and the speed of testing shall be  $50 \pm 5$  mm/min.

Clamp the specimens with an essentially equal length in each jaw. Measure the elongation at break from the recorder chart by drawing a perpendicular line from the break point to the time axis. Alternatively, the elongation at break may be measured by visual means described in 6.3.1.6. Measure the distance along the time axis from the foot of this perpendicular line to the beginning of the load-time curve. Optionally, an extensometer may be used to determine the elongation.

Calculate the elongation at break for each specimen by dividing the maximum elongation of the gauge length recorded during the test by the original gauge length. If the cross-head rate and the chart rate are not the same, provide the correct magnification ratio for calculations involving the time axis.

Calculate the tensile strength of each test specimen by dividing the maximum force (in newtons) recorded during the test by the original mean cross sectional area (in square millimeters) of the test specimens. Report the tensile strength in megapascals.

#### 6.4 Density

Cut two specimens from the semi-finished product and test in accordance with ISO 1183. If Method D is used, the solution in the tube should have a linear gradient as specified in the table appropriate for the fluoropolymer being tested. It is acceptable to use the newly available equipment that uses special balances with a specific programme that gives the density value (taking into account the density of water, its temperature, and the temperature of the test specimen).

NOTE—Problems caused by the effect of temperature on the density of PTFE can be minimized when the measurement is made using the immersion procedure (Method A) if a sensitive thermometer (e. g., one reading  $\pm 0,1$  °C) is used in the liquid, the temperature is adjusted to be at least 22 °C but not over 25 °C, and the density value is corrected to 23 °C by the relationship:

$$D_c = D_m + (T_m - 23) \times 0,00052$$

where  $D_c$  is the density corrected to 23 °C,  $D_m$  is the measured density, and  $T_m$  is the temperature at which the measurement was made.

#### 6.5 Loss of mass at 300 °C

Weigh, to a precision of 1 mg, 10 g of the semi-finished product. Heat the weighed test specimen for 6 h in an air oven at a temperature of  $300 \pm 5$  °C. Cool the test specimen in a desiccator and reweigh.

Report any loss in mass as a percentage of the original mass. Examine the test specimen after heating and report any sign of melting.

#### 6.6 Dimensional stability - General method

##### 6.6.1 Apparatus

6.6.1.1 **Micrometer**, complying with ISO 3611 except that the measuring faces shall be hemispherical.

6.6.1.2 **Oven**, capable of being maintained at  $285 \pm 5$  °C.

### 6.6.2 Test specimen

Machine a test specimen from the sample which is as long as possible within the range 25 mm to 100 mm and which has a thickness and width or a diameter, as appropriate, within the range 5 mm to 25 mm. The faces at each end of the test specimen shall be parallel to within 0,25 mm. Whenever possible, the test specimen shall be cut so the length is parallel to the direction of the moulding pressure or to the axis of the extrusion.

If it is not possible to prepare a test specimen of at least 25 mm length, the test may be carried out using a shorter test specimen but a commensurate adjustment shall apply to the requirements for precision of measurement.

For rod less than 5 mm in diameter, cut a test specimen of 50 mm to 100 mm length and machine the faces at each end so that they are flat and at right angles to the axis.

### 6.6.3 Procedure

Condition the test specimen at  $23 \pm 2$  °C for at least 4 hr and while it is maintained at that temperature, using a micrometer, measure its length to a precision of  $\pm 0,025$  mm.

Place the test specimen in an oven, maintained at  $285 \pm 5$  °C, for a period of at least 4 h. At the end of this period allow the test specimen to cool at a rate not exceeding 30 °C/h. When cooled, recondition the test specimen at  $23 \pm 2$  °C for at least 4 h and measure its length at several points to a precision of  $\pm 0,025$  mm.

NOTE—If the test specimen is thin in proportion to its length, e. g., rod of less than 5 mm diameter, it is probable that some degree of bending will occur both before and during the stress-relieving process. If bending occurs, the test specimen may be straightened while carrying out the measurement.

### 6.6.4 Expression of results

Report the maximum change in length as a percentage of the original length.

## 6.7 Dimensional stability – Special method for determination of dimensional and geometric stabilities of thick-walled tube

### 6.7.1 Apparatus

6.7.1.1 **Micrometer**, complying with ISO 3611.

6.7.1.2 **Oven**, capable of being maintained at  $285 \pm 5$  °C.

### 6.7.2 Test specimen

Machine the outside diameter of an approximately 30 mm length of the tube, taking care to remove only the minimum material necessary to obtain complete "clean up" and produce a circular cross section accurate to  $\pm 0,025$  mm.

Cut and "face off" the specimen of tube to produce a cylinder at least 25 mm long and with end faces at right angles to the longitudinal axis and parallel to each other to within 0,025 mm.

### 6.7.3 Procedure

Condition the test specimen at  $23 \pm 2$  °C for at least four hours and then measure the length and outside diameter, using the micrometer, to a precision of  $\pm 0,025$  mm whilst it is maintained at that temperature.

Stand the test specimen on an end-face on a smooth plate in the oven, maintained at  $285 \pm 5$  °C, and allow it to remain in the oven for at least thirty minutes plus one hour for each 6 mm of wall thickness in excess of 6 mm, e.g.,

for a wall thickness less than 6 mm.....	30 min
6 mm up to but not including 12 mm.....	90 min
12 mm up to but not including 18 mm.....	150 min

At the end of the appropriate period, allow the oven to cool at a rate not greater than 30 °C/h.

When the test specimen is cool, recondition it at 23 °C for at least four hours and measure its length and outside diameter at several points to a precision of  $\pm 0,025$  mm.

### 6.7.4 Expression of results

Report the maximum change in length and diameter as a percentage of the original values.

## 6.8 Electrical properties

### 6.8.1 Dielectric strength (electric strength)

This characteristic shall be determined according to the procedures of IEC 243-1 or IEC 243-2, as appropriate. Run the test in oil for test specimens thicker than 0,3 mm and use the electrode configuration shown in Figure a of IEC 243-1.

### 6.8.2 Electrical flaws

This characteristic shall be determined in accordance with ASTM D 1389.

### 6.9 Hardness

This characteristic shall be determined by the ball indentation method of ISO 2039-1, using a test load  $F_m = 132$  N. Alternatively, Shore D hardness may be determined as described in ISO 868.

### 6.10 Colour

Typically, PTFE that does not contain a colourant is white to transparent. Visual perception of whiteness is usually adequate to appraise this characteristic of semi-finished products. If a quantitative value is required, the test procedure described below shall be used.

Specimens of PTFE for whiteness measurement shall be taken from the semi-finished product without any further treatment. Preferably a specimen at least 5 cm square should be taken, if possible, considering the particular semi-finished product. The whiteness shall be determined using either a reflectance spectrometer or a tristimulus (filter) colorimeter. If hemispherical (integrating sphere) geometry is used, the specular component shall be excluded. Test specimens shall be backed during measurement by a standard opaque backing material, preferably black.

Measurements shall be made according to CIE 15.2 and the results shall be given as CIE 1931 or CIE 1964 tristimulus values and chromaticity coordinates.

#### 6.11 Radiographic examination

A non-destructive test for radiographic examination is to be used. ASTM E94 is appropriate.

#### 6.12 Resistance to environmental stress cracking (ESC)

General tests for ESC of plastics are described in ISO 4599 and ISO 4600.

#### 6.13 Deformation under load

This property shall be determined in accordance with ASTM D621-64 (1988) at a temperature of 23 °C and a load of 15 N for 24 h.

NOTE—ASTM D621-64 (1988) has been withdrawn by ASTM, but copies are available from Information Handling Services, Global Engineering Documents, 15 Inverness Way East, Englewood, CO 80112, USA.

**Annex A**  
(informative)

**Other standards relating to testing semi-finished products of PTFE**

In addition to the test methods provided in this part of ISO 13000 for use with the requirements of ISO 13000-1, there are other standard methods available. These methods could have use in international commerce for special applications. A list of some of the important standards is included here for information. This list is a supplement to informative annex A in ISO 13000-1 where many test methods are included in the list of specifications related to semi-finished products of PTFE.

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

ISO 12086-2:1995 *Plastics — Fluoropolymer dispersions, and moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties.*

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.*

IEC 674-2:1988 *Specification for plastic films for electrical purposes — Part 2: Methods of test.*

IEC 684-1:1980 *Specification for flexible insulating sleeving — Part 1: Definitions and general requirements.*

IEC 684-2:1984 *Specification for flexible insulating sleeving — Part 2: Methods of test.*

IEC 684-3-240:1991 *Specification for flexible insulating sleeving — Part 3: Specification requirements for individual types of sleeving — Sheets 240 to 243: Heat-shrinkable PTFE sleeving*

Sheet 240: *Heat shrinkable PTFE sleeving, low shrink ratio, thick wall*

Sheet 241: *Heat shrinkable PTFE sleeving, low shrink ratio, intermediate wall*

Sheet 242: *Heat shrinkable PTFE sleeving, low shrink ratio, thin wall*

Sheet 243: *Heat shrinkable PTFE sleeving, high shrink ratio.*

ASTM D1675-95 *Method of Testing Polytetrafluoroethylene Tubing.*

ASTM D2902-95 *Specification for Fluoropolymer Resin Heat-Shrinkable Tubing for Electrical Insulation.*

ASTM D3294-91a *Specification for PTFE Resin Molded Sheet and Molded Basic Shapes.*

ASTM D3295-96 *Specification for PTFE Tubing.*

ASTM D3308-91a *Specification for PTFE Resin Skived Tape.*

ASTM D3369-96 *Specification for Polytetrafluoroethylene (PTFE) Resin Cast Film.*

ASTM D3418-82 (1988) *Test method for transition temperatures of polymers by thermal analysis.*

ASTM D4591-93a *Method for Determining Temperatures and Heats of Transitions of Fluoropolymers by Differential Scanning Calorimetry.*

ASTM D4969-91a *Specification for PTFE Coated Glass Fabric.*

BS 6564:Part 1:Section 1.1:1989 *Polytetrafluoroethylene (PTFE) materials and products — Part 1: Polytetrafluoroethylene powders for moulding and extrusion — Section 1.1: Specification.*

BS 6564:Part 1:Section 1.2:1989 *Polytetrafluoroethylene (PTFE) materials and products — Part 1: Polytetrafluoroethylene powders for moulding and extrusion — Section 1.2: Method of specifying.*

BS 6564:Part 2:1991 *Polytetrafluoroethylene (PTFE) materials and products — Part 2: Specification for fabricated unfilled polytetrafluoroethylene products.*

BS 6564:Part 3:1989 *Polytetrafluoroethylene (PTFE) materials and products — Part 3: Specification for fabricated glass-fibre filled polytetrafluoroethylene products.*

DIN 16782-1:1991 *Kunststoff-Formmassen; Polytetrafluorethylen (PTFE)-Formmassen; Einteilung und Bezeichnung.*

DIN 16782-2:1991 *Kunststoff-Formmassen; Polytetrafluorethylen (PTFE)-Formmassen; Herstellung von Probekörpern und Bestimmung von Eigenschaften.*

The JIS standards listed below are the Japanese issue.

JIS K 6891 1995 *Testing methods for polytetrafluoroethylene molding powder.*

JIS K 6892 1995 *Testing methods for polytetrafluoroethylene powder for paste extrusion.*

JIS K 6893 1995 *Testing methods for polytetrafluoroethylene aqueous dispersion.*

JIS K 6896 1995 *Polytetrafluoroethylene powder for molding and extrusion materials.*

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