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.

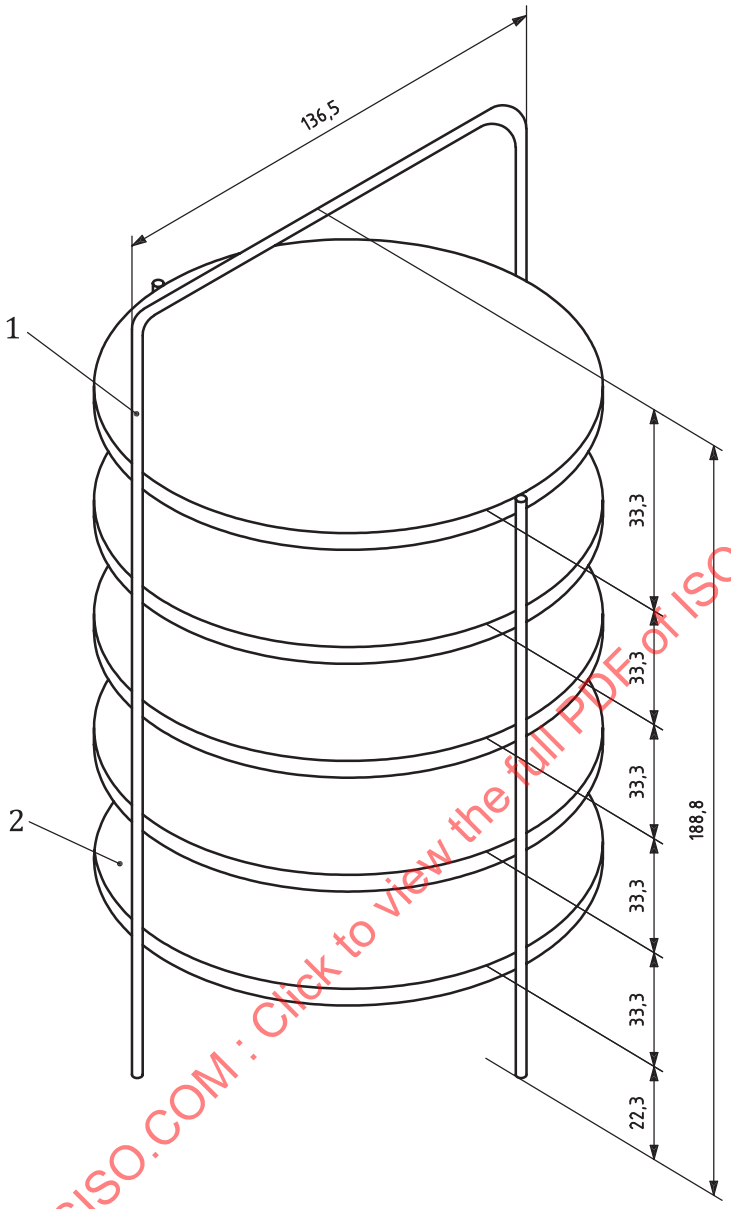
WARNING — PTFE resin can evolve small quantities of gaseous products when heated above 260 °C. Some of these gases are harmful. Consequently, exhaust ventilation must be used whenever the resins are heated above this temperature.

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

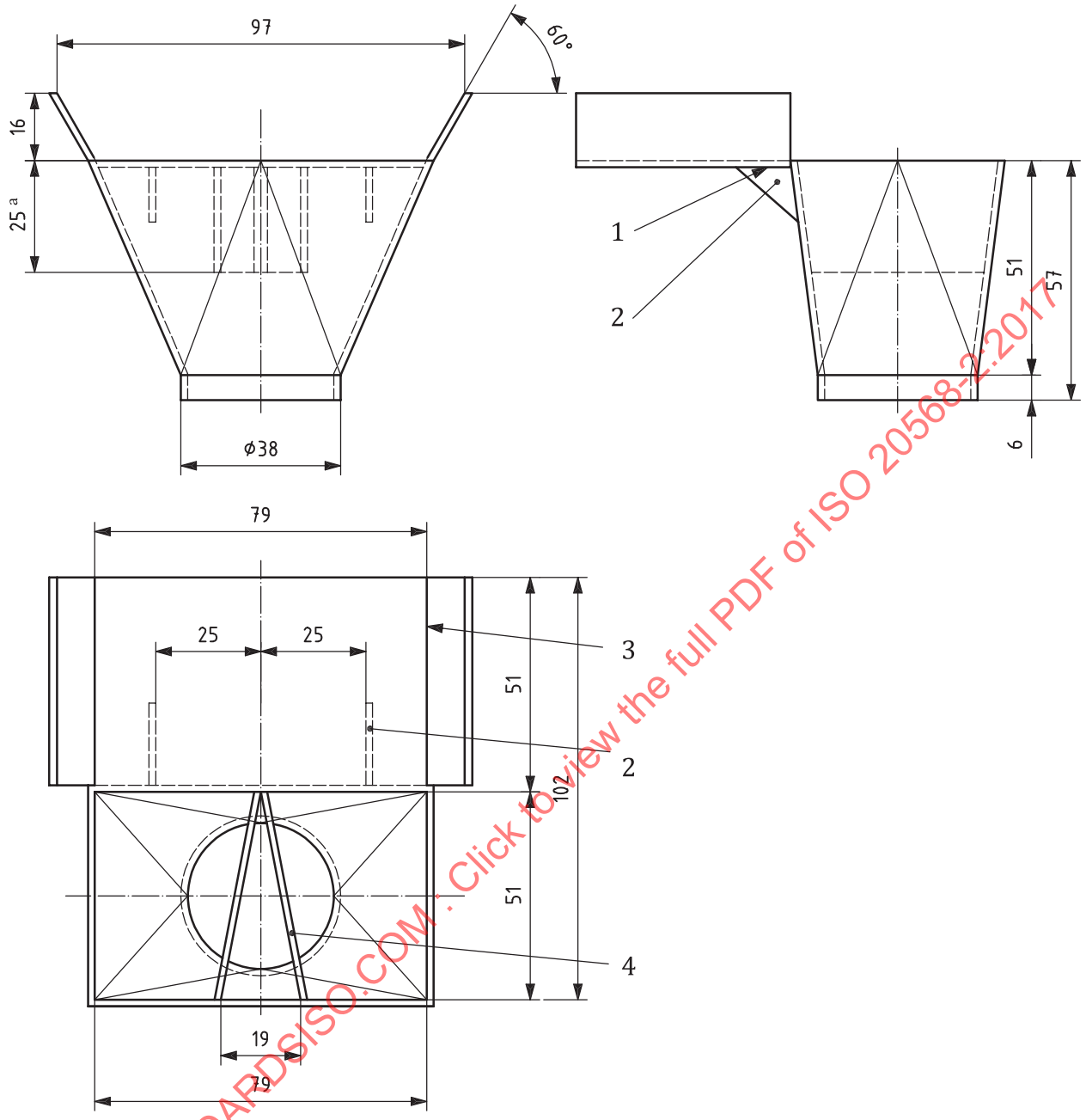
Calculate the bulk density, in grams per litre, as follows:

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

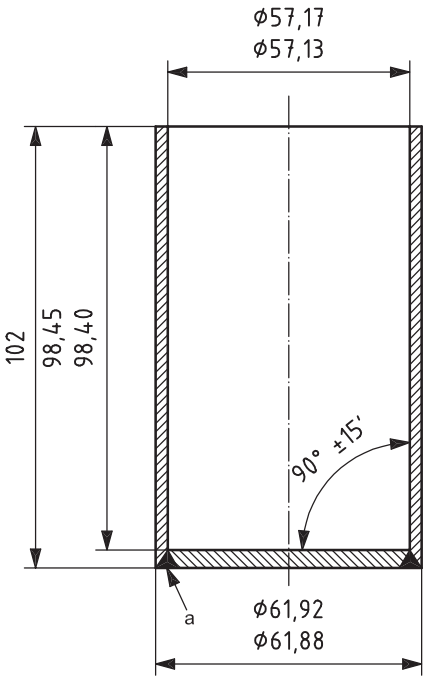
4 straightening vanes (locate two partitions as shown)

[a] Depth of partitions.

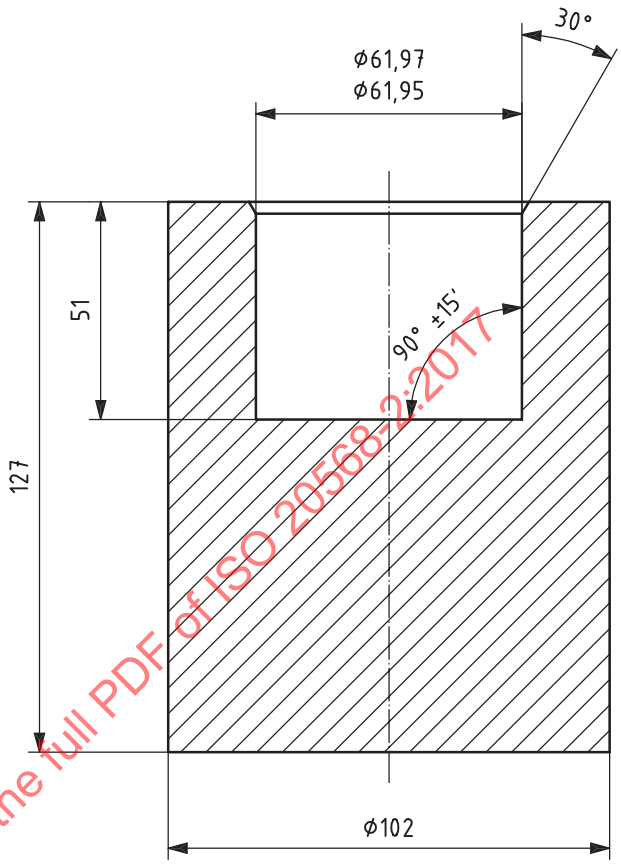
NOTE Funnel material: type 304 stainless steel, 16 gauge (1,6 mm thickness).

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

Dimensions in millimetres



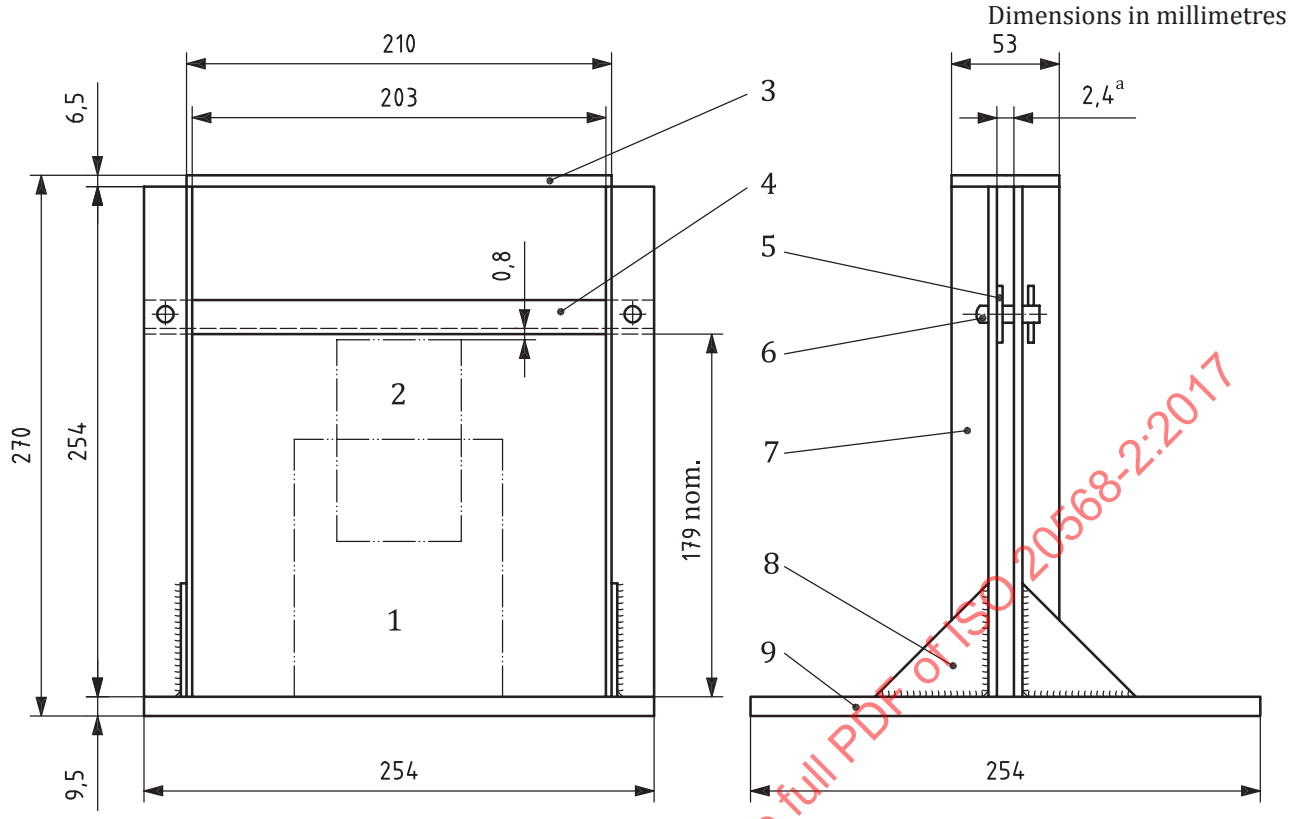
a) Volumetric cup
(Material: type 304 stainless seamless tubing)



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



Key

1 cup stand

2 cup

3 210 mm × 53 mm × 6 mm type 304 stainless-steel plate (or strap-welded across top for rigidity)

4 leveller blade (e.g. Atkins saw blade No. 614-P), sawtooth edge, six teeth per 25 mm, 1,6 mm deep

5 use shimstock or washers to take up clearance in 2,4 mm wide gap between the angle and saw blade

6 6 mm diam. × 19 mm long brass rivet (two required) plus 2,4 mm diam. × 19 mm long brass cotter pin (two required) for mounting saw blade firmly in position (drill hole through angle and blade to 0,12 mm clearance with diameter of cotter pin)

7 25 mm × 25 mm × 3 mm type 304 stainless-steel angles (four required — two each end)

8 51 mm × 51 mm × 3 mm stainless-steel gussets (four required — two each end)

9 type 304 stainless-steel plate

a Gap left between angles for mounting saw blade.

Figure 4 — Leveller stand for determination of bulk density

7.1.3 Particle size and size distribution

The wet and dry-sieve procedures of 7.1.3.1 and 7.1.3.2 are widely used with PTFE. The resistance-variation test procedure in 7.1.3.3 (the Coulter principle) is often used with PTFE fine-cut suspension powders. The light-scattering procedures in 7.1.3.4 are becoming more widely used. The use of automatic or other instruments that have been shown to provide equivalent results is an acceptable alternative.

7.1.3.1 Wet-sieve analysis

WARNING — Perchloroethylene is under investigation by government agencies and industry for its carcinogenic effects. Establish protective measures in accordance with applicable health and

safety requirements to prevent skin contact and provide ventilation of vapours. The supplier's SDS sheet should be consulted for more information about required safety measures.

7.1.3.1.1 Apparatus and materials

7.1.3.1.1.1 Balance, capable of weighing to ± 0,1 g.

7.1.3.1.1.2 Standard sieves, 203 mm diameter, conforming to ISO 565. It is suggested that the following sieve openings (sieve numbers) be used: 1,4 mm (No. 14), 1 mm (No. 18), 710 µm (No. 25), 500 µm (No. 35), 355 µm (No. 45), 250 µm (No. 60) and 180 µm (No. 80). The equivalent sieve numbers, given for information, are those defined in ASTM E11. Other sieve configurations may be used provided they give equivalent results. It is desirable to use a set of sieves that have openings that are uniformly related on a logarithmic scale.

7.1.3.1.1.3 Ventilated hood.

7.1.3.1.1.4 Six tared beakers, capacity 150 ml. As an alternative, the sieves can be tared, dried and weighed on a balance to avoid errors that can be introduced during transfer of fractionated samples to the tared beakers.

7.1.3.1.1.5 Sieving and solvent-spraying apparatus, a suggested arrangement for an apparatus with recirculating spray liquid is shown in Figure 5. The apparatus shall be located, and the operations carried out, in a ventilated hood or adequately ventilated area.

7.1.3.1.1.6 Spray liquid, 20 l. See the comments and warning in 7.1.3.1. Although perchloroethylene has been the usual choice, an alternative liquid may be used after its applicability and any hazards associated with its use should be investigated thoroughly and the use of the liquid may be satisfactory.

7.1.3.1.2 Procedure

7.1.3.1.2.1 Weigh out a 10 g test sample for powders with a particle size less than 100 µm or a 50 g test sample for powders with a larger particle size. Adjust the rate of flow of the spray liquid to 6 l/min ± 0,5 l/min.

7.1.3.1.2.2 Place the weighed resin on the top sieve and spray it with the organic spray liquid for 1 min ± 0,2 min. The shower head shall be about level with the top of the sieve and be moved in a circular fashion. Take care to break up all of the lumps and to wash the material from the sides of the sieve.

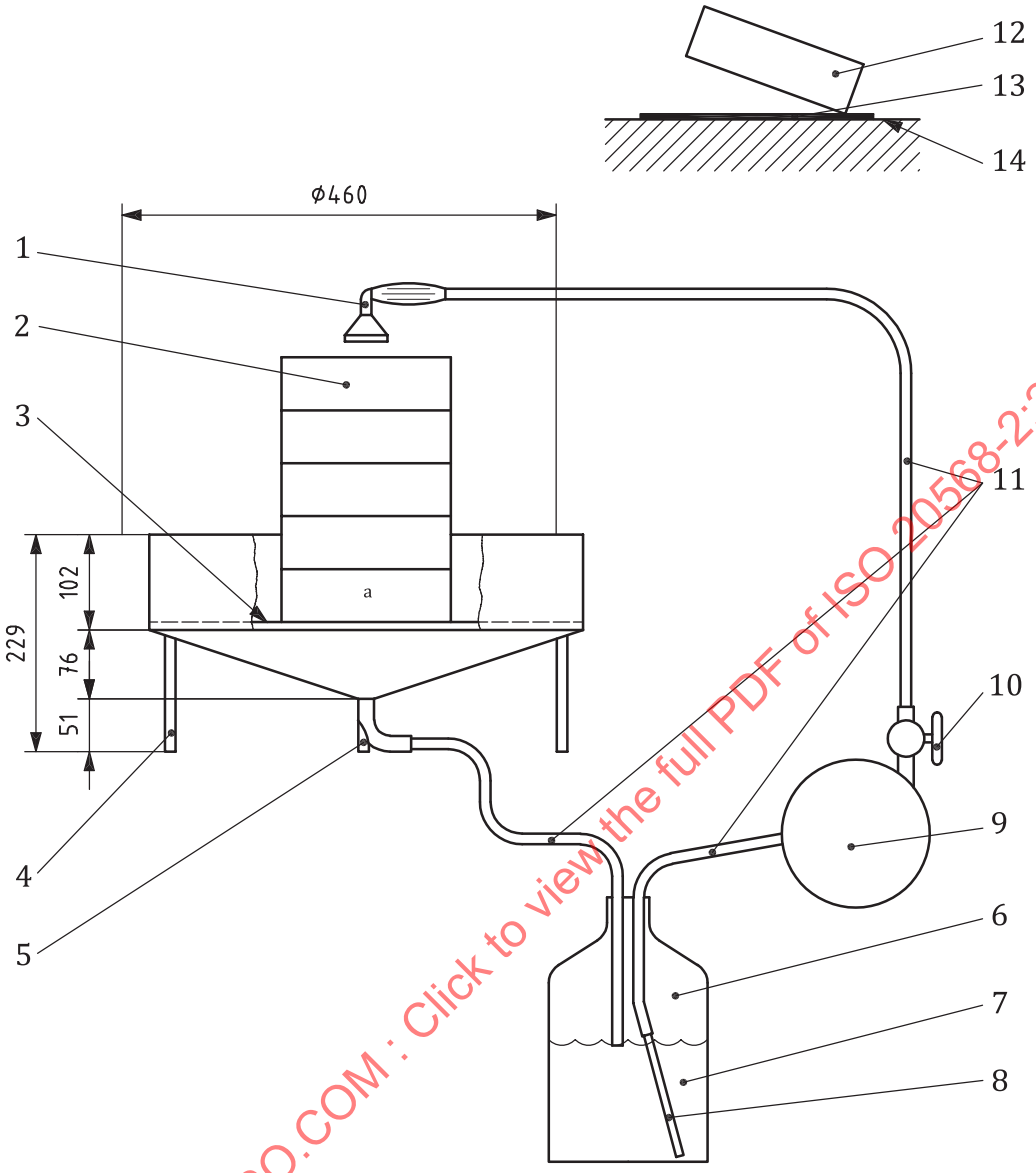
7.1.3.1.2.3 Remove the top sieve and place it in the hood to dry until all of the sieves are ready for oven drying as described in 7.1.3.1.2.4.

7.1.3.1.2.4 Repeat the procedure specified in 7.1.3.1.2.2 and 7.1.3.1.2.3 until all the sieves have been sprayed. Dry the sieves in a ventilated oven at a temperature of at least 90 °C up to a maximum of 130 °C for at least 15 min up to a maximum of 30 min and then cool to room temperature. Remove the resin from each sieve by tapping on a piece of paper as shown in the insert in Figure 5. Pour each fraction into a tared beaker and weigh to ±0,1 g.

7.1.3.1.2.5 Record the mass of resin on each sieve.

7.1.3.1.2.6 Clean the sieve by inverting it over filter paper and spraying it with spray liquid. Take care to prevent the resin from getting into the spray liquid.

Dimensions in millimetres



Key

1	portable all-purpose shower head	8	13 mm ext. diam. glass tubing
2	stacked sieves	9	centrifugal pump capable of delivering 6 l/min at shower head
3	grating	10	clamp to adjust flow rate
4	support	11	all-plastic tubing, int. diam. 13 mm
5	13 mm diam. drain	12	sieve
6	20 l carboy	13	sheet of paper
7	perchloroethylene (or alternative solvent)	14	table top

a Use a fine sieve at the bottom of the stack to prevent material from going into the reservoir. A standard 38 µm sieve has been found to be convenient.

Figure 5 — Apparatus for wet-sieve analysis

7.1.3.1.3 Expression of results

7.1.3.1.3.1 Calculate the net percentage of resin on each sieve as follows:

Net percentage of resin on sieve $Y = F \times m$

where

F = 2 for a 50 g test sample or 10 for 10 g test sample;

m is the mass, in grams, of resin on sieve *Y*.

7.1.3.1.3.2 Calculate the cumulative percentage of resin on each sieve as follows:

Cumulative percentage of resin on sieve *Y* = sum of net percentage on sieve *Y* and on sieves having sizes greater (i.e. numbers smaller) than sieve *Y*.

EXAMPLE Cumulative percentage on 500 µm (No. 35) sieve equals net percentage on 1,4 mm (No. 14) plus net percentage on 1,0 mm (No. 18) plus net percentage on 710 µm (No. 25) plus net percentage on 500 µm (No. 35) sieve.

7.1.3.1.3.3 Plot the cumulative percentage versus the sieve opening size (or sieve number) on log/log paper as shown in the sample plot in Figure 6. The sieve numbers and sieve opening sizes in micrometres are indicated below the figure. Draw the best straight line through the points and read the particle size at the 50 % cumulative percentage point ($d50$). Take this value as the average particle size. It is permissible to carry out the calculation of $d50$ by use of computer programmes that provide "best-fit" analysis using linear regression procedures involving a log-normal model.

7.1.3.2 Dry-sieve analysis

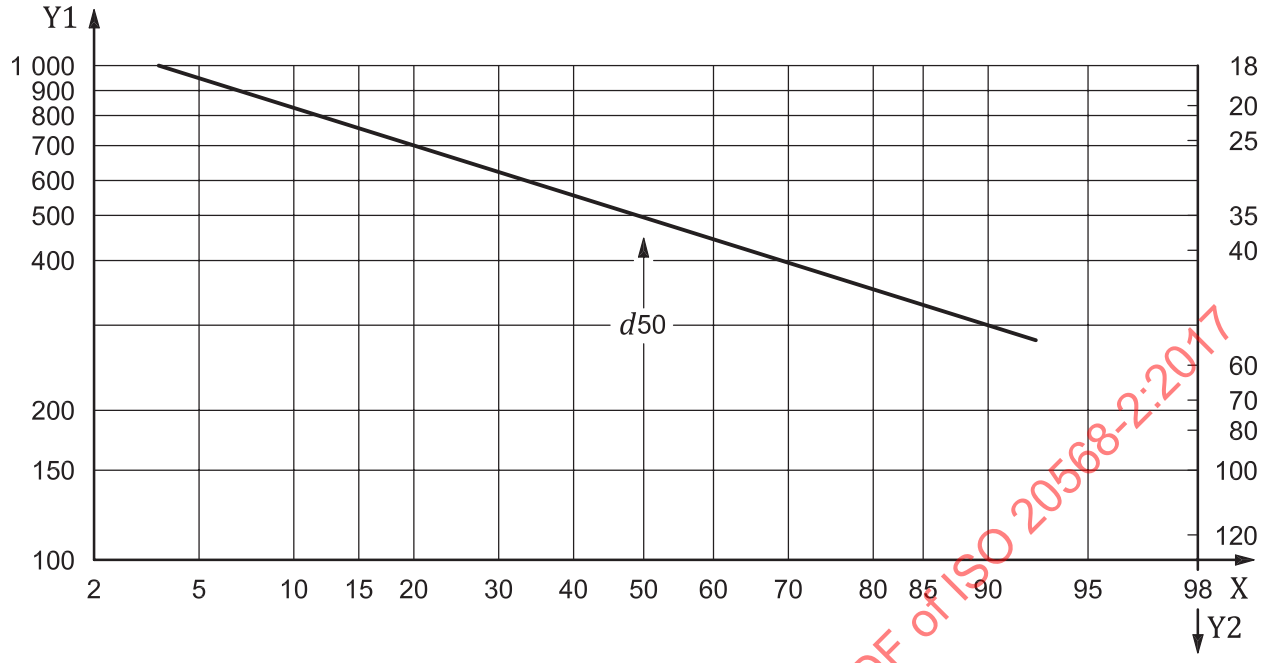
7.1.3.2.1 Apparatus

7.1.3.2.1.1 Balance, capable of weighing to ±0,1 g.

7.1.3.2.1.2 Standard sieves, 203 mm diameter, conforming to ISO 565. It is suggested that the following sieve openings (sieve numbers) be used: 1,4 mm (No. 14), 1 mm (No. 18), 710 µm (No. 25), 500 µm (No. 35), 355 µm (No. 45), 250 µm (No. 60) and 180 µm (No. 80). The equivalent sieve numbers, given for information, are those defined in ASTM E11. Other sieve configurations may be used provided they give equivalent results. It is desirable to use a set of sieves that have openings that are uniformly related on a logarithmic scale.

7.1.3.2.1.3 Mechanical sieve shaker, capable of imparting a uniform rotary and tapping action.

7.1.3.2.1.4 Freezer, any commercial ice freezer (a dry-ice chest may be used).



Key

X cumulative percentage, %

Y1 sieve opening size, µm

Y2 sieve no.

Sieve no.	Sieve opening size µm
14	**1 400**
18	**1 000**
25	**710**
35	**500**
45	**355**
60	**250**
80	**180**

Figure 6 — Typical log/log probability plot for sieve analysis

7.1.3.2.2 Procedure

7.1.3.2.2.1 Place 50 g ± 0,1 g of the sample in an aluminium pan, and cool the pan and contents to less than 10 °C.

7.1.3.2.2.2 Measure the tare mass of each of the sieves listed in 7.1.3.2.1.2. Place the conditioned test sample on the top sieve of the assembly and shake in the sieve shaker for 10 min ± 0,5 min. The dewpoint temperature of the sieving room shall be less than the temperature of the conditioned test sample so that water will not condense on the test sample during the test.

Determine the mass of resin retained on each sieve.

7.1.3.2.3 Expression of results

7.1.3.2.3.1 Calculate the net percentage of resin on each sieve as follows:

Net percentage of resin on sieve $Y = 2 \times m$

where

m is the mass, in grams, of resin on sieve Y.

7.1.3.2.3.2 Calculate the cumulative percentage of resin on each sieve as follows:

Cumulative percentage of resin on sieve Y = sum of net percentage on sieve Y and on sieves having sizes greater (i.e. numbers smaller) than sieve Y.

EXAMPLE Cumulative percentage on 500 µm (No. 35) sieve equals net percentage on 1,4 mm (No. 14) plus net percentage on 1,0 mm (No. 18) plus net percentage on 710 µm (No. 25) plus net percentage on 500 µm (No. 35) sieve.

7.1.3.2.3.3 Plot the cumulative percentage versus the sieve opening size (or sieve number) on log/log paper as shown in the sample plot in Figure 6. The sieve numbers and sieve opening sizes in micrometres are indicated below the figure. Draw the best straight line through the points and read the particle size at the 50 % cumulative percentage point ($d50$). Take this value as the average particle size. It is permissible to carry out the calculation of $d50$ by use of computer programmes that provide "best-fit" analysis using linear regression procedures involving a log-normal model.

7.1.3.3 Particle size and size distribution by the Coulter principle using a resistance-variation tester

7.1.3.3.1 Apparatus

7.1.3.3.1.1 Electric sensing-zone particle counter, with an orifice tube, such that most of the particles lie within its measurement range (2 % to 60 % of the orifice-tube diameter). Calibration of the instrument in absolute terms shall be done by the count-integration procedure on narrow distributions of standard latices (essentially monosized particles suspended in distilled water containing a surfactant) that are available from various sources. Poly (styrene-co-divinylbenzene) lattices are particularly recommended.

7.1.3.3.1.2 Analytical balance.

7.1.3.3.1.3 Magnetic stirrer.

7.1.3.3.1.4 Ultrasonic tank.

7.1.3.3.2 Procedure

7.1.3.3.2.1 Prepare a solution of a non-ionic surfactant[3)] (at a concentration of 0,2 g/l to 0,3 g/l in an aqueous electrolyte, such as a 1 % (mass per unit volume) solution of sodium chloride or a special 1 %

3) Triton X-100, a surfactant of the octylphenol series, has been found to be satisfactory. Other similar materials should be equally effective. 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.

(mass per unit volume) saline solution[4]. For example, four drops of the surfactant in 500 ml of electrolyte solution is sufficient to obtain a satisfactory suspension of the powder with moderate foaming. Filter the solution using a 0,3 µm barrier filter[5].

7.1.3.3.2.2 Weigh 100 mg of powder into a 100 ml beaker and add 40 ml of the surfactant solution.

7.1.3.3.2.3 Stir the slurry of powder using the magnetic stirrer at a high speed to ensure good wetting and deagglomeration of the powder. The proper dispersion conditions for a given mixer should preferably be determined in advance by plotting the average powder-particle diameter versus the speed and time used for mixing. It is important to avoid grinding of the powder (decrease in size of the ultimate particles). A typical set of conditions might involve stirring the slurry for 2 min to 3 min followed by 15 min at 700 r/min to 750 r/min.

7.1.3.3.2.4 Put the beaker into the ultrasonic tank, ensuring that the level of the suspension coincides with the level of water in the tank. The processing time and the frequency of the ultrasonic treatment are selected to avoid any undesirable effects of warming on the morphology of the suspended particles (e.g. fracture of the particles). An ultrasonic cell disrupter operated at 20 kHz for 4 min should be sufficient to obtain a uniform suspension of the fluoropolymer powder.

7.1.3.3.2.5 Resume stirring with the magnetic stirrer just rapidly enough to maintain a uniform suspension while removing 10 µl to 200 µl of the suspension, depending on the concentration. The analysis is made in a special 200 ml round-bottom container filled with the special 1 % (mass per unit volume) saline solution described in 7.1.3.3.2.1. Be careful to avoid the formation of air bubbles when filling.

7.1.3.3.2.6 Count particles in time mode using three measurements.

7.1.3.3.2.7 Correct the diameter as read to take into account the fraction with a diameter smaller than the last channel.

7.1.3.3.3 Expression of results

Plot a volume or mass percent curve on suitable graph paper as described for sieve analysis in 7.1.3.2.3.3.

7.1.3.4 Particle size and size distribution by light scattering of a laser beam

7.1.3.4.1 Apparatus

The particle-size analyser shall be based on Fraunhofer diffraction, Mie scattering or a combination of both light-scattering analysis techniques. Care shall be taken to ensure that the analyser system or subsystem is optimum for the size range of the powder being tested.

7.1.3.4.2 Procedure

Follow the instructions from the manufacturer of the instrument unless there is a standard method available for the particular material being tested.

4) A specially prepared solution, known as Isoton, is available from Coulter Counter Ltd. 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.

5) Available from the Millipore Corporation. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these materials. Equivalent products may be used if they can be shown to lead to the same results.

7.2 Testing of polytetrafluoroethylene (PTFE) dispersion

7.2.1 Preparation of test samples

PTFE solids in the dispersion tend to settle upon standing. Therefore, homogenize the dispersion by gentle mixing before sampling. Gentle mixing can be accomplished by rolling a drum for 5 min at 3 r/min to 4 r/min, by stirring with a smooth rod for 3 min to 4 min, or by other types of gentle agitation.

WARNING — Excessive agitation can coagulate the dispersion.

7.2.2 Percentage PTFE and surfactant in aqueous dispersion

7.2.2.1 PTFE solids and surfactant content by mass loss

Percentage PTFE solids and the percentage surfactant can be determined by successive evaporations of water and surfactant. The percentage surfactant is based on the mass of PTFE present in the dispersion. All percentages are based on mass.

7.2.2.2 Apparatus

Required are an aluminium weighing dish, an oven capable of reaching 120 °C ± 5 °C, an oven capable of reaching 380 °C ± 10 °C, a desiccator and a balance capable of weighing to 0,1 mg.

7.2.2.3 Procedure

Weigh the aluminium weighing dish to 0,1 mg (m_1). Add 10 g of PTFE dispersion and reweigh immediately to 0,1 mg (m_2). Dry the test portion for 2 h at 120 °C ± 5 °C. Reweigh the test portion to 0,1 mg (m_3) after cooling to room temperature in the desiccator. After weighing, evaporate the surfactant by placing the test portion in an oven at 380 °C ± 10 °C for 35 min ± 1 min. Allow the sample to cool in the desiccator to room temperature and weigh to 0,1 mg (m_4).

7.2.2.4 Expression of results

For surfactants that are completely volatile, use Formulae (1) and (2):

$$\% \text{ PTFE} = \frac{m_4 - m_1}{m_2 - m_1} \times 100 \tag{1}$$

$$\% \text{ surfactant} = \frac{m_3 - m_4}{m_4 - m_1} \times 100 \tag{2}$$

For surfactants that are not completely volatile, use Formulae (3) and (4):

$$\% \text{ PTFE} = \frac{(m_3 - m_1) - (m_3 - m_4)(1 + k)}{m_2 - m_1} \times 100 \tag{3}$$

$$\% \text{ surfactant} = \frac{(m_3 - m_4)(1 + k)}{(m_3 - m_1) - (m_3 - m_4)(1 + k)} \times 100 \tag{4}$$

where

k is the mass of the non-volatile portion of the surfactant divided by the mass of the volatile portion of the surfactant.

Upon request, the supplier shall inform the user whether the surfactant can be completely removed by the procedures of 7.2.2.3 and, if not, the manufacturer shall define the surfactant or the volatile and non-volatile portions of the surfactant.

8 Testing of melt processable fluoropolymers

8.1 Testing of CPT, ECTFE, EFEP, ETFE, FEP, PFA, PVDF, PVF, VDF/CTFE, VDF/HFP, VDF/TFE, VDF/TFE/HFP

8.1.1 Melting-peak temperature

8.1.1.1 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 D4591 or ISO 11357-3 are appropriate for this determination.

8.1.1.2 Use differential scanning calorimetry (DSC) as described in ISO 11357-3 and ASTM D4591 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.

8.1.2 Melt mass-flow rate (MFR)

Melt mass-flow rate or melt volume-flow rate shall be determined in accordance with ISO 1133-1 as modified by details provided in this document. Use of automated or other instruments that have been shown to provide equivalent results shall be an acceptable alternative to the detailed procedures given in this document.

8.1.2.1 Test conditions

The melt-flow rate is determined using the conditions for the fluoropolymer type shown in Table 2 and using a modification of the extrusion plastometer described in ISO 1133-1. The sample may be pellets or powder. For use with semifinished forms or moulded articles, pieces of approximately the same size may be cut from a moulded or extruded form. Strips may also be handled conveniently.