INTERNATIONAL STANDARD

ISO 13000-2

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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

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

This second edition cancels and replaces the first edition (ISO 13000-2:1997), which has been technically revised.

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*

Plastics — Polytetrafluoroethylene (PTFE) semi-finished products —

Part 2:
Preparation of test specimens and determination of properties

WARNING — 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.

1 Scope

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

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 472, *Plastics — Vocabulary*

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 868, *Plastics and ebonite — Determination of indentation hardness by means of a durometer (Shore hardness)*

ISO 1183 (all parts), *Plastics — Methods for determining the density of non-cellular plastics*

ISO 1923, *Cellular plastics and rubbers — Determination of linear dimensions*

ISO 2039-1, *Plastics — Determination of hardness — Part 1: Ball indentation method*

ISO 3611, *Micrometer callipers for external measurement*

ISO 4599, *Plastics — Determination of resistance to environmental stress cracking (ESC) — Bent strip method*

ISO 4600, *Plastics — Determination of environmental stress cracking (ESC) — Ball or pin impression method*

ISO 13000-1, *Plastics — Polytetrafluoroethylene (PTFE) semi-finished products — Part 1: Requirements and designation*

IEC 60243-1:1998, *Electrical strength of insulating materials — Part 1: Tests at power frequencies*

IEC 60243-2, *Electric strength of insulating materials — Part 2: Additional requirements for tests using direct voltage*

ASTM D 621-64, *Test methods for Deformation of Plastics Under Load* (Withdrawn — see Subclause 6.13 for details of availability)

ASTM D 1389, *Standard Test Method for Proof-Voltage Testing of Thin Solid Electrical Insulating Materials*

ASTM E 94, *Standard Guide for Radiographic Examination*

CIE Publication No. 15, *Colorimetry*

3 Terms and definitions

For the purposes of this part of ISO 13000, the terms and definitions given in ISO 472 and ISO 13000-1 apply.

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. Sampling shall be statistically adequate to satisfy the requirements of the test method concerned.

5 Preparation of test specimens

The specimens used for testing shall be taken directly from or shall 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 purposes shall be determined in accordance with the International Standards referenced in this part of ISO 13000 or the procedures given in this part of ISO 13000. For the determination of density, tensile properties, hardness and electrical properties, condition the test specimens at 23 °C ± 2 °C for a period of at least 4 h prior to test. The other tests require no conditioning.

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

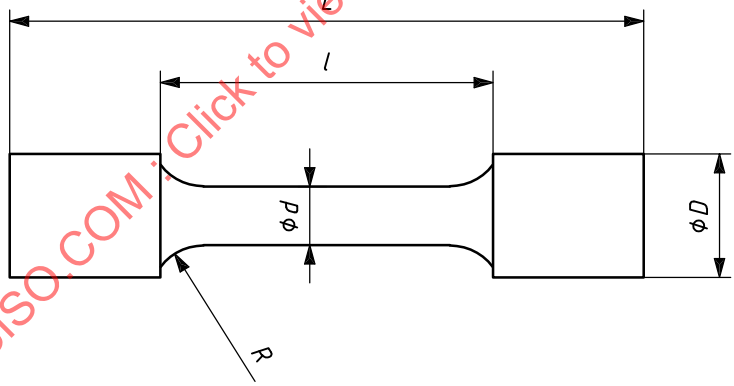
The appropriate type of test specimen shall be chosen from those given in 6.3.1.2 to 6.3.1.5. At least three test specimens shall be prepared from the sample and three specimens shall be tested.

NOTE Test specimens prepared from moulded basic shapes, rods or tubes conforming to 6.3.1.2 are likely to give results similar to those obtained with 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 result 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 of reporting test results. The micro-tensile test specimen (see 6.3.1.3) 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, and so that the shape is as shown in Figure 1. The value of the larger diameter (D) shall be 7 mm ± 0,5 mm, 10 mm ± 0,5 mm or 20 mm ± 0,5 mm.



Ratios of dimensions of turned dumb-bell specimens:

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

$L = 5D$ to $6D$

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

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

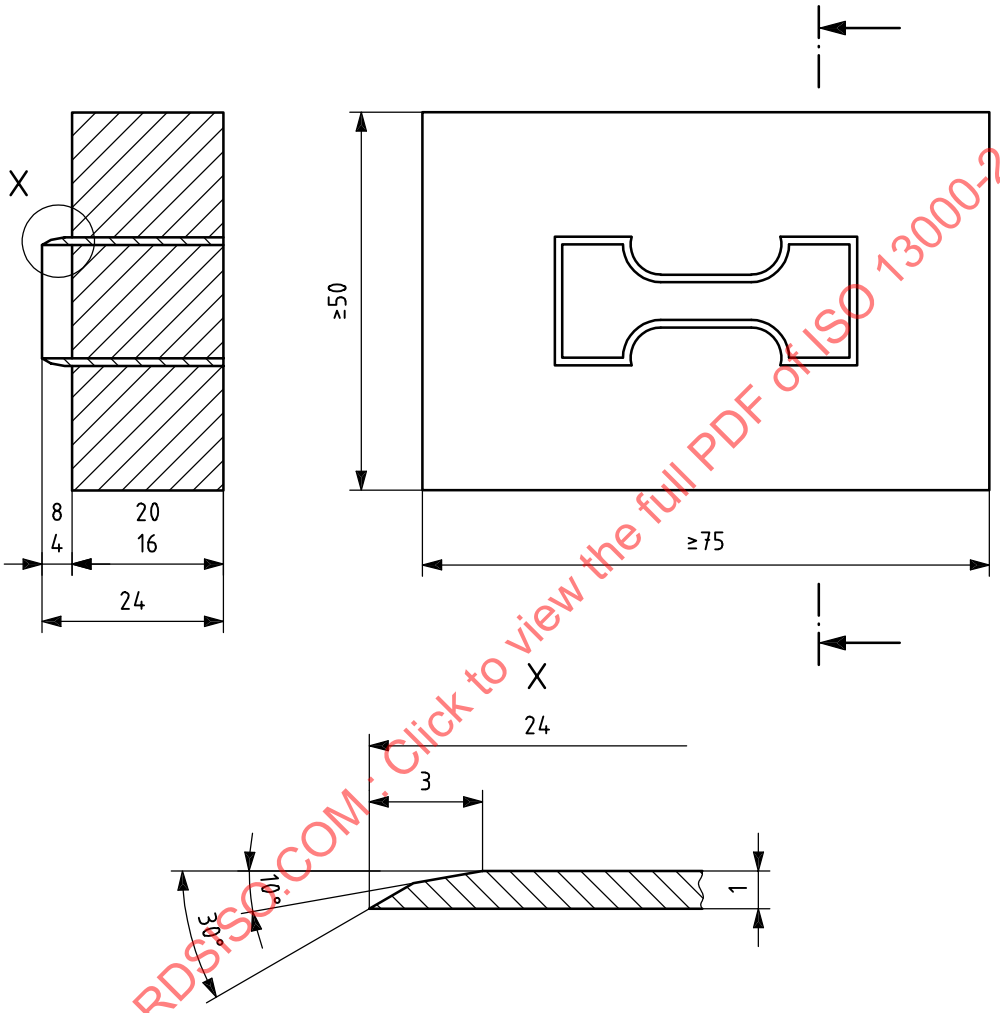
Figure 1 — Turned dumb-bell specimen

6.3.1.3 Micro-tensile (small punched dumb-bell) test specimens

The sample shall be machined to produce a sheet or disc of thickness 1,5 mm to 2,5 mm, or turned to produce a rectangular-section 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 micro-tensile specimens shall be punched from a 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 2 %

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”.

Dimensions in millimetres

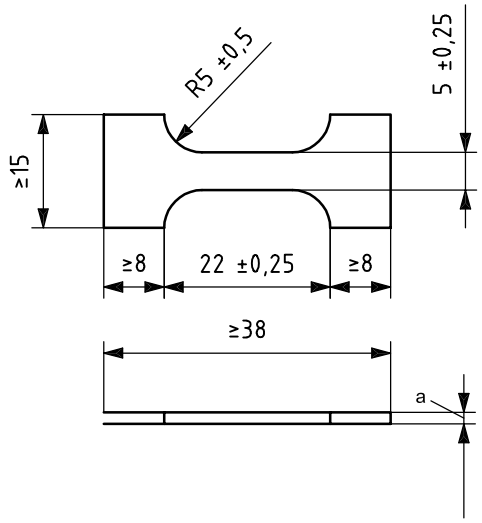


Inside dimensions of die are same as those of test specimen.

Rockwell C hardness of die: 45 to 50.

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

a) Steel-rule die



b) Micro-tensile specimen

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

Figure 2 — Knife-edged die for micro-tensile (type A) specimens, and punched-out specimen

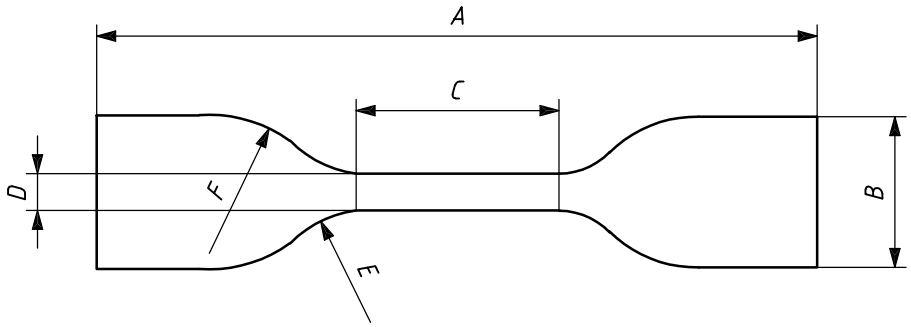
Table 1 — Dimensions of dumb-bell test specimens

		Micro-tensile specimen (see Figure 2) mm	Large dumb-bell (see Figure 3) 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

6.3.1.4 Test specimens prepared from thin or narrow tape and from small-diameter tubing of less than 7,0 mm ID

When testing tape of less than 0,1 mm in thickness that has a width ⩾ 25 mm, the test specimens shall be parallel-sided strips 25 mm wide and of an appropriate length to suit the test 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-sided portion shall nowhere deviate by more than 2 % from the mean.

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



A	Overall length, minimum:	115 mm
B	Width at ends, minimum:	25 mm ± 1 mm
C	Length of narrow, parallel-sided portion:	33 mm ± 2 mm
D	Width of narrow, parallel-sided portion:	6 mm ± 0,4 mm
E	Small radius:	14 mm ± 1 mm
F	Large radius:	25 mm ± 2 mm

Figure 3 — Punched large dumb-bell (type B) specimen

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 % over a test length of 25 mm to 30 mm. This reduction in diameter will facilitate gripping the specimen in the test machine without breakage occurring within the grips.

6.3.1.6 Marking of test specimens

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 with two reference marks 10 mm to 25 mm apart (15 mm to 20 mm for small dumb-bell test specimens) and approximately equidistant from the midpoint, taking care to avoid damaging the 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 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 the types described in 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 mm/min ± 5 mm/min.

Clamp the specimens with an essentially equal length in each jaw. If using a chart recorder, measure the elongation at break from the chart by drawing a perpendicular line from the break point to the time axis and measuring the distance along the time axis from the foot of this perpendicular line to the beginning of the load-time curve. Alternatively, the elongation at break may be measured by visual means, holding a ruler against the reference marks and noting the elongation at the break point. 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 millimetres) of the test specimens. Report the tensile strength in megapascals.

6.4 Density

Cut two specimens from the semi-finished product and determine the density in accordance with the appropriate part of ISO 1183. 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). It is acceptable to use newly available equipment that uses special balances with a specific programme that gives the value of the density directly (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 (ISO 1183-1:2004, method A) if a sensitive thermometer (e.g. one reading to ± 0,1 °C) is used in the liquid, the temperature is adjusted to at least 22 °C but not over 25 °C, and the density is corrected to 23 °C using the relationship:

$$\rho_c = \rho_m + (T_m - 23) \times 0{,}000\ 52$$

where

ρ_c is the density corrected to 23 °C;

ρ_m is the measured density;

T_m is the temperature at which the measurement was made.

6.5 Loss in mass at 300 °C

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

Report any loss in mass as a percentage of the original mass. Examine the test sample 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 °C ± 5 °C.

6.6.2 Test specimen

From the sample, machine a test specimen which is as long as possible within the range 25 mm to 100 mm and which has a thickness and width or 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 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 rods less than 5 mm in diameter, cut a test specimen 50 mm to 100 mm in 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 °C ± 2 °C for at least 4 h and, while it is maintained at that temperature, measure its length to an accuracy of ± 0,025 mm using a micrometer.

Place the test specimen in an oven, maintained at 285 °C ± 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 °C ± 2 °C for at least 4 h and measure its length at several points to an accuracy of ± 0,025 mm.

NOTE If the test specimen is thin in proportion to its length, e.g. if it is a rod less than 5 mm in 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 the determination of the dimensional and the geometrical stability of thick-walled tubes

6.7.1 Apparatus

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

6.7.1.2 **Oven**, capable of being maintained at 285 °C ± 5 °C.

6.7.2 Test specimen

Machine the outside surface of an approximately 30 mm length of the tube, taking care to remove only the minimum material necessary to "clean up" the specimen and produce a circular cross-section accurate to ± 0,025 mm.

Cut and "square off" the ends of the specimen to produce a tube at least 25 mm long 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 °C ± 2 °C for at least 4 h and then, using the micrometer, measure the length and outside diameter to an accuracy of ± 0,025 mm with the specimen maintained at that temperature.

Stand the test specimen on an end-face on a smooth plate in the oven, maintained at 285 °C ± 5 °C, and allow it to remain in the oven for at least 30 min plus 1 h 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 4 h and measure its length and outside diameter at several points to a accuracy of ± 0,025 mm.