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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CONTENTS

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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 ± 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 ± 0,5 mm, 10 ± 0,5 mm, or 20 ± 0,5 mm.

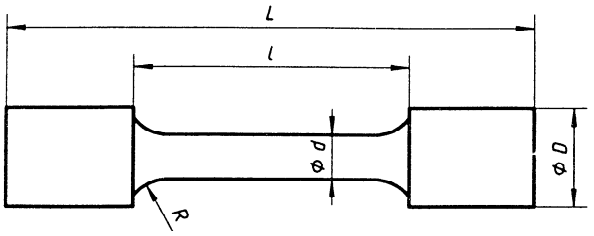


Table of ratios for turned dumb-bell specimens

d = 0,45 D to 0,55 D
L = 5 D to 6 D
l = 3 D to 3,5 D
R = 0,2 D 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

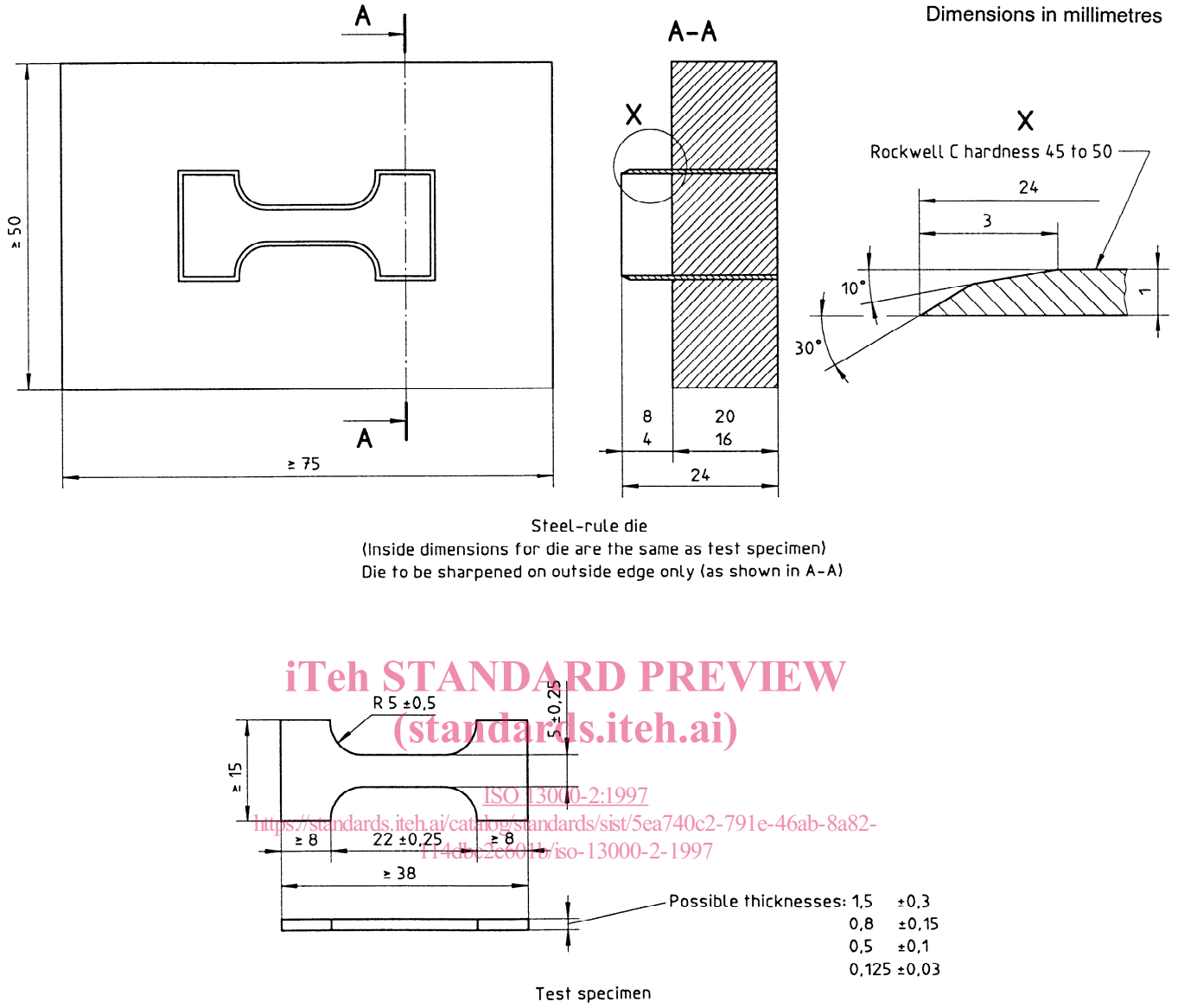
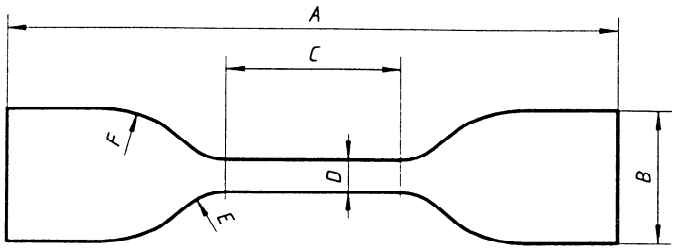


Figure 2 – Micro tensile die and punched micro tensile (type A) specimen for tensile testing

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 ≥ 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 ± 1 mm
C	length of narrow, parallel-sided portion	33 ± 2 mm
D	width of narrow parallel-side portion	6 ± 0,4 mm
E	small radius	14 ± 1 mm
F	large radius	25 ± 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 ± 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 ± 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:

$$Dc = Dm + (Tm - 23) \times 0,00052$$

where Dc is the density corrected to 23 °C, Dm is the measured density, and Tm 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 ± 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.