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Protective clothing — Protection against chemicals — Measurement of cumulative permeation of chemicals with low vapour pressure through materials

Habillement de protection — Protection contre les produits chimiques — Mesure de la perméation cumulée à travers des matériaux des produits chimiques ayant une faible pression de vapeur

Reference number
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Foreword

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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 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 94, *Personal safety — Protective clothing and equipment*, Subcommittee SC 13, *Protective clothing*.

Introduction

When assessing the performance of personal protective equipment (PPE) against chemical risks, it is clearly important to determine the ability of PPE materials to resist chemical ingress. The term "ingress" is used here specifically so as not to distinguish between penetration and permeation. Penetration is ingress through physical holes in the fabric such as pores, stitch-holes, and gaps in a protective coating. Penetration is usually quite a rapid process, leading to the ingress of chemical within a matter of minutes, whereas permeation is usually a slower process with molecular diffusion through a polymer or elastomer. However, from the perspective of the wearer of PPE (gloves, footwear, protective clothing) the mechanism of ingress is far less relevant than the fact that a quantity of chemical may at some point migrate through the fabric of the personal protective equipment.

Standards to measure permeation are designed to measure the performance of materials that include a polymer or elastomer as the movement in these materials is at the molecular level. However, although they are intended to measure movement of chemicals at a molecular level, it may be difficult to differentiate between penetration and permeation in materials with small pores, pinholes, and gaps in coating.

A number of standards, including ISO 6529, EN 16523-1 and ASTM F739, measure permeation of chemicals that are volatile and/or soluble in water or other liquid or gaseous collection media. In the above-mentioned standards, volatility or solubility in water or other liquid that does not interact with the material allows use of gaseous or liquid collection medium. However, based on the scopes of ISO 6529, EN 16523-1 and ASTM F739, these standards are not well-suited for quantifying the ingress of chemicals and mixtures that are involatile and/or insoluble in water and other liquids that do not interact with the material being tested. This document complements the above-mentioned standards as it is suitable for measuring chemicals that cannot be measured by other standards and vice versa.

This document is intended to be used to evaluate the barrier effectiveness materials used in personal protective equipment against permeation by solid and liquid chemicals with low vapour pressure and/or low solubility in commonly used liquid and gaseous collection media. This test method is not suitable for measurement of volatile chemicals that may evaporate before the chemical analysis is complete.

Protective clothing — Protection against chemicals — Measurement of cumulative permeation of chemicals with low vapour pressure through materials

1 Scope

This document describes laboratory test methods to determine the resistance of materials, closures, and seams used in personal protective equipment (PPE) to permeation by solid or liquid chemicals with low vapour pressure (less than 133,322 Pa at 25 °C) and/or insolubility in water or other liquids commonly used as collection media. These chemicals that are often part of pesticide formulations and other mixtures cannot be measured using other standards for measuring permeation. This test method is suitable for field strength and concentrated pesticide formulations as well as other mixtures in which the active ingredient is a chemical with low vapour pressure and/or low solubility in commonly used liquid and gaseous collection media.

This test method is not intended to be used in place of standards such as ISO 6529, EN 16523-1 and ASTM F739, which measure permeation of chemicals that are either volatile or soluble in water or other liquids that do not interact with the material being tested. This document is not suitable for measurement of volatile chemicals that may evaporate before the chemical analysis is complete.

The degree of contamination depends on numerous factors, such as type of exposure, application technique, and chemical formulation. As the level of exposure can vary considerably, this method is designed to rate relative performance of PPE materials for different durations.

This method is designed to measure cumulative permeation. Breakthrough time cannot be measured by this method. This test method does not measure resistance to penetration or degradation.

The test method standard may be used for the evaluation of PPE materials that are new or those for which the product standard requires treatment, such as laundering or simulated abrasion. Details of the treatment shall be reported.

2 Normative references

No normative references are required for this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

- ISO Online browsing platform: available at http://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

3.1
analytical technique
method of quantifying the amount of permeated chemical in the collection medium

Note 1 to entry: Such methods are often specific to individual chemical and collection-medium combinations.

3.2
breakthrough detection time

elapsed time measured from the start of the test to the sampling time that immediately precedes the sampling time at which the test chemical is first detected

3.3
cumulative permeation mass

total amount of chemical that permeates during a specified time from the time the material specimen is first contacted with the test chemical

3.4
degradation

deleterious change in one or more properties due to contact with a chemical or heat

3.5
limit of quantification

minimum quantity of a substance which can be measured

Note 1 to entry: It is the value where the uncertainty of measurement is equal to 50% of the determined value.

3.6
penetration

process by which a chemical and/or microorganism moves through porous materials, seams, pinholes, or other imperfections in a material on a non-molecular level

Note 1 to entry: For the purpose of this document, penetration refers to only chemicals, not micro-organisms.

Note 2 to entry: For the purpose of this document, materials include protective clothing, footwear, and glove materials.

3.7
permeation

process by which a chemical moves through a material on a molecular level

Note 1 to entry: Permeation involves (1) adsorption of molecules of the chemical onto the contacted (outside) surface of a material, (2) diffusion of the adsorbed molecules into and through the material, and (3) desorption of the molecules from the opposite (inside) surface of the material.

Note 2 to entry: For the purpose of this document, materials include protective clothing, footwear, and glove materials.

3.8
solid collection medium

solid material on the “clean” side of the test specimen in which any permeated chemical is collected

3.9
test chemical

chemical or mixture of raw materials, including but not limited to, active ingredients, inert ingredients, and a base solvent used in a formulation

Note 1 to entry: For the purpose of this test method, test chemicals are limited to chemicals (including mixtures) with low vapour pressure less than 133,322 Pa at 25 °C and/or insolubility in water or other liquids commonly used as collection media.

3.10
vapour pressure

pressure exerted by the vapour above the liquid in equilibrium at a given temperature

3.11
insoluble chemical

chemical having solubility of less than 50 mg/l at 23 °C in water or other liquids commonly used as collection media

3.12

closure

system or component which makes possible the closing of an item of PPE

4 Principle

A prepared collector disc shall be used to measure the cumulative permeation of chemicals with low vapour pressure and/or insolubility in water and other commonly used collection media.

The collector disc is placed under the test material in the test assembly (see Annex A). The cell is placed on a horizontal surface and filled with test chemical.

After the predetermined duration of the test, the cell is emptied and the collector disc is removed for extraction and quantitative analysis. The predetermined duration stated in the product standard should be used when testing.

The data are used to calculate cumulative permeation.

5 Apparatus and supplies

5.1 The permeation cell consists of a base and a cylinder that are assembled with three bolts to form a cell. The centre of the base and the bottom surface of the cylinder are raised to improve contact between the two surfaces. See Annex B for a technical drawing, including measurements for the permeation cell. The gasket, collector disc, and test specimen are placed between the base and the cylinder. The test specimen's normal inside surface is in contact with the collector disc. The material's outer surface is toward the side that faces the test cylinder. The two pieces are connected with three bolts. The top surface of the cylinder has a spout to make it easier to drain the test chemical. The schematic diagram of the permeation cell is shown in Annex A.

The cylindrical, hollow insert illustrated in Figure B.4 fits into the centre of the cell. The insert is to maintain contact between the collector disc and the test specimen while the cell is being assembled. It shall be filled to bring the mass to (100 ± 1) g.

See Annex B for the drawing and description of PTFE cell used for the interlaboratory study. Measurements (including tolerance limits) in Figures B.2 to B.4 pertain to the cell in Annex B.

NOTE 1 Annex C includes a list of suppliers for the PTFE permeation cell, gaskets, and collector discs.

NOTE 2 Cells made with other materials and designs have not been tested.

NOTE 3 Other cells with similar design that give equivalent results could also be used.

5.2 Gasket, with (90,0 ± 0,1) mm outer diameter (OD) and (35,0 ± 0,1) mm inner diameter (ID) with holes for the bolts.

The gasket shall be used to prevent leakage. Care shall be taken to ensure that test chemical cannot leak out of the challenge side of the apparatus, flow around the edge of the test specimen, and then leak into the collection side. This is most likely to happen by capillary action if the outer side of a multicomponent material is from woven or nonwoven material. The size of the gasket may be changed to control leakage. Information on selection and size of gaskets is included in Annex D.

5.3 Collector disc, a solid collection medium, with (50 ± 1) mm diameter and (20,0 ± 0,5) mm pre-cut centre, made of Benchkote® Plus[1]. The pre-cut centre is attached to the outer ring at two opposite ends.

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[1] Benchkote® Plus is the trade name of a polyethylene-backed, absorbent paper manufactured by Whatman/GE Healthcare. Benchkote® Plus is available through scientific product suppliers. This information is given for the convenience of users and does not constitute an endorsement by ISO of this product.

NOTE The pre-cut centre of the collector disc is designed so that it is attached to the outer ring at two points and can easily be removed for extraction. Only the pre-cut centre is extracted to prevent contamination around the edges that can affect test results.

5.4 Solvent, shall be selected for preparing the collector disc and extracting the chemical from the collector disc. Selection of the solvent is based upon the chemical to be extracted and analytical method used for chemical analysis. Care should be taken to ensure that the solvent present in the collector disc does not degrade or damage the PPE material being tested.

NOTE 1 Acetonitrile is suitable for preparing the collector disc when extraction is conducted with HPLC grade acetonitrile or other liquids that are miscible with acetonitrile.

NOTE 2 A small amount of solvent in the collector disc should not affect the PPE material. A blank test without challenge chemical can be carried out to confirm that there are no adverse effects of the solvent on the PPE material.

5.5 Pipettor, for pipetting (1,00 ± 0,05) ml of solvent to prepare the collector disc.

5.6 Absorbent paper, one 80 mm × 80 mm square for preparation of each collector disc. The absorbent paper squares shall be cut from Benchkote® Plus[1] paper sold as sheets.

NOTE Benchkote® Plus absorbent paper sold as sheets is specified due to the difference in absorbency between Benchkote® Plus paper sold as a roll and as sheets.

5.7 Stopwatch or **electronic timer**.

5.8 Balance, accurate to 0,001 g. for weighing collector discs and 0,1 g for weighing test chemical.

5.9 (200 ± 2) g weight with a flat base that is (60 ± 1) mm in diameter to be placed on top of the collector disc.

NOTE The weight may be made of aluminium, brass, or any other metal.

5.10 Tweezers.

5.11 Hole punch tool, for punching holes accurately in test specimens.

5.12 Torque wrench, for tightening nuts in 4 Nm to 6 Nm range.

5.13 Orbital shaker, capable of (1,7 ± 0,2) s^{-1}

5.14 Bottles, airtight chemically resistant, wide-mouth bottles (minimum opening of 25 mm) for extraction.

6 Test parameters

6.1 Analytical method

6.1.1 The method used to quantify the mass of test chemical in the collector disc shall be determined before conducting the test.

6.1.2 Any analytical method may be used provided that:

— it is suitably sensitive to the test chemical. The limit of quantification (LOQ) shall be 0,1 µg/cm² or lower;

- it is either not significantly sensitive to the collection medium or any such sensitivity can be accurately determined and discounted; and
- it is not significantly sensitive to any likely minor impurities either inherent in the collection medium or introduced into the collection medium by prolonged contact with the test specimen. For example, when testing "powdered gloves," the analytical method should not be sensitive to powders applied to the insides of gloves during manufacture in order to facilitate donning.

6.1.3 Analytical techniques such as gas chromatography or high performance liquid chromatography may be used to measure the mass of the test chemical. This requires extraction (in most cases) and analysis of the extract.

6.2 Determination of extraction efficiency

6.2.1 To measure the extraction efficiency, spike three collector discs with 2 µg of the chemical to be analysed. For spiking, use the solution prepared for calibration.

6.2.2 Extract the collector discs using the procedure in 8.2.

6.2.3 Analyse the extracts using the analytical technique selected in 6.1.

6.2.4 Calculate extraction efficiency using Formula (1):

$$E_\mathrm{E} = (m_\mathrm{e}/m_\mathrm{a}) \times 100 \tag{1}$$

where

E_E extraction efficiency, in %;

m_e amount extracted, in µg;

m_a amount applied, in µg.

6.2.5 The extraction efficiency shall be between 90 % and 110 % for all three spiked collector discs. Repeat the procedure to determine extraction efficiency with another solvent if the extraction efficiency is not within the acceptable limit.

6.3 Test specimens

This document does not define from where to select the test specimens. The selection of test specimens shall be given in product or performance specification standards.

The test for a PPE material or seam is performed on 3 test specimens, unless otherwise specified in the performance or product standard.

For multiple layer PPE materials in which the layers are separate, the internal layer that has no effect on the chemical protection may be removed (e.g. thermal insulation layer).

Each PPE material or seam specimen shall be cut to a minimum diameter of 56 mm to ensure that it covers the entire surface of the cell. Larger specimens cut as circles or squares that extend beyond the edge of the 90 mm outer diameter of the cell are permissible.

NOTE It may be preferable to let the specimen extend beyond the edge, especially for materials in which the surface promotes wicking.

7 Preparation of test chemical and test specimens

Samples should be subjected to treatments such as laundering specified in the product standard and conditioned for a minimum of twelve hours prior to testing. Test specimens shall be conditioned and tested at (23 ± 3) °C. The temperature of the test chemical shall also be (23 ± 3) °C at the time of testing.

If the conditioning and testing are not conducted in the same location, testing of specimens shall commence within 10 min of removing the specimens from the conditioning atmosphere.

NOTE 1 In many cases this can be achieved by storing the specimen at the test temperature for several hours.

NOTE 2 It is intentional that no conditioning humidity is specified for test specimens. Once the test has commenced, the contamination side will be in constant contact with liquid test chemicals and therefore does not have to be conditioned at a certain humidity.

8 Procedure

8.1 Contamination

8.1.1 Shake the test chemical well prior to testing.

A magnetic stirrer is recommended.

8.1.2 Insert the three bolts with the washers through the base of the permeation cell and place it on a flat surface.

8.1.3 Prepare the collector disc.

8.1.3.1 Weigh the collector disc, and then place it in a petri dish with the polyethylene film facing down.

8.1.3.2 Pipette (1,00 ± 0,05) ml of the solvent onto the absorbent side, ensuring that the entire surface is covered.

NOTE The collector disc is prepared with solvent to improve contact between the collector disc and the test specimen.

8.1.3.3 Use tweezers to carefully lift the collector disc and place it in the centre of the 80 mm × 80 mm Benchkote® Plus absorbent paper with the absorbent side in contact with the absorbent side of the collector disc.

8.1.3.4 Place the (200 ± 2) g weight on top of the collector disc for (60 ± 5) s.

8.1.4 Use tweezers to carefully lift the prepared disc, weigh the disc, record the mass, and place the collector disc immediately on the base of the permeation cell with the polyethylene side of the collector disc facing the base.

The test cell shall be assembled close to the balance to minimize the evaporation of solvent once the collector disc is weighed.

NOTE When acetonitrile is used as the solvent, the amount of solvent in the 35 mm collector disc is typically 0,49 g to 0,55 g.

8.1.5 Quickly place the test specimen on top of the collector disc. Care should be taken to ensure that the inside of the material is in contact with the collector disc.

8.1.6 Place the gasket on top of the test specimen and then place the insert that weighs (100 ± 1) g in the 35 mm opening.

NOTE The insert maintains contact between the collector disc and the test specimen while the cell is being assembled.

8.1.7 Place the cylinder on the gasket with the spout-side facing up. Tighten the nuts by hand and then use a torque wrench to ensure that there is sufficient pressure to create a reliable liquid-tight seal.

NOTE The pressure required to achieve a liquid-tight seal is dependent on factors such as the material and gasket. Typical range is 4 Nm to 6 Nm. Higher pressure may damage the cell.

8.1.8 Remove the insert after tightening the nuts.

8.1.9 Weigh (35 ± 1) g of the test chemical, and carefully pour it into the test chamber in its entirety.

8.1.10 Check to ensure there is no leakage. If there is leakage, remove the liquid and start again from 8.1.1.

8.1.11 After the pre-determined time, empty the cell, carefully remove the nuts, and remove the cylinder and test specimen.

NOTE Pre-determined time (e.g. one hour) is specified as part of product or performance standards.

8.1.12 Use tweezers to carefully lift the 20 mm, pre-cut centre of the collector disc and place it in a wide-mouth, glass bottle with a screw top.

8.2 Extraction and analysis

8.2.1 Add (20,00 ± 0,05) ml of the selected solvent to the wide-mouthed bottle using a graduated cylinder, bottle-top dispenser, or other suitable apparatus. Ensure that the collector disc is in contact with the solvent and the bottle is secured tightly.

8.2.2 Set the orbital shaker speed to (1,7 ± 0,2) s^{-1}.

8.2.3 Place the bottles on the orbital shaker and set the timer for (30 ± 1) min.

8.2.4 Start the shaker and the timer, and extract for (30 ± 1) min.

8.2.5 After (30 ± 1) min, transfer the extract from the bottle to the storage bottles. Tighten the caps on the openings of the storage bottles.

NOTE Use good laboratory practice for the disposal of toxic substances and clean-up of laboratory glassware/apparatus.

8.2.6 Analyse the extracts using the analytical procedure selected in 6.1.

9 Expression of results

For each specimen, use Formula (2) to calculate cumulative permeation:

$$P_c = (c \cdot V)/A \quad (2)$$

where

P_c is the cumulative permeation, µg/cm^2 with one decimal place;

c is the concentration of chemical of interest in extractant, µg/ml;

V is the volume of extractant, ml;

A is the area of the collector disc that was extracted, cm^2.

The result, R_1, is the average value of the 3 specimens.

If the 3 test results are within 20 % of R_1 (included in the range [0,8 × R_1, 1,2 × R_1]), the test is validated and R_1 is reported.

If the 3 test results are not in the defined range, a new set of 3 test specimens shall be tested.

R_2 is determined as the average value of the 3 new test specimens.

If the second set of 3 test results are within 20 % of R_2 (included in the range [0,8 × R_2, 1,2 × R_2]), the second test is validated. R_2 is reported.

If the second set of 3 test results is not in the defined range, the 6 results are reported and the tested material will be reported as inhomogeneous.

The uncertainty of measurement for each test method described in this document shall be assessed and should be used to decide acceptance/rejection as specified in the product standard.

Note One of the following approaches should be used:

- a statistical method, e.g. that given in ISO 5725-2;
- a mathematical method, e.g. that given in ISO Guide 98-3;
- uncertainty and conformity assessment as given in ISO Guide 98-4.

10 Report

The following information shall be included in the test report:

a) a reference to this document, i.e. ISO 19918;

b) the identification of the material, including supplier, trade name, and proprietary components;

c) details of the treatment (such as laundering and simulated abrasion) of test specimen. This is required only if the test specimens were subjected to a treatment prior to testing;

d) test duration expressed in minutes;

e) temperature of the test chemical and testing conditions;

f) the information that describes the test chemical. If a commercial product was used, include the trade name, active ingredient, and the concentration used for testing;

g) solvent used for preparation of collector disc and extraction;

h) details of the analytical technique;

i) the results for each specimen and the mean;

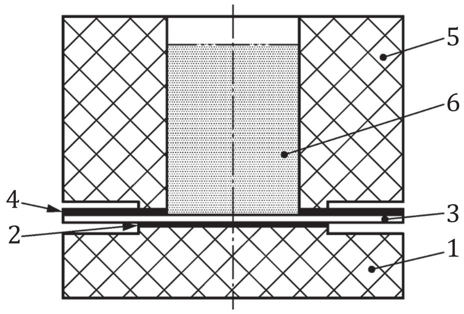
j) any deviation to the standard and their justifications.

11 Precision and bias

The precision and bias information is included in Annex E.

Annex A
(informative)

Schematic diagram of the permeation cell



Key

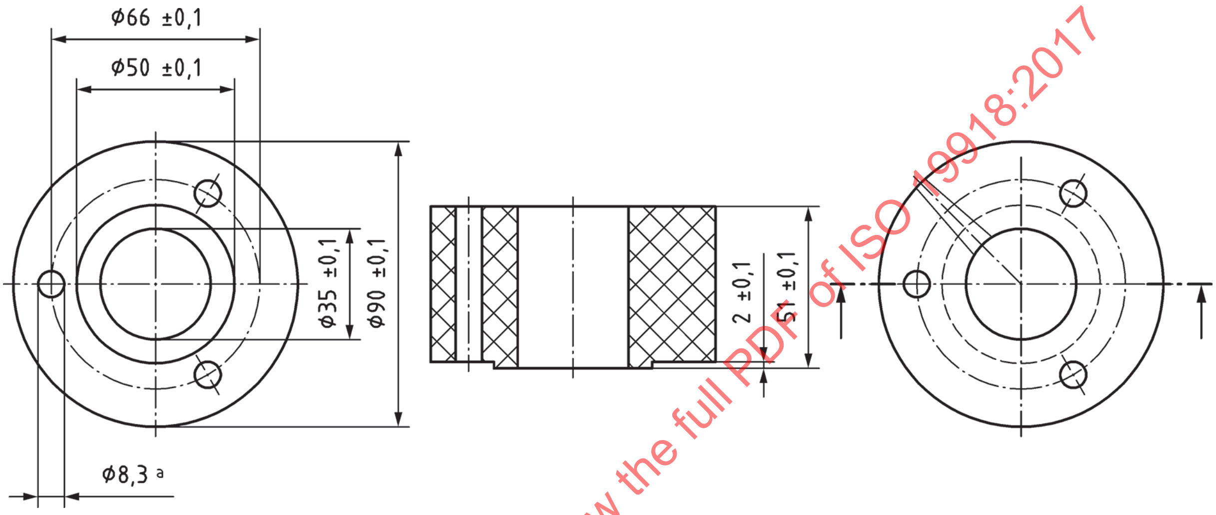
1	cell base
2	collector disc
3	test specimen
4	gasket
5	cell cylinder
6	test chemical

Figure A.1 — Schematic diagram of the test cell (side view)

Annex B
(normative)

Drawing and measurements of the permeation cell, washer, and bolts

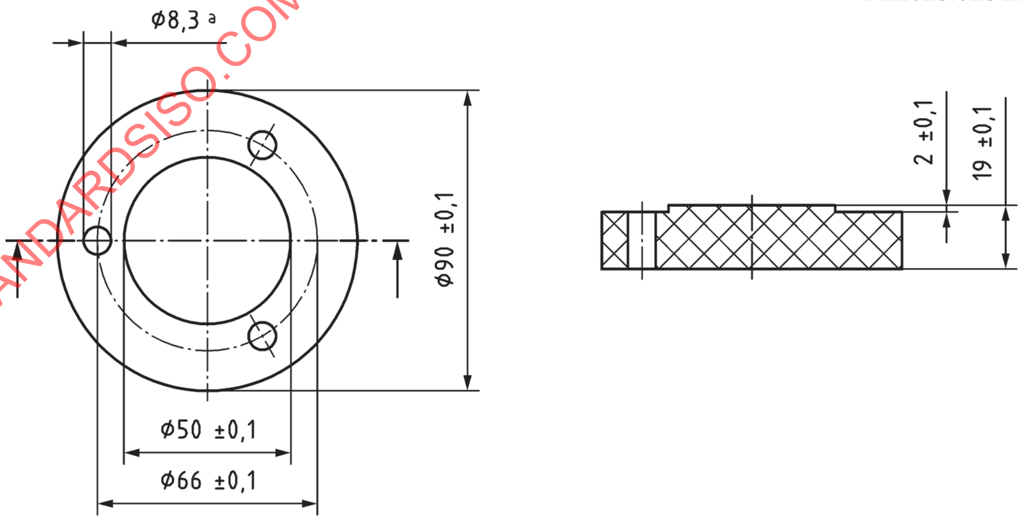
Dimensions in millimetres



Bottom view **Cross-section** **Top view with spout**

Figure B.1 — Drawing and measurements of cell cylinder

Dimensions in millimetres



Top view **Cross-section**

Figure B.2 — Drawing and measurements of cell base

Dimensions in millimetres

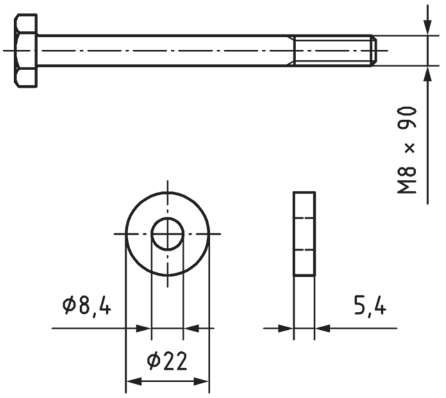


Figure B.3 — Drawing and measurements of bolt and washer

Dimensions in millimetres

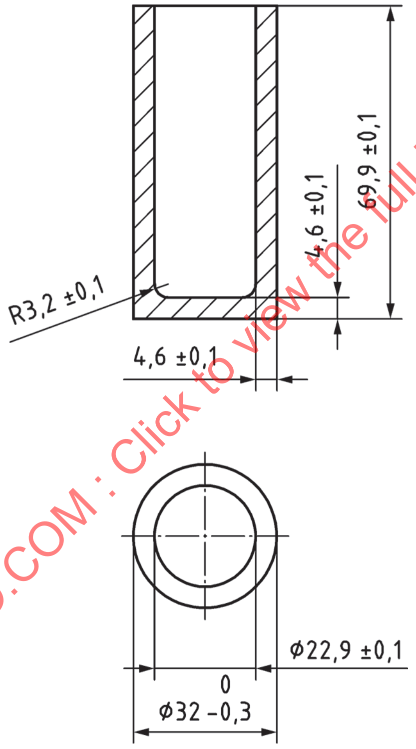


Figure B.4 — Drawing and measurements of cylindrical, hollow insert

Annex C
(informative)

Sources of permeation test cells, and permeation cell parts[2)]

PTFE permeation cell and cylindrical hollow insert may be purchased from:

Applied Plastics Technology, Inc.

45 Broad Common Road

Bristol, RI 02809

USA

Email: info@ptfeparts.com

Phone: +41 401 253 0200

Gaskets and collector discs may be purchased from:

Seal & Design Inc.

4015 Casilio Parkway

Clarence, NY 14031

USA

Email: gasket@sealanddesign.com

Phone: +716 759 2222

The above information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

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2) The products given in Annex C are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.