INTERNATIONAL STANDARD

ISO 20568-2

First edition
2017-05

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 20568-2:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see http://www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This first edition of ISO 20568-2 cancels and replaces ISO 12086-2:2006, which has been technically revised.

A list of all parts in the ISO 20568 series can be found on the ISO website.

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 7.1.1.4 and 7.1.3.1 point out specific hazards.

1 Scope

This document describes the preparation of test specimens and provides test methods to define characteristics of thermoplastic fluoropolymer resins. Results from the testing can be used as the basis for designation, material specifications or both.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

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

ISO 1133-1:2011, *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 1: Standard method*

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

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

ASTM D1430, *Standard Classification System for Polychlorotrifluoroethylene (PCTFE) Plastics*

ASTM D4591, *Standard Test Method for Determining Temperatures and Heats of Transitions of Fluoropolymers by Differential Scanning Calorimetry*

ASTM D4894, *Standard Specification for Polytetrafluoroethylene (PTFE) Granular Molding and Ram Extrusion Materials*

ASTM D4895, *Standard Specification for Polytetrafluoroethylene (PTFE) Resin Produced From Dispersion*

ASTM E11, *Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 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.

5 Conditioning and test conditions

For determinations of specific gravity, 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.

Conduct tests at a laboratory temperature of 23 °C ± 2 °C for determining specific gravity.

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.

6 Determination of properties

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 document.

7 Testing of PTFE

7.1 Testing of polytetrafluoroethylene (PTFE) granular moulding and ram extrusion materials, and for PTFE resin produced from coagulation of dispersion

7.1.1 Standard specific gravity (SSG)

7.1.1.1 Use the PTFE powder as received.

7.1.1.2 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 compression. Place flat aluminium foil discs, normally 0,13 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 performing 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 D4895 provides additional details.

7.1.1.3 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) until the desired pressure is attained. The pressure shall be 34,5 MPa for PTFE granular moulding and ram extrusion materials, and 14 MPa for PTFE resin produced from coagulation of dispersion. 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 As a guide, increasing the pressure at a rate of 3,5 MPa/min is suggested until the desired maximum is attained.

7.1.1.4 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 1.

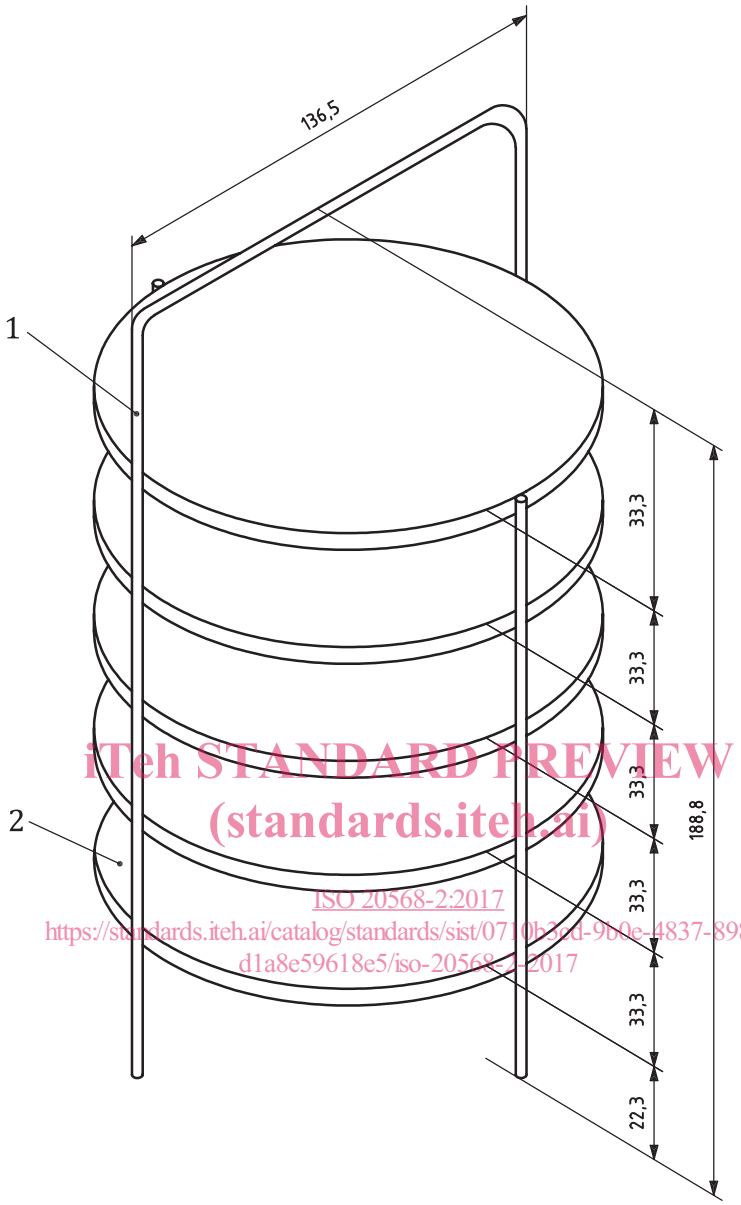
WARNING — PTFE resin can evolve small quantities of gaseous products when heated above 260 °C. Some of these gases are harmful. Consequently, exhaust ventilation must be used whenever the resins are heated above this temperature.

Table 1 — Sintering conditions for preparing SSG specimens

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

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 1. The rack, as shown in Figure 1, 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.) Consult ASTM D4894 or ASTM D4895 for additional details.

Dimensions in millimetres



Key

1 support rods, diam. 6,35 mm (four required)

2 shelves, made of type 3003-H14 20 GA aluminium (five required)

NOTE Aluminium plates tack-welded to rods.

Figure 1 — Rack for sintering oven

7.1.2 Bulk density

Bulk density gives an indication of how a resin can 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 7.1.2.2 shall be used to measure this property. This procedure can also be found in ASTM D4894 and ASTM D4895.

7.1.2.1 Apparatus

7.1.2.1.1 Funnel, as shown in Figure 2.

7.1.2.1.2 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[1)].

7.1.2.1.3 Controller[2)].

7.1.2.1.4 Volumetric cup and cup stand, as shown in Figure 3. 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 and with the top surface of the stand. All sharp external corners shall be removed from the cup stand.

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.

7.1.2.1.5 Levelling device, as shown in Figure 4, affixed permanently on 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.

7.1.2.1.6 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.

7.1.2.1.7 Balance, having an extended beam, and with a capacity of 500 g and a sensitivity of 0,1 g or equivalent.

7.1.2.2 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.

7.1.2.3 Expression of results

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

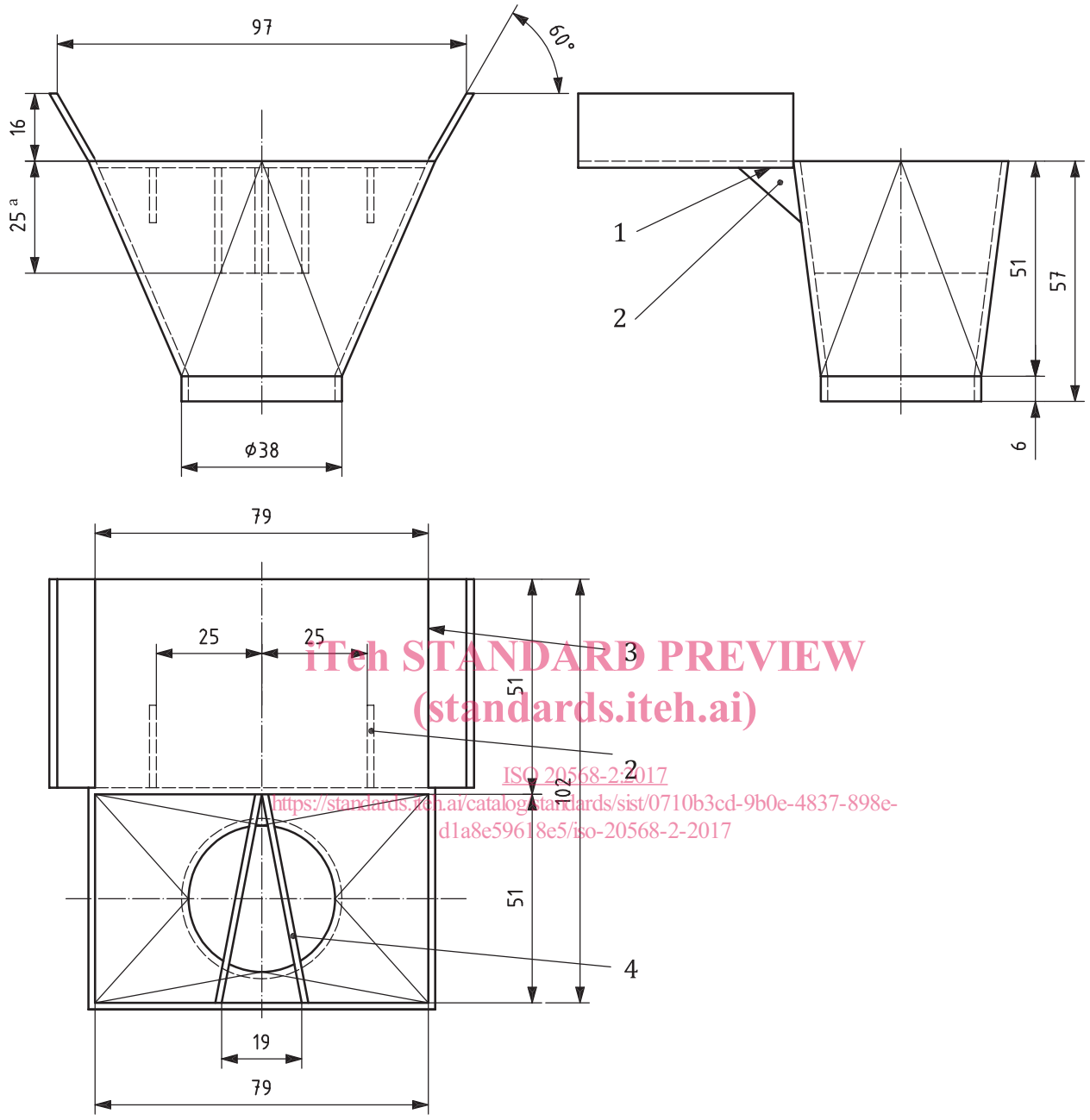
Mass of resin in cup × 4 = Bulk density

---

1) A laboratory-sized vibrating feeder has been found satisfactory for this purpose. Originally used was a "Vibra-Flow" feeder, Type F-T01A, with trough, which may still be available. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

2) A suitable controller for the feeder should be used. Originally used was a "Syntron" controller, Type CSCRBI, which may still be available. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

Dimensions in millimetres



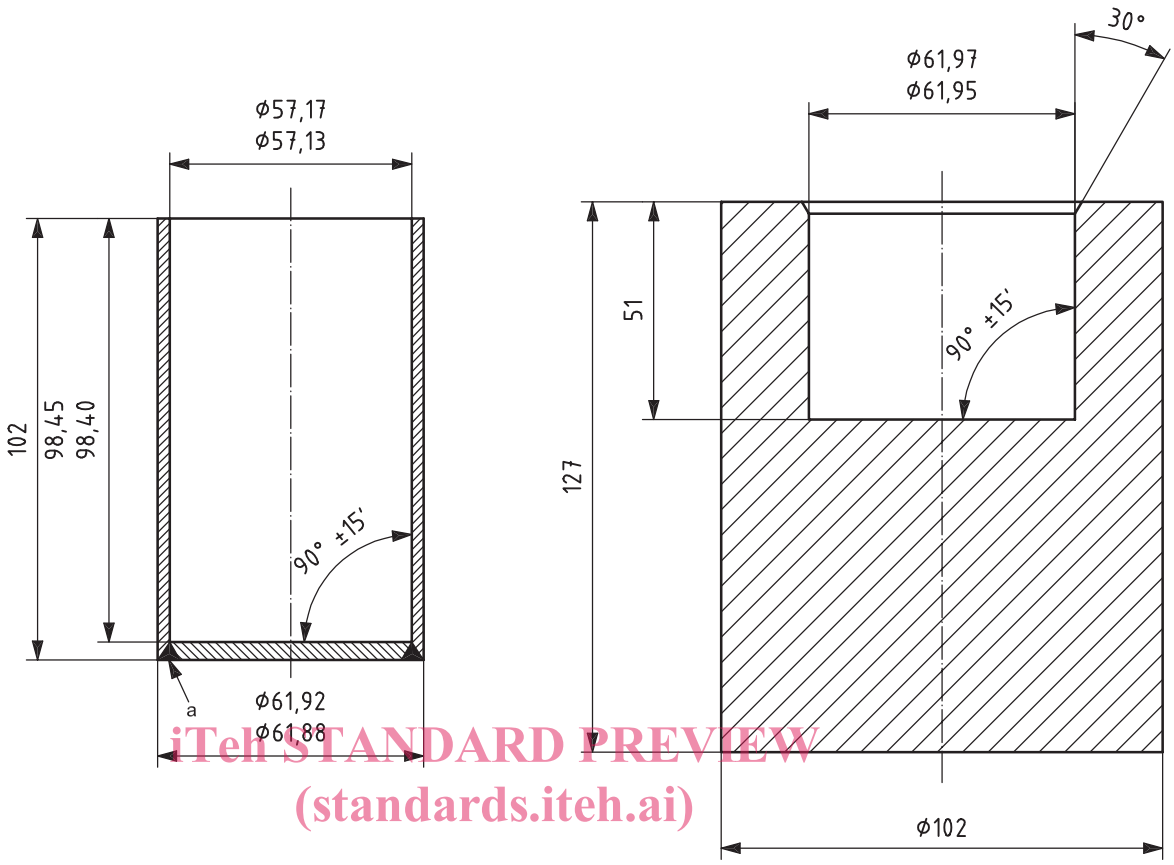
Key

1 weld
2 two support gussets, approx. 13 mm × 13 mm × 1,6 mm thick, located in positions shown
3 bend
4 straightening vanes (locate two partitions as shown)
a Depth of partitions.

NOTE Funnel material: type 304 stainless steel, 16 gauge (1,6 mm thickness).

Figure 2 — Details of funnel used for determination of bulk density

Dimensions in millimetres



a) Volumetric cup (Material: type 304 stainless seamless tubing)

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

a Weld all round and grind smooth.

Figure 3 — Volumetric cup and cup stand for determination of bulk density