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Protective clothing — Protection against chemicals — Determination of resistance of protective clothing materials to permeation by liquids and gases

Vêtements de protection — Protection contre les produits chimiques — Détermination de la résistance des matériaux utilisés pour la confection des vêtements de protection à la perméation par des liquides et des gaz

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6529 was prepared by Technical Committee ISO/TC 94, *Personal safety — Protective clothing and equipment*, Subcommittee SC 13, *Protective clothing*.

This third edition cancels and replaces the second edition (ISO 6529:2001), which has been technically and editorially revised in order to:

a) clarify explicitly that the testing of gloves and, when fitted to chemical protective clothing, boots, is included;

b) include a normative annex defining specific default procedures for testing, calculating and reporting permeation resistance;

c) permit the testing of fabrics, regardless of whether they are, to some degree, air-permeable or totally air-impermeable;

d) introduce a pre-test which is used in order to determine the number of replicate tests to be carried out;

e) include more explanatory notes;

f) introduce an informative annex giving technical advice on the testing of seams and closures;

g) include worked examples of the calculation of results;

h) specify the minimum frequency of sampling the collection medium during a test;

i) clarify the reporting of sets of replicate results in cases in which the inter-sample variation is large.

Introduction

People involved in the production, use, transportation and emergency response with liquid and gaseous chemicals can be exposed to numerous compounds capable of causing harm upon contact with the human body. The deleterious effects of these chemicals can range from acute trauma such as skin irritation and burn to chronic degenerative disease, such as cancer. Since engineering controls may not eliminate all possible exposures, attention is often placed on reducing the potential for direct skin contact through the use of protective clothing that resists permeation, penetration and degradation.

The test methods described in this International Standard are intended to be used to evaluate the barrier effectiveness of materials used for protective clothing (see Note) against ingress by liquid or gaseous chemicals. Options are provided for conducting this testing under both conditions of continuous or intermittent contact with the chemicals.

These test methods provide options for reporting test results in terms of breakthrough time, permeation rate and cumulative permeation. These parameters are key measures of the effectiveness of a clothing material to act as a barrier to the test chemical. Long breakthrough times, low permeation rates and low cumulative permeation mass are characteristic of high level barrier materials.

Resistance to penetration by liquid chemicals should be determined by using ISO 6530 while resistance to penetration by liquid chemicals under pressure should be determined by using ISO 13994. These International Standards are listed in the Bibliography.

It has been assumed in the drafting of this International Standard that the execution of its provisions will be entrusted to appropriately qualified and experienced people with a sound understanding of analytical chemistry. Appropriate precautions should be taken when carrying out this type of testing in order to avoid injury to health and contamination of the environment.

NOTE Finished items of protective clothing include gloves, arm shields, aprons, suits, hoods, boots, etc. The phrase “specimens from finished items” encompasses seamed and other discontinuous regions as well as the usual continuous regions of protective clothing items.

Protective clothing — Protection against chemicals — Determination of resistance of protective clothing materials to permeation by liquids and gases

1 Scope

This International Standard describes laboratory test methods to determine the resistance of materials used in protective clothing, including gloves and including footwear, when the footwear is an integral part of the clothing, to permeation by liquid or gaseous chemicals under the conditions of either continuous or intermittent contact.

Method A is applicable to testing against liquid chemicals, either volatile or soluble in water, expected to be in continuous contact with the protective clothing material.

Method B is applicable to testing against gaseous chemicals expected to be in continuous contact with the protective clothing material.

Method C is applicable to testing against gaseous and liquid chemicals, either volatile or soluble in water, expected to be in intermittent contact with the protective clothing material.

These test methods assess the permeation resistance of the protective clothing material under laboratory conditions in terms of breakthrough time, permeation rate and cumulative permeation. These test methods also enable qualitative observations to be made of the effects of the test chemical on the material under test.

These test methods are only suitable for measuring permeation by liquids and gases. Permeation by solid challenge chemicals is beyond the scope of this International Standard.

NOTE It can be difficult or impossible to normalize the results of permeation tests carried out against solid challenge chemicals. The normalized rate of permeation is dependent on the area of fabric exposed to the challenge chemical. In the case of solids this will, in turn, depend also on factors such as particle size, size distribution, particle shape and packing considerations.

These test methods address only the performance of materials or certain materials' constructions (e.g. seams). These test methods do not address the design, overall construction and components, or interfaces of garments, or interfaces between garments and gloves or garments and footwear, or other factors which may affect the overall chemical protection offered by protective clothing, gloves or footwear or combinations of chemical protective clothing, gloves and footwear.

It is emphasized that these tests do not necessarily simulate conditions to which materials are likely to be exposed in practice. In most cases the conditions of the permeation test will be far more challenging than expected workplace conditions.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 13994, *Clothing for protection against liquid chemicals — Determination of the resistance of protective clothing materials to penetration by liquids under pressure*

3 Terms and definitions

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

3.1
analytical technique

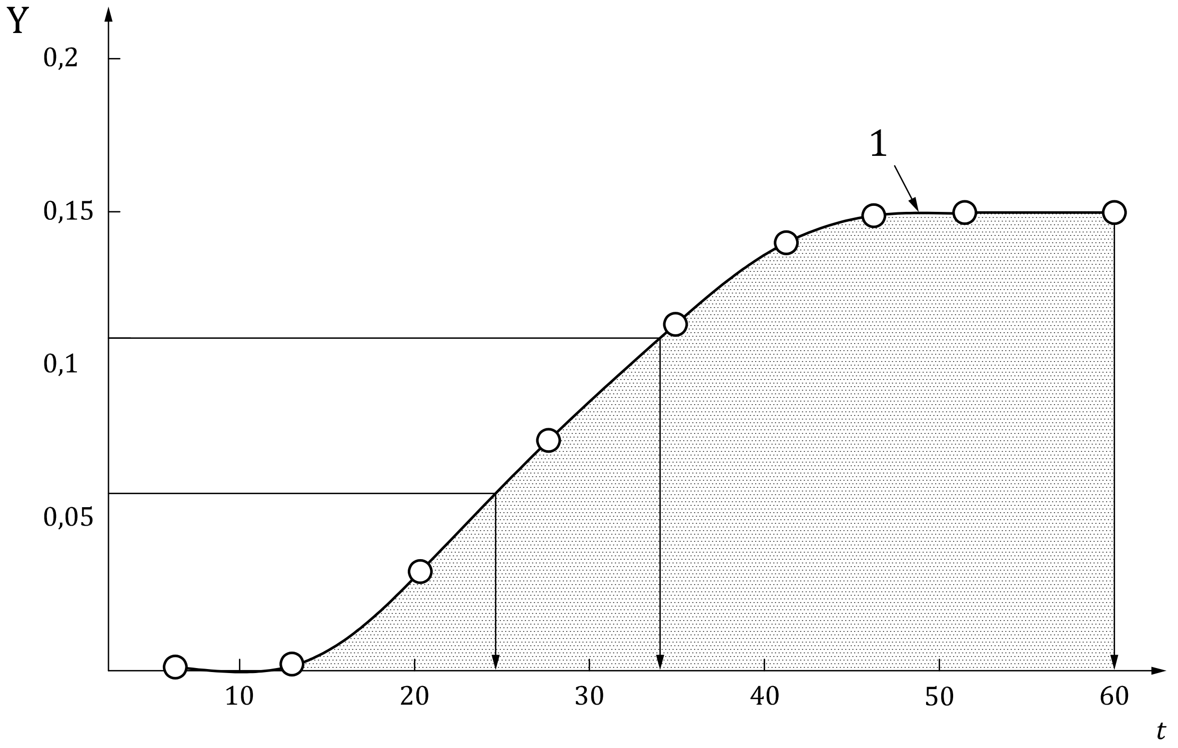
method of quantifying the amount of permeated chemical in the collection medium

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

EXAMPLE Applicable analytical techniques can include ultraviolet (UV) and infrared (IR) spectrophotometry, mass spectrometry, pH measurement, ion chromatography, conductimetry, colourimetry, atmospheric analytical detector tubes and radionuclide tagging/detection counting. Although gas- and liquid-chromatography are separation techniques rather than detection methods they can be used in conjunction with suitable detectors to quantify the amount of permeated chemical in the collection medium.

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



Key

1 steady-state permeation (the circles represent actual measured data-points)

Y permeation rate (µg/cm^2/min)

t time (min)

NOTE 1 The breakthrough detection time is dependent on the sensitivity of the method and the frequency of sampling of the collection medium.

NOTE 2 The breakthrough detection time for a method sensitivity of 0,05 µg/cm^2/min is 23 min but would be reported at 20 min, which corresponds to the last sampling time preceding breakthrough (since interpolation of data is not permitted). The normalized breakthrough detection time at a normalization permeation rate of 0,1 µg/cm^2/min is 33 min, but similarly would be reported at 28 min, which corresponds to the preceding sampling time. The steady-state permeation rate is approximately 0,15 µg/cm^2/min.

NOTE 3 The cumulative permeated mass over a 60 min period is equal to the area of the shaded region under the graph.

Figure 1 — Schematic permeation graph showing actual and normalized breakthrough times and cumulative permeation mass

3.3

closed-loop

refers to a testing mode in which the collection medium volume is fixed and continuously circulated or recycled

Note 1 to entry: The collection medium volume may change slightly from sampling without replacement of the sampled collection medium.

Note 2 to entry: The closed-loop collection medium need not necessarily be completely physically enclosed. For example, liquid collection media may be open to the air in an expansion vessel or constant-pressure header vessel.

3.4

collection medium

liquid or gas on the inner “clean” side of the test sample in which any permeated chemical is collected

3.5

contact time

in an intermittent contact (Method C) test, the duration that the challenge-side chamber of the permeation cell contains test chemical during each cycle

3.6

cumulative permeation mass

total amount of chemical that permeates during a specified period of time since the start of the test

Note 1 to entry: Quantification of cumulative permeation enables the comparison of permeation behaviour under intermittent contact conditions with analogous data for tests carried out under continuous-contact conditions.

Note 2 to entry: The measurement of cumulative permeation may depend on the sensitivity of the permeation-test system.

3.7

cumulative permeation time

time at which the total quantity of chemical that has permeated through each square centimetre of fabric has reached a predetermined mass

3.8

cycle time

in an intermittent contact (Method C) test, the interval of time from the start of one contact period to the start of the next contact period

3.9

degradation

deleterious change in one or more physical properties of a protective clothing material

Note 1 to entry: Deleterious changes can be manifest as either an increase or decrease in a physical property. For example, if the protective clothing material has been embrittled an increase in puncture resistance may be observed.

3.10

minimum detectable mass permeated

smallest mass of test chemical that is detectable with the complete permeation-test system

Note 1 to entry: This value is not necessarily the intrinsic limit of detection for the analytical instrument.

3.11
minimum detectable permeation rate
lowest rate of permeation that is measurable with the complete permeation-test system

Note 1 to entry: This value is not necessarily the intrinsic limit of detection for the analytical instrument.

3.12
normalization permeation rate
permeation rate used for determining the normalized breakthrough detection time

Note 1 to entry: This test method provides two choices of normalization permeation rates: 0,1 μg/cm^2/min or 1,0 μg/cm^2/min, although Annex F prescribes the latter for tests carried out in support of CE certification.

3.13
normalized breakthrough detection time
time at which the permeation rate reaches the normalization permeation rate

Note 1 to entry: See Figure 1.

3.14
open-loop
testing mode in which fresh collection medium flows continuously through the collection chamber of the test cell and is not reused or recycled

3.15
penetration
flow of a chemical through closures, porous materials, seams and holes or other imperfections in a protective clothing material on a non-molecular level

3.16
permeation
process by which a chemical moves through a protective clothing material on a molecular level

Note 1 to entry: Permeation involves a) sorption of molecules of the chemical into the contacted (outside) surface of a material, b) diffusion of the sorbed molecules in the material, and c) desorption of the molecules from the opposite (inside) surface of the material into the collection medium.

3.17
permeation mass
quantity of test chemical that passes through the protective clothing material within a given time

3.18
permeation rate
quantity of test chemical that passes through a given exposed surface area of protective clothing material in a given time

Note 1 to entry: Permeation rate is usually expressed in the units micrograms per square centimetre per minute (μg/cm^2/min).

3.19
protective clothing material
any material or combination of materials used in an item of clothing for the purpose of isolating parts of the body from a potential hazard

Note 1 to entry: This includes chemical protective gloves and, when attached to chemical protective clothing, boots.

3.20
purge time
in an intermittent contact test (Method C), the time immediately following the termination of the contact time when the test chemical is removed from the challenge-side chamber and air or nitrogen is blown over the outside surface of the protective clothing material

3.21
steady-state permeation rate

constant rate of permeation that usually occurs after breakthrough when the chemical contact is continuous and all forces affecting permeation have reached equilibrium

Note 1 to entry: Steady-state permeation may not be achieved during the period for which permeation testing is conducted.

3.22
test chemical

challenge chemical

liquid or gas that is used to challenge the protective clothing material specimen

Note 1 to entry: The liquid or gas may be either one component (i.e. a neat liquid or gas) or have several components (i.e. a mixture or solution).

4 Principle

The protective clothing material specimen acts as a partition between one chamber of a permeation test cell, which contains the test chemical, and another chamber, which contains the collection medium.

The test chemical may be either a liquid or a gas. The protective clothing material specimen may contact the test chemical either continuously or intermittently depending on the choice of the method used.

The collection medium, which may be liquid or gas, is (either periodically or continuously) analysed quantitatively for its concentration of the challenge chemical. The amount of that chemical that has passed through the material specimen is recorded as a function of time after its initial contact with the material.

Different test configurations may be used depending on the choice of the test chemical, collection medium and conditions of the test.

The breakthrough detection time, normalized breakthrough detection time, permeation rate, and cumulative permeation of the test chemical may be determined by either graphical representation or appropriate calculations, or both.

5 Choice of analytical technique and collection medium

5.1 General

The combination of the analytical technique and the collection medium shall be selected to optimize sensitivity for the detection of the test chemical. Wherever possible the collection medium shall represent actual occupational conditions.

NOTE When in use, the inner face of chemical protective clothing fabrics is usually exposed to either air inside the garment, aqueous solutions from perspiration, or both. The use of, for example, ammonia or methane as collection media would not be appropriate since neither will be present inside the protective clothing during normal use. Use of such collection media may also affect the test results since these substances may permeate the test specimen from the collection side of the test cell.

5.2 Gaseous collection media

A gaseous collection medium is usually used under continuous flow conditions for the collection of permeating molecules that are capable of vaporizing from the inner surface of the fabric under the conditions of the test both quantitatively and in sufficient quantities for analysis. The gaseous collection medium shall be a gas or gas mixture which does not interfere with the detection of the test chemical and does not itself permeate or degrade the fabric under test. The quality of the gas supply shall be of sufficient consistency over the duration of the test that changes do not interfere with detection of the test chemical.

EXAMPLES Nitrogen or dry air.

If ambient air is used as a collection medium care shall be taken to ensure that the moisture content does not vary significantly during the test.

Helium and hydrogen shall not be used as collection media since these gases can permeate some plastics and elastomers, including gaskets and sealing-washers.

If reactive gases such as oxygen are used as collection media appropriate safety measures should be adopted.

5.3 Liquid collection media

A liquid collection medium is usually used for the collection of permeated molecules of low volatility that are soluble in the collecting medium under the conditions of the test in sufficient quantities for analysis. The liquid collection medium shall be water, an aqueous solution or another liquid which does not interfere with the detection of the test chemical and does not itself permeate or degrade the fabric under test.

NOTE There are circumstances under which the above criteria are mutually exclusive. For example, when testing a PVC fabric for resistance to permeation by an involatile isocyanate it will be found that the challenge chemical is insoluble in aqueous collection media and that the test fabric is readily permeated or degraded by virtually all non-aqueous liquids. Under such circumstances testing is, unfortunately, not possible.

If there is any doubt as to whether a liquid collection medium will degrade or permeate a test fabric then the test fabric shall first be tested for permeation resistance to the collection medium. In many cases this will be possible by open-loop testing using a gaseous collection medium. If any permeation or degradation is observed over an 8 h exposure then the collection medium shall be deemed to be inappropriate.

5.4 Other collection media

Other collection media such as solid sorbents may be used provided that suitable collection efficiencies are demonstrated for the test chemical being used. The efficiency of such collection media shall, wherever possible, be validated by comparison to similar tests using either a gaseous or liquid collection medium.

Care shall be taken to ensure that there is complete and continuous contact between the inner face of the fabric and the collection medium. This is particularly important when considering solid particulate collection media since the degree of contact will depend on particle size.

6 Apparatus

6.1 Thickness gauge, suitable for measuring thickness to the nearest 0,02 mm, as specified in ISO 2286-3, ISO 5084 or similar, to determine the thickness of each protective clothing material specimen tested.

NOTE 1 The purpose of this gauge is not to give a definitive measurement of fabric thickness but to highlight any inter-sample thickness variations. Permeation can be extremely sensitive to very minor variations in sample thickness.

NOTE 2 The thickness of the sample is not used in the calculation of any results in this test standard.

6.2 Analytical balance, capable of being read to the nearest 0,01 g.

NOTE The level of precision stated above is that necessary for measuring the mass per unit area of test fabrics. If mass is used in any procedures for the calibration of detection equipment it may be necessary to have a balance capable of being read to the nearest 0,0001 g

6.3 Permeation test cell, consisting of a two-chambered cell for bringing the test fabric into contact with the test chemical on the fabric's normal outside surface (clothing exterior) and with a collection medium on the fabric's normal inside surface (clothing interior).

NOTE 1 Annex B contains a list of suppliers of permeation test cells.

Test cells conforming to one of the diagrams and associated descriptions in Annex C are suitable but other designs are acceptable provided that they meet the following criteria.

- The area of the outer surface of the fabric that is exposed to the test chemical shall coincide with the area of the inner surface of the fabric which is exposed to the collection medium.
- The capacity of the challenge side of the apparatus shall be sufficiently large that volume and/or concentration of test chemical are not significantly diminished by permeation.
- The challenge side of the apparatus shall be so designed that the mass or flow of the test chemical does not apply undue force to the fabric under test.
- The collection side of the apparatus shall be so designed that the mass or flow of the collection medium does not apply undue force to the fabric under test.
- The difference in pressure between the two surfaces of the test sample shall not exceed 5 000 Pa.

NOTE 2 Undue pressure may cause the fabric to stretch thereby rendering it thinner and less resistant to permeation.

NOTE 3 If either the test chemical or the collection medium flows into and out of the apparatus the pipes into and out of the cell will need to be of sufficient internal cross-sectional area that no significant pressure is generated under flow conditions.

The apparatus shall be so designed that the outer surface of the test sample is in complete contact with the test chemical and the inner surface is in complete contact with the collection medium.

NOTE 4 The validity of the results can be affected by air bubbles in liquid collection media and liquid test chemicals resting against the fabric under test.

The collection side of the apparatus shall be so designed that the collection medium is thoroughly mixed and that aliquots of collection medium analysed for the presence of test chemical are representative of the whole of the collection medium.

NOTE 5 This can be achieved in a number of ways including physical agitation or turbulent flow of the collection medium.

The materials of construction of the apparatus shall be such that they do not alter the nature or composition of the test chemical, the collection medium or any of the test chemical that has permeated through the fabric.

The apparatus shall be so designed that the only way that test chemical can get into the collection medium is by first permeating through the test fabric.

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 sample and then leak into the collection side. This is particularly likely to happen by capillary action if one or both surfaces of the test fabric are made from a woven or non-woven textile.

6.4 Equipment for the collection medium, for either a gaseous or a liquid collection medium, capable of a collection medium flow-rate of five volume changes per minute for the collection chamber of the permeation test cell.

- **Pump** (if necessary).
- **Method of flow control**.
- **Piping or tubing**.

- **Permeation test cell** (see 6.3).
- **Analytical detector,** suitable for the test chemical.

6.5 Equipment for open-loop or closed-loop permeation testing, consisting of the components shown in Figures 2 and 3.

- **Pump** (if necessary).
- **Method of flow control**.
- **Piping or tubing**.
- **Permeation test cell**.
- **Means for stirring or agitating the collection medium** (when appropriate).

  NOTE When used, agitation or stirring of the liquid collection medium at a rate of 0,1 r/s has been found to be satisfactory for some permeation testing.

- **Analytical detector**, suitable for the detection and quantification of the test chemical in the collection medium.

6.6 Stopwatch, or **electronic timer**.

6.7 Constant temperature chamber, bath or room, used to maintain the permeation test cell within ± 1,0 °C of the nominal test temperature.

NOTE 1 For reasons of safety, chemical permeation testing is often carried out in laboratory fume-cupboards or in association with other fume-extraction equipment. If air conditioning equipment is used to maintain the test temperature, air flowing into the laboratory (to replace air and fumes extracted) may first need to be conditioned to the appropriate temperature.

If a water-bath is used to maintain the test temperature then steps shall be taken to ensure that water does not leak into either side of the test cell or wick into any internal or external textile component of the test specimen.

NOTE 2 This requirement can be achieved by first enclosing the test cell in a waterproof bag before the cell and bag are then immersed in the water-bath.

7 System configuration

The apparatus shall be arranged in either the open-loop or closed-loop configuration.

NOTE 1 The specific configuration is generally dependent on the method of collection in combination with the techniques used for detection of the test chemical or its component chemicals.

NOTE 2 Closed-loop testing may provide different results from open-loop testing as a consequence of differences in the system configuration and collection media. The rate of desorption of permeated molecules from the inner face of the test chemical can depend on the nature of the collection medium.

7.1 Open-loop

In the open-loop configuration, the collection medium flows (see NOTE 1) from a supply reservoir, through the collection side of the permeation test cell, to a detector where it is analysed for the presence of test chemical. After analysis the collection medium flows to waste.

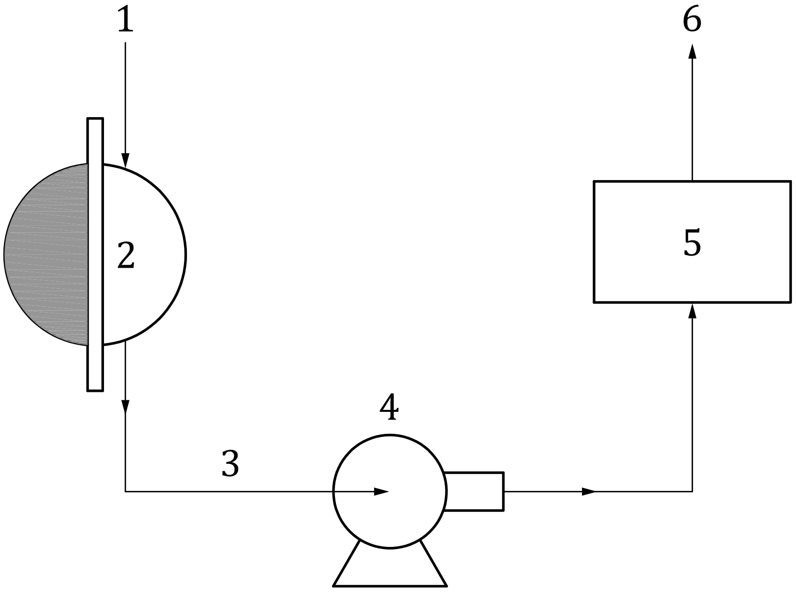
NOTE 1 The collection may flow under gravity (in the case of a liquid) under pressure (in the case of a gas) or may flow by the action of a suitable pump (as illustrated below).

NOTE 2 The open-loop configuration is typically used in conjunction with gaseous collection media when testing against volatile organic challenge chemicals.

The apparatus shall be selected, designed and configured so as optimize the test sensitivity. For the open-loop configuration this is usually best achieved by keeping the area of fabric exposed to the test chemical as large as practicable. In this regard, larger models of test-cell tend to give higher sensitivity.

The flow-rate of collection medium through the collection side of the test cell shall be sufficiently high that any molecules of test chemical permeating through the test fabric are rapidly mixed into the collection medium. The minimum flow-rate required to achieve this will depend on the exact design of the permeation test cell. A flow-rate equal to five times the volume of the collection side of the test cell per minute shall be used unless, for a specific design of test cell, it has previously been demonstrated that a lower flow-rate gives equally good mixing. In such cases mixing shall be deemed sufficient if a further increase in the degree of mixing does not alter the measured breakthrough time or steady-state permeation rate.

NOTE 3 Lower flow-rates result in lower dilution of permeated chemical and thereby increase minimum detection levels.



Key

1	fresh collection medium
2	sampling side for collection medium
3	collection medium sample
4	pump
5	sample analyser
6	waste

Figure 2 — Example configuration of open-loop permeation test equipment

NOTE 4 The pump may be either down-stream of the test cell (as shown in Figure 2) or up-stream.

7.2 Closed-loop

In the closed-loop configuration the collection medium flows from a supply reservoir, through the collection side of the permeation test cell, to a detector where it is analysed for the presence of test chemical. After analysis the collection medium flows back to the supply reservoir.

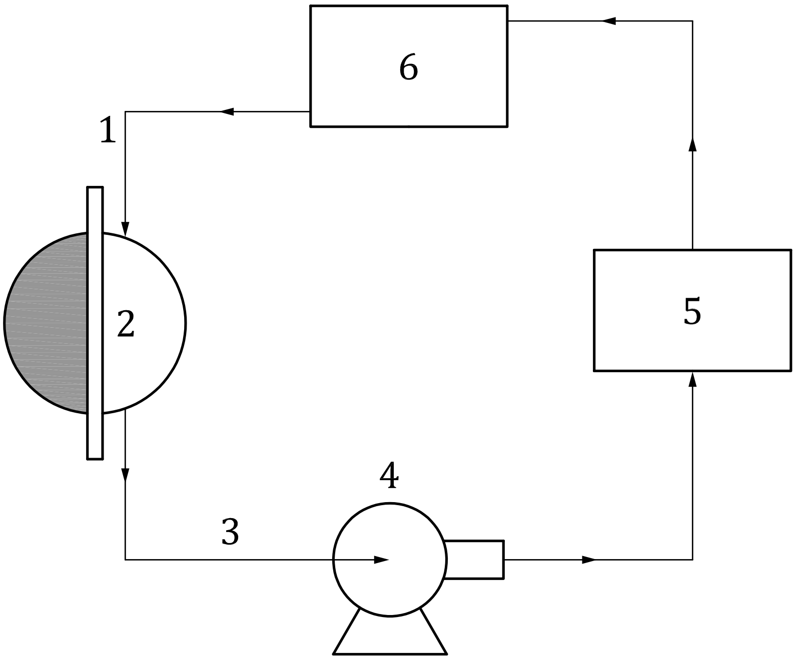
The apparatus shall be selected, designed and configured so as optimize the test sensitivity. For the closed-loop configuration this is usually best achieved by keeping the volume of collection medium in the loop as low as practicable. In this regard, smaller models of test-cell tend to give higher sensitivity.

NOTE 1 The closed-loop configuration is typically used in conjunction with aqueous collection media when testing against inorganic acid and alkali challenge chemicals.

NOTE 2 The use of a peristaltic pump for circulating a liquid closed-loop collection media can overcome problems of contamination from pump lubricants and corrosion of pump components that might otherwise be exposed to the collection medium. However, some designs of peristaltic pump can cause excessive pulsing of the flow of collection medium. Such pulsing can apply excessive pressure to the fabric under test.

The flow-rate of collection medium through the collection side of the test cell shall be sufficiently high that any molecules of test chemical permeating through the test fabric are rapidly mixed into the collection medium. The minimum flow-rate required to achieve this will depend on the exact design of the permeation test cell.

NOTE 3 A liquid flow-rate of 100 ml per minute has been shown to be sufficient for the commonly-used designs of permeation test cell.



Key

1	recycled collection medium
2	sampling side for collection medium
3	collection medium sample
4	pump
5	sample analyser (non-destructive)
6	collection medium reservoir

Figure 3 — Example configuration for closed-loop permeation test equipment

NOTE 4 The pump may be either down-stream of the test cell (as shown in Figure 3) or up-stream.

8 Detection

8.1 Frequency of analysis

8.1.1 In tests where the final result is to be expressed as a time, the frequency of analysis of the collection medium immediately before the time at which the final result is measured shall be as listed in Table 1.

Table 1 — Minimum sampling rates for collection medium

Final result	Minimum rate of sampling
≤ 10 min	Every 75 s
>10 min but ≤30 min	Every 150 s
>30 min but ≤60 min	Every 150 s
>60 min but ≤120 min	Every 6 min
>120 min but ≤240 min	Every 6 min
>240 min but ≤480 min	Every 11 min
>480 min	At least one measurement after 8 h

NOTE 1 In cases where the result cannot reliably be estimated more frequent analysis may be prudent.

NOTE 2 The values of 75 s, 150 s, 6 min and 11 min are intended as maximum permissible values of nominal 60 s, 120 s, 5 min and 10 min sampling frequencies.

8.1.2 In closed-loop tests where the final result is to be expressed as a cumulative mass at a predetermined time, the collection medium need only be analysed once at the predetermined time. It may, however, be prudent to make a number of measurements around the predetermined time.

8.1.3 In open-loop tests where the final result is to be expressed as a cumulative mass at a predetermined time, the collection medium shall be analysed at at least the minimum frequencies prescribed in Table 1 until permeation is detected. Once permeation has been detected the collection medium shall be analysed at least every 75 s.

8.1.4 In cases in which sampling and analysis of the collection medium are not continuous, results shall not be inferred by interpolation between discrete data-points.

NOTE The graph of permeation rate or mass versus time is seldom linear and therefore interpolation is likely to be unreliable and liable to give misleading results.

8.2 Analytical methods

Any analytical method may be used provided that:

- it is suitably sensitive to the test chemical;
- it is either not significantly sensitive to the collection medium or any such sensitivity can be accurately determined and discounted;
- 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 fabric.

NOTE For example, when testing "powdered" gloves the analytical method should not be sensitive to powders applied to the insides of such gloves during manufacture in order to facilitate donning

It is desirable that the analytical method should also provide some confirmation of the identity of the substance permeating into the collection medium. Such confirmation may, for example, be in the form of chromatographic retention time, absorption frequency, or may reasonably be inferred by the selective nature of the detection method, for example ion-selective electrochemistry. Confirmation of the chemical permeating into the close loop is particularly important in cases where the test chemical degrades the fabric under test. This can lead to the release of degradation products into the closed loop before breakthrough of the challenge chemical occurs.

Some test chemicals can be present in two or more equilibrium states. For example, when in aqueous solution weak organic acids are present in dissociated and undissociated forms. When quantitatively analysing for such chemicals due account shall be taken of such equilibria. For example, when detecting weak organic acids by ionic conductivity account shall be taken of the fact that the conducting species are in dynamic equilibrium with a non-conducting molecule.

Some detection methods involve the partial or complete destruction of the detected molecule. For example, flame ionization detection (FID) involves the combustion of the detected chemical. If such detection methods are used in conjunction with closed-loop tests account shall be taken of the amount of detected chemical that is lost.

Closed-loop detection methods may involve the removal of aliquots of collection medium for analysis. Depending on the analytical technique used such aliquots may or may not be returned to the closed loop after analysis. Account shall be taken of any reduction in volume of the closed loop, and also of any reduction in the amount of test chemical in the collection medium.

9 Sampling

9.1 Sampling procedure

9.1.1 Clothing samples may be taken from:

- specific areas of the suit prescribed by a relevant product-performance standard;
- the fabric of whole garments;
- sheets of garment fabric which has not been manufactured into a garment;
- seams cut from whole garments;
- sample seams in garment fabric which has not been manufactured into a (whole) garment.

When taking samples from whole garments, the specimens taken shall be representative of the whole garment. In the case of multi-layer constructions specimens shall include all layers arranged in proper order with the exception that readily-permeable layers on either the inner or outer face of the composite may be omitted in order to facilitate a better seal between the fabric and the test cell. If such layers are removed this shall be reported.

If, in the design of an item of protective clothing, different materials or thicknesses of material are specified at different locations, specimens shall be selected from each location.

Each material specimen shall be cut to an appropriate size and shape consistent with the size and shape of the test cell.

A minimum of three test specimens shall be taken at random from each protective clothing material, composite, seam, area (in the case of heterogeneous design) or other condition. More samples may need to be taken in accordance with 10.1. If desired, random specimens may be taken as described in ISO 2859-1.

9.1.2 Boot samples may be taken from:

- specific areas of the boot prescribed by a relevant product-performance standard;
- the thinnest topologically flat part of the shank;
- flat sheets of boot fabric of a thickness corresponding to the minimum thickness of the finished boot.

9.1.3 Glove samples may be taken from:

- specific areas of the glove prescribed by a relevant product-performance standard;
- the glove palm;
- the back of the glove;
- the wrist area of the glove.

9.2 Preparation of test specimens

Prior to testing, each test sample shall be brought to within ± 3 °C of the temperature at which the test is to be carried out.

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

NOTE 2 It is intentional that no conditioning humidity is specified. Once the test has commenced the sample will be exposed to either 100 % humidity (in the case of aqueous test chemicals and collection media), 0 % humidity in the case of non-aqueous test chemicals and collection media, or both.

NOTE 3 Other specific conditioning or preconditioning regimes are not precluded.

9.3 Measurement of test specimen thickness and mass

The thickness of each specimen shall be measured to the nearest 0,02 mm.

The mass of each specimen shall be measured to the nearest 1 g/m^2.

NOTE 1 Methods for measuring the thickness can be found in ISO 2286-3 and ISO 5084. Methods for measuring mass can be found in ISO 2286-2 and ISO 3801.

NOTE 2 In the case of fabrics in which the a protective film is laminated to a textile it may not be possible to determine whether inter-sample thickness variation is the result of variation in film thickness or textile thickness.

10 Procedure

10.1 Pre-screening

A sample of the protective clothing material shall first be tested according to ISO 13994, Procedure A, using water as the challenge liquid. This pre-test is carried out in order to determine whether the material is resistant to liquid penetration.

NOTE 1 It has been found that protective clothing materials which are not resistant to liquid penetration can give variable permeation test results.

If the fabric is shown to be resistant to liquid penetration then a set of three replicate permeation tests shall be carried out. If the fabric has not been shown to be resistant to liquid penetration then a set of five replicate permeation tests shall be carried.

NOTE 2 A greater number of replicate tests is carried out on non-penetration-resistant fabrics in order to better characterize potentially variable permeation performance.

10.2 Calibration

The response of the analytical detection system to the test chemical shall be calibrated. In the case of open-loop tests, the minimum quantification limit shall be equivalent to a rate of permeation of < 0,05 µg/cm^2/min and, if cumulative permeation is to be reported, the maximum quantification limit shall be equivalent to a rate of permeation of > 10 µg/cm^2/min. In the case of closed-loop tests, the minimum quantification limit shall be equivalent to < 0,05 µg/cm^2 and the maximum quantification limit shall be equivalent to > 500 µg/cm^2. If, for any reason, these limits cannot be achieved the fact shall be reported along with the actual quantification limits of the system.

NOTE 1 A method for determining the minimum quantifiable permeation rate is presented in Annex D.

NOTE 2 The sensitivity of the test method in detecting low permeation rates or amounts of the test chemical that permeate is determined by the combination of the analytical technique and the collection system selected as well as the ratio of the material specimen area to the collection medium volume (for closed-loop tests) or flow-rate (for open-loop tests).

NOTE 3 A sensitive analytical technique typically permits quantitative detection of the test chemical in the collection medium at volume or mass fractions as low as 10^{-7} %.

NOTE 4 Higher ratios of material specimen area to collection medium volume or collection medium flow rate permit earlier detection of breakthrough and detection of lower permeation rates. This is because higher concentrations of the test chemical in the collection medium will develop more rapidly once permeation starts.

10.3 Validation

The test system as a whole shall be routinely validated to ensure consistent performance over time. Validation shall be achieved by regularly repeating a test using the same test conditions, the same test chemical and a sample of the same test fabric.

NOTE Selection of a reliable combination of homogeneous validation test fabric and consistent test chemical will ensure that the validation data are meaningful.

10.4 Preparation of test apparatus

10.4.1 The first protective clothing material specimen shall be mounted in the permeation test cell according to 6.3 and the test cell shall be assembled. The apparatus shall be bolted or clamped together with sufficient force that leak-tight seals are achieved between the test specimen and both the test chemical and the collection medium.

10.4.2 Unless otherwise required by a product performance standard the test shall be carried out at a temperature of 23,0 °C ± 1,0 °C. Appropriate steps shall be taken to ensure that the permeation test cell, collection medium and test chemical are all within the specified temperature range before the test commences.

NOTE 1 This can be achieved by maintaining the permeation test cell, collection medium and test chemical at the test temperature, ± 1 °C for at least 30 min prior to testing.

NOTE 2 Permeation-resistance test results are significantly influenced by temperature. It should be noted that, when in use, protective clothing materials can be exposed to non-ambient conditions.

10.4.3 The collection medium shall be loaded into the collection chamber of the permeation test cell (the chamber to which the normal inside surface of the material specimen is exposed).

10.4.4 The collection medium shall be stirred, circulated or flowed continuously. In the case of liquid collection media, steps shall be taken to ensure that there are no air bubbles on the inner surface of the fabric sample.

NOTE 1 The purpose of agitating the collection medium is twofold:

- to ensure that it is homogeneous for sampling and analytical purposes, and
- to prevent or minimize concentration boundary layers of permeant at the interface of the clothing material and the collection medium.

The degree of agitation necessary to achieve these objectives is dependent on the permeation rate and the relative solubilities of the test chemical in the material under test and the collection medium. At this time, sufficient data are not available to specify minimum agitation rates. However, as guidance, in an open-loop system (i.e. collection medium flowing once through the cell, then being analysed and discarded), interlaboratory data suggest that an adequate degree of mixing is produced when the flow-rate is within the range specified in 7.1.

NOTE 2 For closed-loop systems with a liquid collection medium, adequate mixing levels can be determined by preliminary experiments, in which the rapidity of the dispersion of a dye is observed.

10.4.5 Analysis of the collection medium shall commence, either continuously or discretely, and shall be continued on a predetermined schedule according to the requirements of 8.1.

NOTE Sampling is carried out before the test chemical is added to the permeation test cell in order to establish the baseline values against which subsequent analytical data can be compared.

The method chosen for collection medium withdrawal shall be based on the technique selected for analytical detection. For example, UV or IR spectroscopy is often used for continuous analysis of a sample stream (although compounding and curing agents often used in protective clothing materials can interfere) while gas chromatography requires the analysis of discrete samples. When sampling the collection medium from open-loop tests, the flow of collection medium should never be interrupted. This will minimize adsorption of permeated chemical on the walls of the test cell and associated tubing.

In closed-loop systems with sample withdrawal, replenishment of the collection medium may be necessary in order to maintain a fixed ratio of collection medium volume to surface area of the test specimen in contact with the collection medium.

In cases where samples are continuously or repeatedly withdrawn, analysed, and returned to the test cell, no provision for volume maintenance is necessary.

10.5 Method A — Liquid chemicals with continuous contact

10.5.1 The liquid chemical shall be loaded into the challenge chamber of the permeation test cell (the chamber to which the normal outside surface of the material specimen is facing). Loading shall be done as quickly as practicable so that the time of first exposure of all parts of the test fabric shall be almost the same. The challenge chamber of the test cell shall be so filled that the entire area of the outer surface of the specimen is in contact with the test chemical. The timing of the test shall start when the addition of the liquid commences.

NOTE If the density of the test chemical is high, and the strength of the protective clothing material specimen being tested is low (for example, a thin latex film) the mass of the liquid may distort the specimen, leading to an increase in sample area. In such cases, steps may be taken to reduce the volume of test chemical provided that the specimen is completely covered with test chemical throughout the test period.

10.5.2 The concentration of the test chemical found in the collection medium shall be measured as a function of the time elapsed since the start of the test according to 10.4.5.

10.5.3 Sampling shall be discontinued and the test shall be terminated after one or more of the following conditions is met (see Figure 4):

- steady-state permeation is reached [Figures 4 a) and 4 e)];
- a normalized breakthrough time has been recorded;
- permeation reaches a steady-state permeation rate and then proceeds at an ever increasing rate [Figure 4 c)];
- a maximum rate is reached [Figures 4 b) and 4 d)];
- 8 h have elapsed or a pre-specified time has passed;

  NOTE In some cases the customer may request that a test be continued for more than 8 h or, conversely, may ask that the test be ended after less than 8 h.

— the rate of permeation is such that the test chemical might overload the detection system or severely contaminate the collection or test apparatus

10.5.4 After the test the apparatus shall be disassembled and cleaned as necessary.

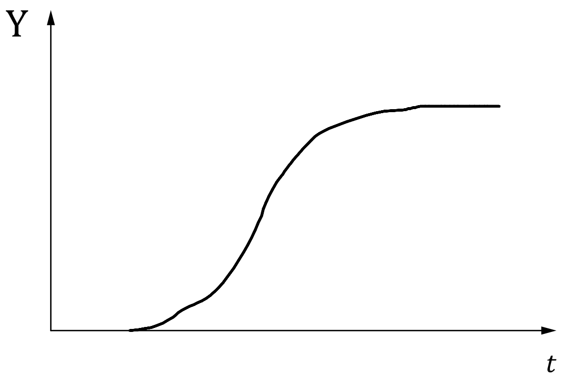
10.5.5 A number of replicate protective clothing material specimens shall be tested according to 10.1.

10.6 Method B — Gaseous chemicals with continuous contact

10.6.1 The flow of the test gas into the challenge chamber of the permeation test cell (the chamber to which the normal outside surface of the material specimen is facing) shall be commenced. Timing of the test shall begin once the equivalent of five chamber volumes of gas have passed through the chamber as determined by means of a rotameter or other flow monitoring device placed in the inlet stream to the chamber. The five volumes of gas shall be passed through the chamber within approximately 1 min. Following this initial period, the gas flow-rate may be reduced to a minimal level sufficient to ensure that the test sample is always in contact with the test gas and that no air can leak back into the challenge chamber. If the test is to be carried out at a non-ambient temperature, the challenge gas shall be brought to the non-ambient temperature before it enters the test cell.

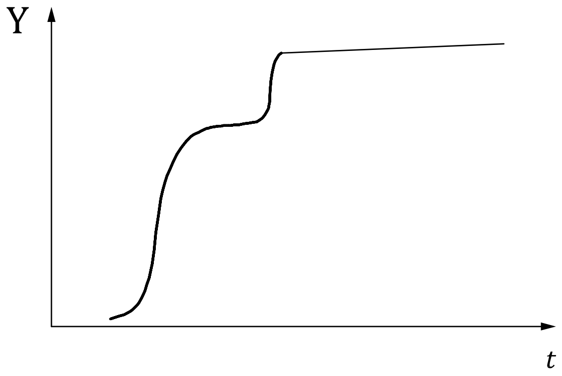
Care shall be taken so as not to pressurize the challenge or collection chamber. Overly high pressures may develop at high gas flow-rates or as a result of attachments that restrict the flow of gas from the chamber. Tightly-packed activated carbon beds or highly restrictive sparger tubes are examples of such attachments.

10.6.2 The concentration of the test chemical found in the collection medium shall be recorded as a function of time since the test was started according to 10.4.5.

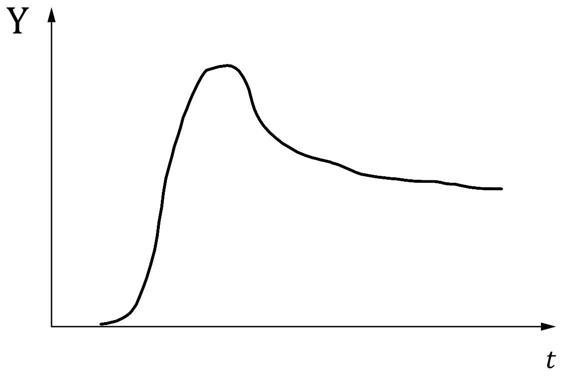


a) Most-typical type of permeation behaviour where the permeation rate stabilizes at a "steady-state" value

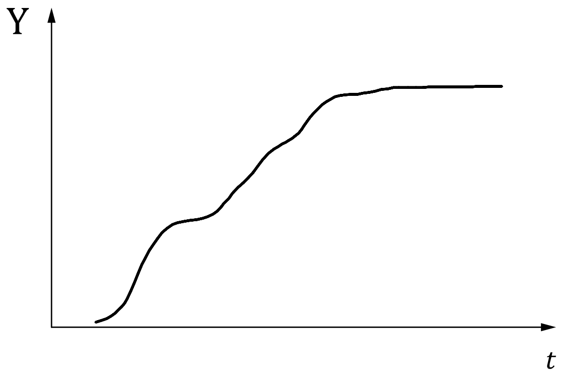
b) Type of permeation behaviour due to the material specimen being structurally modified by the chemical resulting in an increase or decrease in permeation rate



c) Type of permeation behaviour which occurs when the material specimen exhibits a sudden, very large increase in rate



d) Type of permeation behaviour which occurs when there is moderate to heavy swelling of the material specimen although the permeation rate eventually stabilizes



e) Type of permeation behaviour which occurs when there is a high degree of swelling

Key

Y	permeation rate (μg/cm^2/min)
t	time (min)

NOTE 1 Other types of permeation behaviour are possible and not uncommon.

NOTE 2 See Reference [7].

Figure 4 — Five types of permeation behaviour

10.6.3 Sampling shall be ceased and the test terminated after one or more of the following conditions has been met (see Figure 4):

— steady-state permeation is reached [Figures 4 a) and 4 e)];

— a normalized breakthrough time has been recorded;

permeation reaches a steady-state permeation rate and then proceeds at an ever increasing rate [Figure 4 c)];

a maximum rate is reached [Figures 4 b) and 4 d)];

8 h have elapsed or a pre-specified time has passed;

the rate of permeation is such that the test chemical might overload the detection system or severely contaminate the collection or test apparatus.

10.6.4 After the test the apparatus shall be disassembled and cleaned as necessary.

10.6.5 A number of replicate protective clothing material specimens shall be tested according to 10.1.

10.7 Method C — Liquid or gaseous chemical with intermittent contact

10.7.1 For Method C tests the challenge chemical, either gaseous or liquid, is applied to the test sample intermittently according to one of the schemes itemized in Table 2. At the start of the test the test sample is exposed to the challenge chemical in accordance with either Method A (for liquids) or Method B (for gases). The challenge chemical is then left in contact with the sample for the specified contact time (see Table 2), after which the challenge chemical is removed and the challenge side of the test cell is flushed with air or an inert gas. Flushing is continued for the specified purge time. This sequence is then repeated for the number of cycles specified.

10.7.2 For liquid challenge chemicals, the chemical may be removed from the cell by pouring or draining the test chemical from the challenge chamber through the inlet or outlet ports. Complete removal of viscous or tacky liquids may not be possible. Incomplete removal is acceptable since in the work environment complete removal of such chemicals is not likely.

While pouring the challenge chemical from the test cell care should be taken not to inadvertently disconnect pipe-work connecting the collection side of the test cell to the detector or pump.

10.7.3 For gaseous challenge chemicals, chemical may be removed from the cell by stopping the flow of challenge gas and then starting the flow of purge gas.

10.7.4 Purging shall be effected by causing dry air or an inert gas to flow through the challenge side of the test cell at a rate of 10 ± 1 chamber volumes per minute. The requirements for the purge gas shall be the same as for gaseous collection media.

10.7.5 The test shall continue for the specified number of cycles unless any of the conditions of 10.4.2 (for liquids) or 10.5.3 (for gases) has been met.

Table 2 — Recommended test conditions for intermittent contact testing

Method	Contact time	Purge time	Number of cycles
	min	min	
C1	1	10	11
C2	5	10	8
C3	10	60	2

10.7.6 The concentration of the test chemical found in the collection medium shall be measured as a function of the time elapsed since the start of the test. The minimum frequency of sampling shall be every 75 s throughout the test.

NOTE The sampling schedule described in 8.1 is not used in Method C because it has been found that less-frequent sampling can give misleading results.

10.7.7 A number of replicate protective clothing material specimens shall be tested according to 10.1.

10.8 Calculation of results

Results shall be expressed in one or more of three ways: normalized breakthrough time, cumulative mass of test chemical that has permeated over a set period of time, or the time taken for a predetermined mass of test chemical to permeate. The method of expressing the results, and the exact details of the reported calculation, will depend on the reason for carrying out the test and/or the product standard which the protective clothing is intended to meet.

The raw data are likely to be in one of the following forms:

- quantity of permeated chemical versus time;
- concentration of permeated chemical versus time (usually for closed-loop tests);
- rate of permeation versus time (usually for open-loop tests).

10.8.1 Normalized breakthrough time

This is the time at which the rate of permeation reaches a predetermined reporting criterion (for an example of data for calculation, see Table 3). The data shall be transformed into the form of rate of permeation (measured in µg of chemical per cm^2 of fabric exposed to the test chemical during the test per minute) versus time (measured in minutes).

NOTE 1 A computer spreadsheet can be used for this purpose.

The time at which the rate of permeation first exceeds the normalized breakthrough criterion shall be determined. This criterion will usually be either 0,1 $\mu g/cm^2/min$ or 1,0 $\mu g/cm^2/min$.

NOTE 2 This may either be achieved by inspection of the data or by graphical means.

The time of the collection medium analysis immediately preceding the time at which the permeation rate first reaches the normalized breakthrough criterion shall be recorded.

NOTE 3 This is because interpolation of data can be unreliable and is therefore not permitted

EXAMPLE Closed-loop test with a collection-medium volume of 100 ml.

Area of fabric exposed to the test chemical is 5 cm^2.

Normalized reporting criterion is 0,1 $\mu g/cm^2/min$.

Table 3 — Example data for calculation of normalized breakthrough time

Time of analysis	Concentration of test chemical in closed loop	Mass of test chemical in closed loop	Total mass of test chemical that has permeated unit area of fabric	Rate of permeation
min	µg/litre	µg	µg/cm^2	µg/cm^2/min
0	0	0	0	0
5	0	0	0	0
10	0	0	0	0
12	1	0,1	0,02	0,01
14	2	0,2	0,04	0,01
16	3	0,3	0,06	0,01
18	5	0,5	0,1	0,02
20	7	0,7	0,14	0,02
22	12	1,2	0,24	0,05
24	18	1,8	0,36	0,06
26	38	3,8	0,76	0,20
28	61	6,1	1,22	0,23
30	90	9,0	1,8	0,29

The rate of permeation first exceeds the reporting criterion at 26 min. The normalized breakthrough time is therefore reported as 24 min.

10.8.2 Cumulative permeation mass

This is the total mass of chemical that has permeated through each square centimetre of fabric during a predetermined time interval (for an example of data for calculation, see Table 4). The data shall be divided into discrete time intervals, each centred on the time of an analysis of the collection medium. The result of each analysis shall be transformed into the form of mass of chemical permeated during that time interval (measured in µg of chemical per cm^2 of fabric exposed to the test chemical).

NOTE A computer spreadsheet can be used for this purpose.

The total mass of chemical (in µg of chemical per cm^2 of fabric exposed to the test chemical) that has permeated during the predetermined time shall be calculated by summation of the individual amounts of chemical permeated during each time interval.

EXAMPLE Open-loop test with a flow-rate of 400 ml/min.

Area of fabric exposed to the test chemical is 20 cm^2.

Predetermined time interval is 30 min.

Table 4 — Example data for calculation of cumulative permeation mass

Time of analysis	Discrete time interval	Measured concentration of test chemical in collection medium	Rate at which test chemical is entering the flowing collection medium	Rate of permeation per unit area	Mass of chemical permeated each unit area during discrete time interval	Total mass of chemical permeated each unit area since start of test
min	min	µg/litre	µg/min	µg/cm²/min	µg/cm²	µg/cm²
0		0	0	0	0	0
1	1[a]	0	0	0	0	0
2	1	0	0	0	0	0
....	....	0	0	0	0	0
22	2[b]	0	0	0	0	0
24	2	0	0	0	0	0
26	2	8	3,2	0,16	0,32	0,32
27	1[c]	10	4,0	0,2	0,2	0,52
28	1	13	5,2	0,26	0,26	0,78
29	1	17	6,8	0,34	0,34	1,12
30	1	23	9,2	0,46	0,46	1,58

[a] Analysis carried out every minute for the first 10 min as per 8.1.1.

[b] Analysis carried out every 2 min after first 10 min as per 8.1.1.

[c] Analysis carried out every minute once breakthrough has been detected as per 8.1.3.

The total mass of permeated chemical over the predetermined test time of 30 min is therefore 1,58 µg/cm^2.

10.8.3 Cumulative permeation time

This is the time at which the total quantity of chemical that has permeated through each square centimetre of fabric has reached a predetermined mass (for an example of data for calculation, see Table 5). The data shall be divided into discrete time intervals, each centred on the time of an analysis of the collection medium. The result of each analysis shall be transformed into the form of mass of chemical permeated during that time interval (measured in µg of chemical per cm^2 of fabric exposed to the test chemical).

NOTE A computer spreadsheet can be used for this purpose.

Starting at the first time interval, the successive permeated masses for each time interval shall be added together to form a running total. The time at which the running total exceeds the predetermined mass shall be determined. The time of the analysis immediately preceding this time shall be recorded.

For example:

- closed-loop test with a collection medium volume of 120 ml;
- area of fabric exposed to the test chemical is 4,8 cm^2;
- predetermined cumulative permeation mass is 15 µg/cm^2;

Table 5 — Example data for calculation of cumulative permeation time

Time of analysis	Measured concentration of test chemical in collection medium	Total mass of test chemical to have permeated into the collection medium	Total mass of test chemical to have permeated unit area of fabric
min	µg/litre	µg	µg/cm^2
0	0	0	0
1	0	0	0
2	0	0	0
3	0	0	0
4[a]	0	0	0
5	0	0	0
6	1	0,12	0,025
7	2	0,24	0,050
8	5	0,6	0,125
9	11	1,32	0,275
10	23	2,76	0,575
11[b]	48	5,76	1,200
12	73	8,76	1,825
13	127	15,24	3,175
14	201	24,12	5,025
15	295	35,4	7,375
16	409	49,08	10,225
17	523	62,76	13,075
18	637	76,44	15,925
19	751	90,12	18,775

[a] Analysis carried out every minute for the first 10 min as per 8.1.1.

[b] Analysis carried out every minute once breakthrough has been detected as per 8.1.3.

The cumulative permeation mass first exceeds the reporting criterion at 18 min. The cumulative permeation time for 15 µg is therefore reported as 17 min.

10.9 Visual assessment of test specimen

At the end of the test each test specimen shall be visually inspected in a well-lit area (spectacles shall be used if necessary to ensure normal vision) and it shall be observed whether or not the test specimen has been changed in any way by its contact with the test chemical. This inspection shall be conducted both while the specimen is still “wet” and after it has dried since swelling may not be observed after the specimen has dried. If any change is observed the change shall be recorded.

NOTE Examples of changes include discolouration, flaking, swelling embrittlement and disintegration.

10.10 Repeat tests

10.10.1 The results shall be assessed to determine whether the result obtained from any one test specimen differs from the mean result by more than 20 %.

NOTE Experience in the development of this method indicates that this requirement is generally met, provided that the test specimens have been drawn from a material that is physically homogeneous and not subject to distortion or decomposition when in contact with the test chemical.

10.10.2 If the results do not vary by more than 20 % of the corresponding average for that set of data, a test report in accordance with Clause 11 shall be prepared.

10.10.3 If results do vary by more than 20 % from the mean for that set of data then a plausible reason for this shall be sought. If a reasonable explanation has been found, a test report in accordance with Clause 11 shall be prepared and the explanation shall be included into the test report.

NOTE For example, the test specimens may have been of significantly different thicknesses.

If a reasonable explanation has not been found, then a second set of test specimens shall be prepared and the testing shall be repeated under the same test conditions.

10.10.4 If the results obtained from the second set of test specimens do not vary by more than 20 % from the corresponding average for that set of data, a test report shall be prepared in accordance with Clause 11 based only on the second set of test data.

10.10.5 If the results from the second set of specimens do vary by more than 20 % from the average for that set of data then either 10.10.6 shall be followed or 10.10.7. 10.10.6 is effectively optional.

NOTE The decision whether or not to follow 10.10.6 may be taken by the test house, the organization that has commissioned the testing, or preferably by both after mutual consultation. In some cases it will be obvious, from inspection of the existing data, that the procedure described in 10.10.6 cannot alter the final result and is therefore unnecessary.

10.10.6 A third set of test specimens shall be prepared and the testing shall be repeated under the same test conditions. The three sets of results shall be combined and either the lowest (in the case of normalized breakthrough time or cumulative permeation time results) or highest (in the case of cumulative permeation mass results) test result shall be discarded.

NOTE This subclause makes allowance for the fact that when three sets of tests have been carried out a single anomalous result is unlikely to be representative of the performance of the fabric as a whole and is most probably a consequence of an undetected mistake in the execution of an individual test.

If the remaining results do not vary by more than 20 % from the average a test report shall be prepared in accordance with Clause 11 based only on the remaining (reduced) set of results. The value of the discarded result shall be noted in the test report but it shall be made clear in the report that this value was not used in the calculation of the overall result.

If the remaining results do vary more than 20 % from the average then 10.10.7 shall be followed.

10.10.7 The data from both sets of tests (if 10.10.6 was not followed), or the remaining data from three sets of tests (if optional 10.10.6 was followed) shall be combined. Either the highest (in the case of normalized breakthrough time or cumulative permeation time results) or lowest (in the case of cumulative permeation mass results) test result shall then be discarded and the average of the remaining results shall be calculated. If none of the remaining results varies more than 20 % from the average then a test report shall be prepared in accordance with Clause 11 based only upon the reduced set of results.

If any of the results does vary by more than 20 % from the average then the process of discarding either the highest (in the case of normalized breakthrough time or cumulative permeation time results) or lowest (in the case of cumulative permeation mass results) of the remaining test results shall be repeated iteratively until none of the remaining results varies by more than 20 % of the mean. A test report shall then be prepared in accordance with Clause 11 based only upon the remaining results (or in the extreme case, the only one result remaining after the iterative procedure, which will be the lowest of all single results in the case of normalized breakthrough time or cumulative permeation time results or the highest in the case of cumulative permeation mass results).

The values of all the discarded results shall be noted in the test report but it shall be made clear in the report that these values were not used in the calculation of the overall result.

NOTE In cases in which there is a bimodal distribution of test results the process of iteratively discarding the highest-performing test result ensures that the report is solely based on the lower-performing unimodal part of the distribution.

11 Report

The following information shall be included in the test report:

NOTE Annex F (which is normative) lists the specific default requirements, including reporting requirements, to be adhered to unless otherwise specified.

a) a reference to this International Standard, i.e. ISO 6529:2013;

b) the method used;

EXAMPLES Method A, Method C2.

c) the manufacturer's identification for the material being tested and a description of the sampling method used; i.e. report whether the material was taken from roll goods or garments, the type (fibre and coating compositions), supplier, lot number, and date of receipt of the material tested; if the material was taken from garments, report under subheadings for each material, composite, type of seam, or other conditions tested, and its position on the garment;

d) the thickness of each material specimen and the average thickness, expressed in millimetres, of the material tested;

e) a full description of any layers of a multi-layer fabric that have been removed in accordance with 9.1;

f) the mass per unit area, expressed in grams per square metre, of each material specimen and the average mass per unit area, expressed in grams per square metre, of the material tested;

g) the identity of the test chemical(s) used, its physical state, its concentration (in the case of a solution) and its grade or purity;

h) the test duration, expressed in hours;

i) the collection medium (including flow-rate and stirring, if applicable), system configuration (i.e. open-loop or closed-loop) and analytical technique used; when multiple test results are presented, difference in testing procedures between different reported chemicals shall be indicated;

NOTE It is important that the system configuration for all permeation testing be reported since differences in the system configuration can lead to significant differences in test results.

j) the cycle time, contact time, purge time, and number of cycles for intermittent permeation testing;

k) if the requirements of 10.2 have not been fully met, the minimum and maximum quantifiable permeation rates (when reporting breakthrough time) or the minimum and maximum detectable permeation masses (when reporting cumulative breakthrough mass or time);

l) the breakthrough detection time for each test specimen and the average for all specimens of a test set;

m) the normalized breakthrough detection time, cumulative permeation mass, or cumulative permeation time for each test specimen and the average for all specimens of a test set; the nomenclature for reporting normalized breakthrough time shall include a subscript indicating the permeation rate used for normalization of the breakthrough time;

EXAMPLE $BT_{0,1}$ or $BT_{1,0}$

n) if no permeation was detected or the permeation rate did not reach the selected normalization permeation rate for open-loop permeation testing (Method A) or the selected normalization permeation mass for closed-loop permeation testing (Method B), the fact that no permeation was detected shall be reported;

o) observations of material condition following contact with the test chemical.

For tests for which the results are expressed as normalized breakthrough time, or the time taken for a predetermined mass of test chemical to permeate the following shall also be reported:

- if the permeation process reached steady-state, the steady-state permeation rate shall be reported, expressed in micrograms per square centimetre per minute (μg/cm^2/min), for each material specimen; the average of the steady-state permeation rates for all replicates of a test set shall also be reported;
- the maximum permeation rate, expressed in micrograms per square centimetre per minute (μg/cm^2/min), for each specimen and the average permeation rate for all specimens in a test set;
- the plot of test chemical concentration, permeation rate, or cumulative permeation as a function of time for each specimen tested.

NOTE This test standard describes three different test methods and three sets of different reporting criteria. The customer, end-use, product specification or product standard may not require all of the various test results to be reported.

Annex A
(informative)

Recommended list of chemicals for comparing permeation resistance of protective clothing materials

A.1 Purpose

The purpose of this guide is to provide a recommended list of both liquid and gaseous chemicals for evaluating protective clothing materials in testing programs.

NOTE 1 Results derived from testing programs using these lists of challenge chemicals are not intended to give a definitive characterization of protective clothing materials.

NOTE 2 This list of challenge chemicals is not inclusive of all chemical challenges; the chemicals were chosen to represent broad ranges of liquid and gaseous chemicals. Not all chemical classes are represented. Other chemicals, especially those of interest to the manufacturer or user, should be tested in addition to those recommended in this guide.

A.2 List of recommended liquid test chemicals

Reagents are listed by common name, synonym, and Chemical Abstract Registry Service (CAS) number given in square brackets:

- acetone (2-propanone) [67-64-1],
- acetonitrile (cyanomethane, methyl cyanide) [75-05-8],
- carbon disulphide [75-15-0],
- dichloromethane (methylene chloride) [75-09-2],
- diethylamine [109-89-7],
- ethyl acetate [141-78-6],
- *n*-hexane [110-54-3], or *n*-heptane,
- methanol (methyl alcohol, carbinol) [67-56-1],
- sodium hydroxide (40 % by mass), r = 1,33 kg/l [1310-73-2],
- sulphuric acid (96 % by mass), r = 1,83 kg/l to 1,84 kg/l [7664-93-9],
- sulphuric acid (18 % by mass),
- tetrahydrofuran (THF, 1,4-epoxybutane) [109-99-9], and
- toluene (toluol) [108-88-3].

A.3 List of recommended gaseous test chemicals

Reagents are listed by common name, minimum purity, synonyms, and CAS number given in square brackets:

- ammonia, anhydrous, (99,99 %) [7664-41-7],

- chlorine, 99,5 % [7782-50-5],
- hydrogen chloride, (99,0 %) (hydrochloric acid) [7647-01-0].

A.4 Explanation of the recommended test chemicals

The chemicals listed above in A.2 and A.3 have been selected to provide a test battery that is representative of many of the chemicals that a chemical protective clothing wearer might expect to encounter. It should be emphasized that this is not a generic list taken from another application but a test battery specifically designed to challenge the permeation resistance of chemical protective clothing.

Chemicals listed in the test battery are typically the smallest molecules of their type except where a smaller molecule would be gaseous at normal temperatures and pressures or where smaller homologues spontaneously form dimers or trimers. In these cases the smallest liquid molecule has been chosen on the grounds that liquids are very much more concentrated than gases and that the permeation process is highly dependent on the challenge concentration.

Solids are not listed in the test battery because, with very few exceptions, solids do not permeate chemical barriers over the timescale in which chemical protective clothing is intended to be worn. There is also currently no reliable permeation test method for solids.

The breakthrough time of a chemical is in no way related to the degree of its toxicity. The test battery has been selected to test whether different types of chemical can permeate through a chemical protective fabric, not on the basis of the harm a substance might do to the wearer should permeation occur. The absence of well-known poisons from lists above should not therefore be interpreted as a deficiency in the test battery.

The choice of chemicals has been restricted to those substances against which chemical protective clothing might reasonably be expected to provide protection. Chemicals such as violently air sensitive reagents, unstable explosives and cryogenic liquids have not been considered since protection against these additional hazards is beyond the scope of most chemical protective clothing standards.

The battery consists of two groups of chemicals; worst case examples of chemicals that are known to permeate many chemical barriers, and some of the most common hazardous chemicals that are likely to be encountered by chemical protective clothing wearers. Some of the chemicals in the test battery fall into both categories. Good performance against all of the chemicals in A.2 and A.3 can normally be taken to infer that the fabric offers good all-round permeation resistance against most chemicals.

- **Acetone** – smallest of the ketones – very common industrial solvent
- **Concentrated sulphuric acid** – representative of strong and oxidizing mineral acids – common industrial acid
- **Dichloromethane** – smallest liquid chloroalkane – representative of halogenated solvents – readily permeates many chemical barriers
- **Toluene** – one of the smallest aromatic solvent molecules (benzene is slightly smaller but deemed too carcinogenic to be used for routine testing)
- **Diethylamine** – smallest and most aggressively alkaline liquid organic amine
- **Methanol** – smallest of the alcohol molecules
- **Acetonitril**e – smallest organic nitrile molecule – representative of nitrile monomers
- **Ammonia** – common industrial refrigerant gas
- **Hexane or heptane** – alkane – representative of petroleum fuels
- **Ethyl acetate** –most commonly encountered ester – used in large quantities as an industrial solvent

- **Hydrogen chloride** – representative of polar inorganic gases – common combustion product of some plastics and rubbers – given off by concentrated hydrochloric acid solutions used to clean and disinfect dairy equipment
- **Chlorine** – representative of the halogens – used extensively as a disinfecting agent for drinking and swimming-pool water treatment (fluorine is a smaller halogen molecule but this chemical is so violently reactive with such a wide range of materials that it is seldom encountered)
- **Carbondisulphide** – smallest liquid organic sulphide (thioformaldehyde trimerizes….)
- **Sodium hydroxide solution** – representative of aqueous solutions and strong alkalis
- **Tetrahydrofuran** – smallest of the liquid ether molecules – readily permeates many chemical barriers
- **18 % sulphuric acid** – representative of dilute non-oxidizing acids – a typical chemical against which Types 3 and 4 clothing might be expected to offer protection.

Annex B
(informative)

Sources of permeation test cells and permeation test cell parts

NOTE The products given in Annex B 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.

Sections of borosilicate glass pipe, available from Corning Glass, Catalog No. 72-0702 (1-in. length), or equivalent, are satisfactory for this purpose.

Flanges are available from Corning Glass, Catalog Nos. 72-9062 (aluminium) or 72-9654 (cast iron).

Gaskets are available from Corning Glass, Catalog No. 72-9256.

PTFE gasket material is available from W.L. Gore and Associates, Inc., Industrial Sealant Group, Elkton, MD 21921, USA.

Complete permeation test cells can also be purchased from:

Verre Equipements
3 rue des Quatres Chemins
69660 Collonges au Mont d’Or
France

Cambridge Glassblowing Limited
Brookfield Business Centre
Twentypence Road
Cottenham
Cambridge
CB24 8PS
England

Pesce Lab Sales Inc.
226 Birch Street
Kennett Square
PA 19348
USA

A.A. Pesce Glass Company Inc.
PO Box 1578
Hockessin
DE 19707
USA

LABC-Labortechnik
Müller & Zillger GbR
Josef-Dietzgen-Strasse 1
53773 Hennef
Germany

Annex C
(informative)

Designs and specifications of commonly-used permeation test cells

C.1 Permeation test cell for liquid test chemicals, capable of accommodating liquid chemicals, constructed from two end-fitting sections of straight glass pipe, each nominally sized to either a 25 mm or 51 mm diameter (see Figure C.1).

Materials other than glass may be used for tests involving chemicals incompatible with glass (e.g. hydrofluoric acid).

Permeation test cells made from glass should be arranged as follows.

- One end of each glass section should be sealed closed (for example, with a glass disk equivalent in quality to that of the glass of the original sections).
- The opposite end of each glass section should retain the "as-manufactured" flared end.
- Inlet and outlet ports with appropriate stopcock valves should be added to each glass section as shown.
- When assembled, the two glass sections should be joined horizontally by flanges and a gasket should be used at the joint. A second gasket may be used on the other side of the test specimens if necessary, to obtain a proper seal.

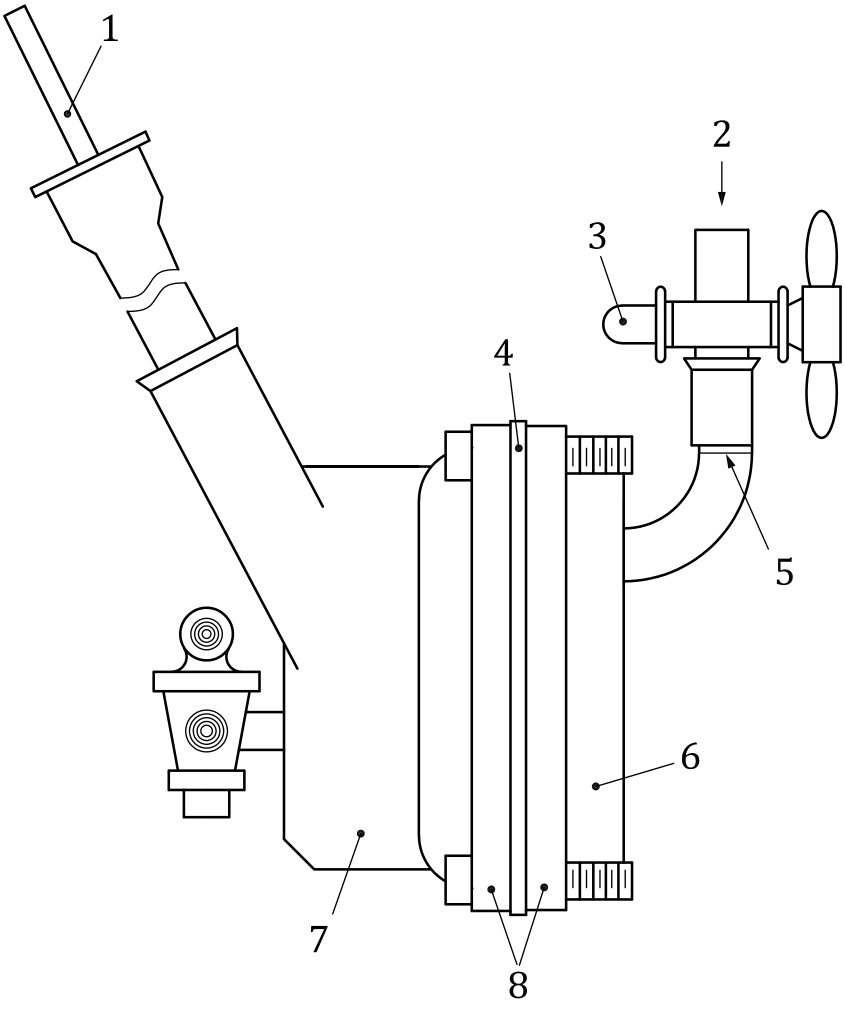
  EXAMPLE 1 PTFE gasket material.

- When carrying out tests against samples or items that are not flat (e.g. seams or closures) additional sealing materials may be necessary.

  EXAMPLE 2 Butyl sealant, round profile.

- In closed-loop tests where increased analytical sensitivity is required, a shorter length of glass pipe may be used to contain the collection medium. This reduces the contained volume and increases the ratio of material specimen area to the collection medium volume. In open-loop tests, lower collection medium flow-rates will increase the system sensitivity by lowering the minimum detectable permeation rate.

The challenge-side chamber may be modified to include an additional outlet port (with stopcock) positioned downward, opposite the liquid chemical inlet port. Such a modification will facilitate the repeated addition and removal of liquid test chemicals during intermittent testing.



Key

1	replaceable stirring rod	5	fill level
2	challenge chemical inlet	6	challenge side for test chemical
3	stop cock adapter	7	sampling side for collection medium
4	sample under test	8	aluminium flanges

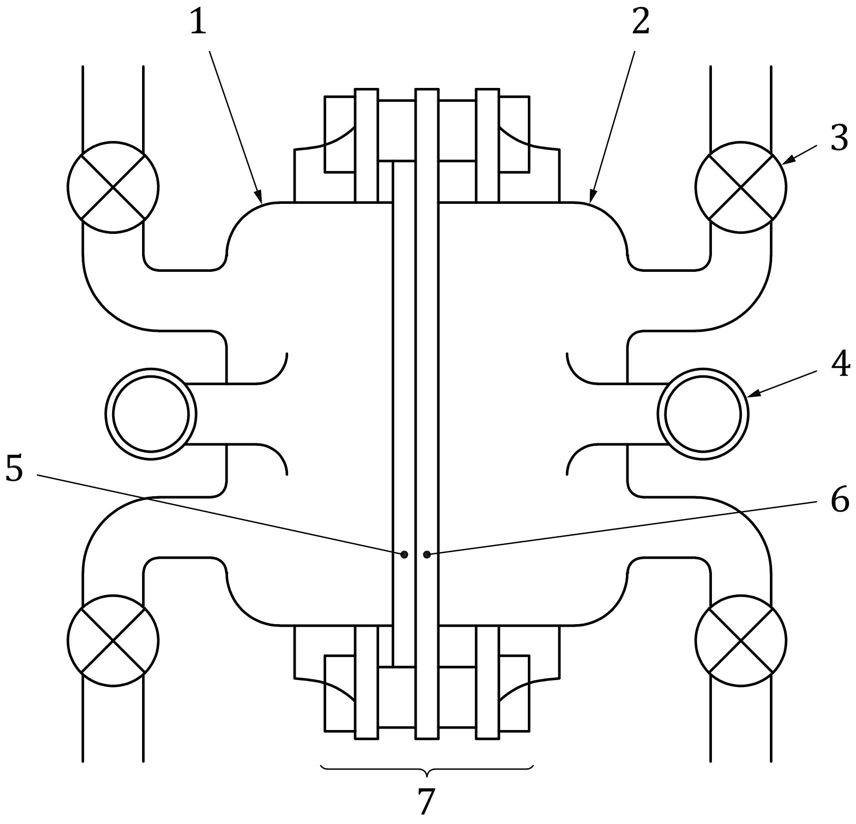
Figure C.1 — Permeation test cell for liquid test chemicals

The clothing material specimen is positioned between the flared ends of the two glass sections that compose the test cell. When the specimen is in place, the test cell is divided into two chambers.

NOTE Sources for permeation test cell parts are provided in Annex B.

C.2 Permeation test cell for gaseous test chemicals (see Figure C.2), capable of accommodating gaseous chemicals, identical to the liquid permeation test cell except that, with reference to Figure C.1, two collection medium sections are used. Thus, the gaseous chemical can be circulated from its reservoir through the challenge-side chamber. Flow should be such that the composition and concentration of the gas in the test chamber does not change with time and the test gas in the chamber is well mixed.

A specimen is positioned between the flared ends of the two glass sections that compose the test cell as shown in Figure 3. When the specimen is in place, the test cell is divided into two chambers.



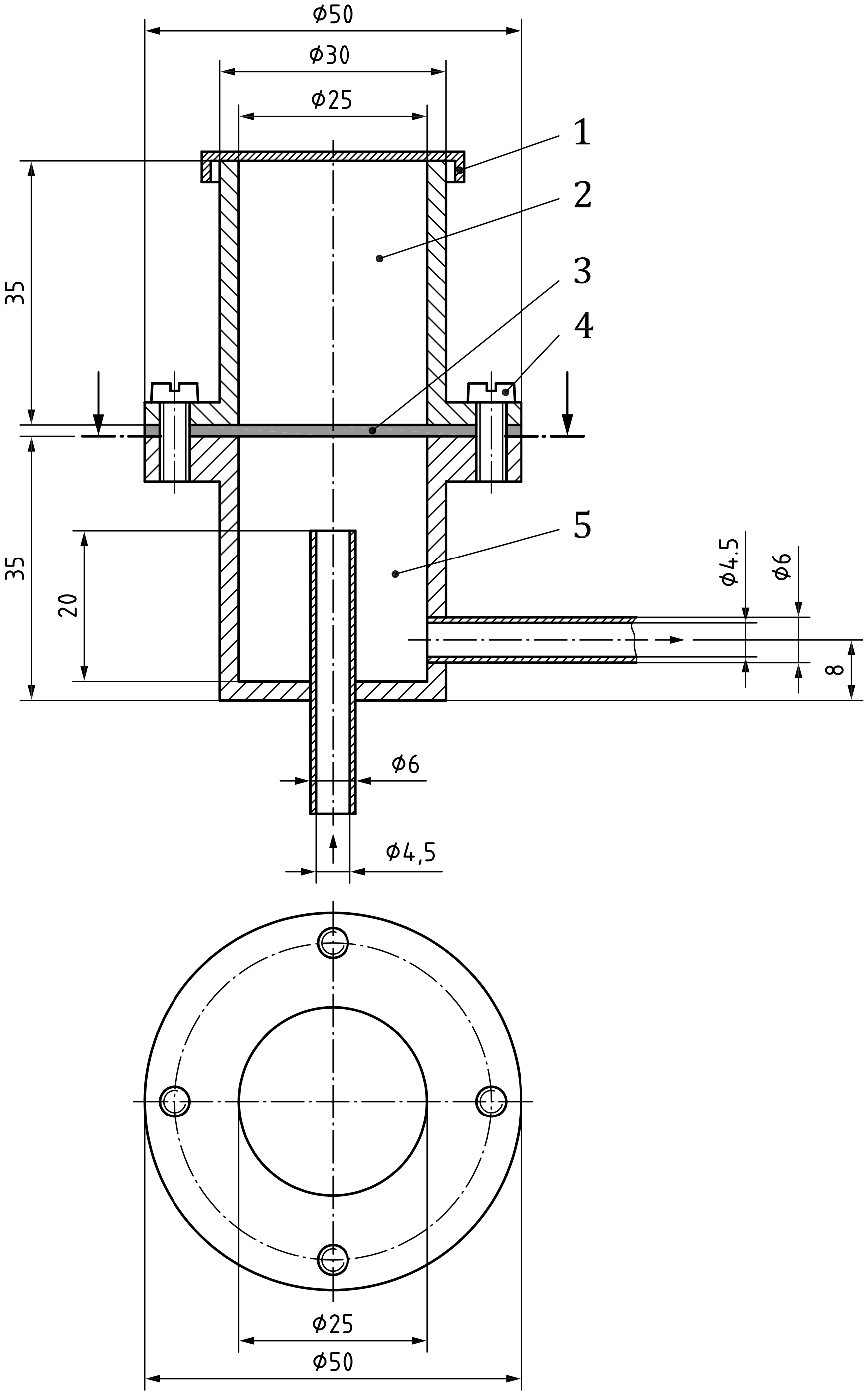
Key

1	sampling side for collection medium	5	protective clothing material specimen
2	challenge side for test chemical	6	gasket
3	valve	7	aluminium flanges
4	filling tub		

Figure C.2 — Permeation test cell configured for gaseous test chemicals

C.3 Alternative permeation test cells, for conducting permeation testing, see for example Figure C.3.

Dimensions in millimetres



Key

1 loose cover

2 liquid compartment

3 test material

4 screws

5 flow compartment

Exposed area of test material: 4,91 cm^2.

Volume of flow compartment: 17,2 cm^3.

Figure C.3 — Alternative permeation test cell

Annex D
(informative)

Suggested procedures for calibrating and measuring the sensitivity of permeation-test systems

D.1 General considerations

For any permeation-test system, regardless of configuration, apparatus, collection medium or challenge chemical, it is important that two parameters are quantified:

— the detector response as a function of challenge chemical concentration in the collection medium;

— the minimum concentration of challenge chemical in the collection medium that can unequivocally be quantified.

The way in which these parameters are determined will inevitably depend on a variety of considerations including things such as detection method, collection medium and challenge chemical. The purpose of this annex is to give general guidance on calibration and measurement of sensitivity. Techniques other than those discussed here may be equally suitable.

D.2 Calibration

D.2.1 Closed-loop systems

The calibration of closed-loop systems is usually straightforward. Solutions or mixtures of challenge chemical in collection medium should be prepared over a suitable range of concentrations. The detector response to these solutions should then be measured and recorded. Graphical or spreadsheet techniques can then be used to provide a graphical or numerical relationship between challenge chemical concentration and detector response.

The calibration solutions should be prepared over the range of concentrations at which the challenge chemical is expected to be encountered. This will depend on the volume of collection medium, the area of fabric exposed to the challenge chemical and the degree of permeation that is to be measured. Rather than working-out complex formulae for this, it is simpler to find the answers to a few simple questions:

— What is the minimum (maximum) amount of permeation that it is necessary to determine for this test?

— If that minimum (maximum) degree of permeation were to occur, what would be the concentration of challenge chemical in the collection medium?

For example, consider a test with a closed-loop volume of 100 ml and a test cell in which 5 cm^2 of fabric are exposed to the challenge chemical. According to 10.2 the minimum detectable permeation should be < 0,05 µg/cm^2. If 0,05 µg were to permeate each cm^2 of fabric then there would be 0,25 µg of chemical in the 100 ml of collection medium in the closed loop. The lowest concentration calibration solution should be at or below 0,25 µg chemical per 100 ml or 2,5 µg per litre. Similarly, 10.2 imposes a maximum detectable permeation of > 500 µg/cm^2. If 500 µg were to permeate each cm^2 of fabric then there would be 2500µg of chemical in the 100 ml of collection medium in the closed loop. The highest concentration calibration solution should at or above 2500 µg per 100 ml or 25 mg per litre.

It may be necessary to prepare and measure a range of solutions of intermediate concentration to determine whether the detector response is linear across the entire concentration range. In many cases it will be found that detector response is indeed linear at the low concentrations encountered in permeation testing. The measurement of intermediate calibration solutions will also give further experimental confidence in the calibration data. It is often possible to use the measurement of a solution of zero concentration as an additional calibration point.

Where a range of calibration solutions or mixtures are used care should be taken to ensure that each solution is prepared independently of the others and not by different dilutions of the same stock solution. The serial dilution of a single stock solution is unreliable since if the stock solution has been incorrectly prepared then subsequent correct dilution will only propagate this error. In such cases the calibration data will appear to be self-consistent but will be invalid.

D.2.2 Open-loop systems

One of the easiest ways to calibrate an open-loop system is to temporarily re-configure it as a closed-loop system. This may involve the use of a suitable re-circulating pump. If this is the case then steps should be taken to check that challenge chemical is not absorbed onto surfaces inside the pump. This may best be demonstrated by an observing detector for a given concentration of challenge chemical over a period of tens of minutes. If the response steadily falls then that can be indicative of loss of challenge chemical either through a leak or through absorption inside the pump.

Destructive open-loop detection methods (such as GLC or PID) cannot be reconfigured as closed-loop for calibration purposes since the detector response would fall as the challenge chemical is destroyed in the detector. Instead, such systems can be calibrated by flowing calibration solutions or mixtures through the system. This may involve the preparation of large volumes of calibration solutions or mixtures since the flow-rate through the detector may be high. In the case of gaseous collection media it may be necessary to purchase or prepare samples of “span gases” containing suitable known concentrations of the challenge chemical. Span gases can sometimes be prepared by filling a sealed chemical-resistant polymer bag with collection medium and then injecting a known volume challenge chemical so that it evaporates inside the bag. The contents of the bag can then be pumped through the detector.

The necessary concentrations of calibration mixtures can be calculated by answering the questions in D2.1. For example, consider an open-loop test with a flow-rate of 500 ml/min and a test cell in which 20 cm^2 of fabric are exposed to the challenge chemical. From 10.2, the minimum detectable permeation rate should be < 0,05 $\mu g/cm^2/min$. If 0,05 µg of chemical were to permeate each cm^2 of fabric over a period of one minute then a total of 1,0 µg of chemical would have permeated into the collection medium. During that one minute period 500 ml of collection medium would have flowed through the collection side of the test cell, so the 1,0 µg would be collected in 500 ml of collection medium. The lowest concentration calibration mixture should therefore be at a concentration of 1,0 µg per 500 ml, or 2,5 µg per litre. The maximum concentration calibration solution can be calculated in a similar manner. Once again, it would be advisable to prepare and measure the concentrations of intermediate calibration mixtures.

Where a range of calibration solutions or mixtures are used care should be taken to ensure that each solution is prepared independently of the others and not by different dilutions of the same stock solution. The serial dilution of a single stock solution is unreliable since if the stock solution has been incorrectly prepared then subsequent correct dilution will only propagate this error. In such cases the calibration data will appear to be self-consistent but will be invalid.

D.2.3 Theoretical calibrations

In some cases it may be possible to calculate the detector response by theoretical calculation. For example, at low concentrations the response of a conductivity cell can be calculated from the cell constant and a table of limiting ionic conductivities. It is, nevertheless, good practice to confirm such calculations by experimental measurements.

D.3 Determination of minimum sensitivity

D.3.1 Theoretical calculation

Once the system has been calibrated it is a relatively straightforward task to determine the minimum sensitivity of the whole system. Once again, rather than giving empirical formulae for this determination, it is simpler to work out the answers to a few questions:

— What is the average detector response when there is no challenge chemical in the collection medium?

— What are the minimum and maximum detector responses when there is no challenge chemical in the collection medium?

— By how much would the detector response have to vary for it to be obvious that there was a small amount of challenge chemical in the collection medium?

Ideally, the detector response should perhaps be zero if there is no challenge chemical in the collection medium, however it is usual to see a small “background” reading and this may fluctuate over time. For it to be obvious that there is a minimal concentration of challenge chemical in the collection medium the detector response must change from the background reading and distinctly beyond the range of normal fluctuations. By examination of the background readings over a period of time it is usually quite easy to estimate the magnitude of a minimally significant change. By reference to the calibration data this minimal change in detector response can be converted into a minimum detection level for the system.

For example, the average background reading of a detector may be 2 units but the reading may fluctuate randomly between −5 units and +9 units with rare minima and maxima at −7 and +11. The detector response varies in whole numbers of units. The lowest-concentration calibration solution (from D.2) gives a detector response of 50 units. Any reading less than 9 units is clearly within the “background noise”. It is possible that a response of 10 or 11 units might be significant, but this could still be “background noise” However a response of 20 units would clearly indicate the presence of challenge chemical in the collection medium.

D.3.2 Experimental validation

Once the minimum quantifiable permeation rate or permeation mass of the system has been estimated this can be confirmed by the measurement of a suitably dilute solution or mixture of the challenge chemical in the collection medium.

During the course of an actual permeation test lower minimum sensitivity levels can sometimes be inferred from an analysis of the trend of results. In the example given in D.3.2 the estimated minimum sensitivity may correspond to a detector reading of 20 units. A reading of 10 or 11 units might be significant, but equally such a reading might be part of the normal background. However, if after a reading of 11 units subsequent measurements are of 12, 14, 18, 22 and 27 units then it is apparent that permeation is taking place and that the reading of 11 units was probably significant after all. Although such analysis may be useful not every permeation test can be relied upon to yield such tractable data. In the above example very low steady-state permeation might only increase the background readings by 2 or 3 units. In such a case it would be impossible to determine when, or indeed whether, permeation had commenced.

D.4 Uncertainty of measurement

It is beyond the scope of this International Standard to give detailed information on the determination of uncertainty of measurement for various permeation test methods. Such information, and further training and advice, are often available from national accreditation bodies. However, two general guidance notes are given below.

— The overall uncertainty of a permeation test result is usually the combination of many other uncertainties in measurement of concentrations, calibration, preparation of calibration mixtures, calibration of volumetric glassware and syringes, measurement of collection medium flow-rates, measurements of time, etc. The easiest way to cope with this is often to take a systematic “bottom up” approach and build-up the final uncertainty from a “pyramid” of supporting uncertainty budgets.

- In many cases it will be found that permeation breakthrough is a sudden “catastrophic” event. Over a period of just 5 min the rate of permeation may rise from zero to 20 µg/cm^2/min and may keep rising. The calculated uncertainty in measurement of the normalized breakthrough rate might be as high as ±10 % or more, however if the permeation rate rises from 0,9 µg/cm^2/min to 1,1 µg/cm^2/min over a period of 90 s then the overall uncertainty of measurement of permeation breakthrough time is likely to be under ±1 min rather than ±10 % of the measured breakthrough time.

Statistical methods of determining uncertainty of measurement are outlined in ISO 5725-2.

A mathematical method of determining uncertainty of measurement may be found in ISO/IEC Guide 98-3.

It should be noted that no individual permeation test can ever be repeated. The test is effectively destructive since the test sample becomes permeated or irreversibly contaminated during the test. It is therefore not possible to assess the repeatability of the permeation test since the element of inter-sample-variation cannot be separated from repeatability.

Annex E
(informative)

Testing the permeation resistance of seams and closures

E.1 General

A number of product standards for chemical protective clothing require that seams and closures be tested for resistance to permeation without going into further details as to how this might be achieved. This annex describes the theoretical and practical problems encountered in testing such features and suggests some possible solutions.

E.2 Theoretical considerations

It is clearly important that seams and closures in chemical protective clothing give adequate levels of chemical protection; there is no point in having a highly permeation-resistant fabric if it leaks at the seams. At first sight it would therefore seem to be reasonable to subject seams and closures to the same regime of chemical permeation testing as the fabric. However, this approach is flawed for three inter-related reasons:

— The permeation test measures permeation per unit area of fabric exposed to the challenge chemical, but seams and closures are effectively one-dimensional or linear features.

— A seam or closure cannot be tested in isolation; any test on a seam will also necessarily include a test on the fabric on either side of the seam. The result will therefore be dependent on the relative proportions of linear seam or closure to planar fabric exposed to the challenge chemical in the test cell.

— The geometry of the standard permeation test cells is circular. The maximum length of seam or closure exposed to the challenge chemical will be equal to twice the cell radius. The area of fabric on either side of the seam or closure will be proportional to the square of the radius. The relative proportions of seam-length to fabric area are therefore dependent on the size of the test cell.

The consequence of these flaws is that the results of tests on seams and closures are strongly dependent on the size and geometry of the permeation test cell. This is illustrated in the following examples:

EXAMPLE 1 The seam in a test fabric is less permeation-resistant than the fabric itself. When tested in a standard nominal 1” diameter test cell the length of seam exposed to the chemical is 2,54 cm and the area of fabric exposed is 5,07 cm^2; a ratio of seam length to fabric area of 1:2. However, when tested in a standard nominal 2” test cell the length of seam exposed to the chemical is 5,08 cm and the area of fabric exposed is 20,27 cm^2; a ratio of seam length to fabric area of 1:4. In the nominal 1” test cell the proportion of less-permeation-resistant seam is higher, so the overall amount of permeation measured will be higher. In the nominal 2” test cell the proportion of more-permeation-resistant fabric is higher so the overall amount of permeation measured will be lower.

EXAMPLE 2 A sewn seam in a fabric with low resistance to permeation is covered with a 2,75 cm wide strip of highly-permeation resistant material. When tested in a nominal 1” test cell with the seam collinear with the cell diameter the only fabric in contact with the challenge chemical is the highly-permeation-resistant strip glued over the seam. The result will therefore be good. However, when the same seam is tested in a nominal 2” test cell some of the low-permeation-resistant fabric will be in contact with the challenge chemical and the result will therefore be not be so good.

EXAMPLE 3 A seam in a highly-permeation-resistant fabric is covered by a 2,75” strip of the same material. The adhesive bonding the two pieces of fabric together is soluble in the challenge chemical. When tested in a nominal 2” test cell the edge of the strip of fabric is exposed to the challenge chemical and the adhesive dissolves in the challenge chemical. The chemical then flows between the two pieces of fabric and penetrates the sewn seam. However, when tested in a nominal 1” test cell the adhesive is not in contact with the challenge chemical.

These examples demonstrate how the result of a permeation test on a seam cannot be normalized and how such results are sensitive to the size of the test-cell.

E.3 Practical considerations

From the above it is apparent that no single test protocol will necessarily determine the chemical resistance of a seam or closure. Different test procedures in different test laboratories will inevitably give different results in most cases. The following guidelines may help different laboratories take a more mutually-consistent approach to the testing of seams and closures.

- The seam or closure should be examined and, where necessary, a section should be dissected in order to reveal the seam construction.
- The seam or closure should be tested to reveal any inherent weaknesses. This may involve placing the seam off-centre in the test cell so as to test the chemical resistance of the adhesive used to attach any seam-stripping.
- Consideration should be given to testing the same design of seam or closure in different cells and with different alignments.
- However the seam or closure is tested, the details of the tests should be listed in the test report in sufficient detail that another test laboratory might replicate the tests.
- Any attempts to influence the way in which a seam or closure is tested, perhaps in order to obtain a particular result, should be strongly resisted. The test laboratory's decision on how to test a seam or closure form an integral part of the testing of such components.

On the more positive side, a highly-permeation-resistant seam or closure in a highly-permeation-resistant fabric will always give excellent permeation test results however they are tested. If there are circumstances under which a seam or closure might allow the ingress of chemicals then it is important that such failings are recognized and reported.

E.4 Experimental considerations

The standard permeation test cells were all designed for testing flat planar samples and do not readily accommodate the raised profiles encountered in seams and closures. It is, however, usually possible to seal most seams, and at least some closures into standard test-cells with one or more of the following modifications:

- The use of soft rubber washers below, and sometimes above, the test sample. Provided that the rubber is sufficiently soft, any raised profiles can be compressed into the rubber. Care should be taken to ensure that any washer does not become so distorted under compression that it reduces the area of fabric in contact with either the challenge chemical or the collection medium. Washers that have suffered prolonged contact with challenge chemicals should be discarded in case they cross-contaminate future tests.
- The use of uncured or partially-cured rubber sealants. These can be hand-made from solid sealant materials used in the automotive, construction or marine industries. These conform to the shape of the seam or closure and form an intimate seal. Again, care should be taken to ensure that excess sealant does not reduce the contact between the sample and either the challenge chemical or the collection medium.
- The use of fluid sealants. Examples of such sealants are silicone bathroom sealants, acrylic decorators' sealants and fast-setting jointing compounds. These can be applied as the test apparatus is assembled and then allowed to fully cure or set before the test is commenced. In addition to the caveats above, it is important to recognize that while curing some of these sealants can release solvents or acetic acid. These chemicals may affect the test sample or may interfere with the analysis of the collection medium.

- Casting samples in resin. In some cases it is possible to cast the sample in an annular-shaped section of a resin or resin composite. The resin covers the outer circumference of the sample but leaves the centre of the sample exposed. This effective forms a flat-surfaced washer above and below the test sample. The entire assembly can then be clamped into the test cell. The resin should be chosen so as not to interfere with the performance of the test.
- The use of profiled packing-pieces. These are thick washers that have been sculpted to match the profile of the test sample. This technique can be particularly useful when testing chemical-resistant zippers. The packing-pieces can be further sealed to the sample by the use of malleable or fast-setting liquid sealants.

When testing seams and closures it is to be expected that a significant number of tests will have to be aborted because of leaks of either challenge chemical or collection medium. It would be prudent to make available sufficient time, resources and test-samples to allow for this.

Annex F
(normative)

Default conditions for testing and reporting permeation resistance

F.1 General

This International Standard includes three permeation test methods, each with various options for testing, calculating and reporting normalized breakthrough time, cumulative permeation mass and cumulative permeation time. This annex prescribes the specific test methods, test conditions and reporting criteria that shall be used by default unless otherwise specified.

NOTE Test methods, test conditions and reporting criteria other than the default conditions may be specified by product standards or in cases where testing is carried out as part of non-standard evaluation of materials.

F.2 Test methods

For tests against liquid challenge chemicals Method A shall be used. For tests against gaseous challenge chemicals Method B shall be used.

F.3 Test conditions

Tests shall be carried out at a temperature of 23 ± 1°C. The test shall be carried out for 8 h or until normalized breakthrough occurs.

F.4 Reporting criteria

The permeation breakthrough time at a normalized permeation rate of 1,0 $\mu g cm^{-2} min^{-1}$ shall be reported as normalized breakthrough time.

Annex G
(informative)

Inter-laboratory and inter-sample variation

The permeation test is, effectively, a destructive test. Once an individual test specimen has been tested it will be contaminated with challenge chemical at a molecular level throughout its thickness. The process of permeation may also have altered the chemical and/or microscopic physical properties of the specimen. It is therefore not possible to assess interlaboratory reproducibility by testing the same specimens in several laboratories. Interlaboratory comparisons can only be made by testing a different set of specimens, even they are all taken from the same sample of a material, in each laboratory. Such a comparison is based on the underlying assumption that all the specimens are identical. However, experience of permeation testing over the last two decades has led to the conclusion that inter-specimen and inter-sample variations can be significantly high.

Although the uncertainty of measurement associated with an individual permeation test (see D.4) may be low, it is frequently found that the results of replicate tests vary by more than can be accounted for by uncertainty of measurement alone. Such inter-specimen variation can often be as high as 20 % from the mean of a set of three results. Permeation test results are known to be strongly sensitive to a range of factors including:

- variation in specimen and sample thickness, even within normal manufacturing tolerances;
- surface blemishes and cosmetic defects;
- minor faults or flaws in underlying textile components of a coated fabric, boot or glove;
- occasional stray fibres from underlying textiles that extend all the way through the coating thereby creating a route for chemicals to rapidly penetrate through the coating by capillary action;
- minor inhomogeneity in polymer or elastomer formulations;
- sample and/or specimen history, including folding, flexing, abrasion, etc.;
- contamination with natural skin oils or cosmetics during normal handling;
- selective surface modification, for example printing or embossing;
- differences in pigmentation, for example different-coloured variants of the same material.

Seemingly insignificant differences between samples of the same clothing, glove or boot material can result in large variations in permeation test results. Inter-sample variation is therefore a significant limiting factor when attempts are made to assess inter-laboratory reproducibility of the permeation test method.

Variations in the design and dimensions of the permeation test cell have shown to have no or little effect on the quality of results. Test results have been found to be sensitive to the choice of aqueous or gaseous collection media, but challenge chemicals for which either medium can be used are in the minority. Variations in the manner in which samples are sealed into the test apparatus are possible, but failure to seal the sample in a leak-tight manner almost always results in apparent immediate breakthrough.

The following data were obtained in a collaborative correlation trial organized by CEN/TC 162 in November 2011 with a group of 14 laboratories. The testing has been carried out in accordance with ISO 6529 and some additional specifications prescribed in the document CEN/TC162/JWG-permeation/N043. These additional specifications included the default conditions for testing and reporting given in Annex F and some further minor differences, which however can be considered as having no essential influence on the interlaboratory variations listed in Table G.1 for three specific combinations of test material and test chemical.

Table G.1 — Results of interlaboratory trial for normalized breakthrough time for three specific combinations of test material and test chemical

Tested material/tested chemical	**Hypalon/toluene**	**Polychloroprene/ Sulphuric acid 96 %**	**Nitrile/methanol**
Source of the material	Especially prepared for the study	Commercial gloves	Commercial gloves
Number of participating laboratories	**13**	**14**	**13**
Number of laboratories after elimination of outliers	**12**	**13**	**13**
Mean value for normalized permeation breakthrough time	**167 min**	**208 min**	**52 min**
Interlaboratory variation	**14 %**	**34 %**	**31 %**

Bibliography

[1] ISO 6530, *Protective clothing — Protection against liquid chemicals — Test method for resistance of materials to penetration by liquids*

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