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**Introduction**

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

For international standards, comments will be reviewed by the relevant UK national committee before sending the consensus UK vote and comments to the international committee, which will then decide appropriate action. If the international standard is approved, it is usual for the text to be published as a British Standard.

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| Date: xx/xx/20xx | Document: ISO/DIS xxxx |
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| 1 | 2 | (3) | 4 | 5 | (6) | 7 |
|---|---|---|---|---|---|---|
| **MB** | Clause No./ Subclause No./Annex **(e.g. 3.1)** | Paragraph/ Figure/Table/Note | **Type of comment** | **Commend (justification for change) by the MB** | **Proposed change by the MB** | **Secretariat observations on each comment submitted** |
| | 3.1 | Definition 1 | ed | Definition is ambiguous and needs clarifying. | Amend to read '...so that the mains connector to which no connection...' | |
| | 6.4 | Paragraph 2 | te | The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK. | Delete reference to UV photometer. | |

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# DRAFT International Standard

# ISO/DIS 6529

# 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*

ICS: 13.340.10

ISO/TC **94**/SC **13**

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**2024-12-17**

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**2025-03-11**

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# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

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

This document was prepared by Technical Committee *[or Project Committee]* ISO/TC 94, *Personal safety - Personal protective equipment*, Subcommittee SC 13, *Protective clothing*.

This fourth edition cancels and replaces the third edition (ISO 6529:2013), which has been technically revised.

The following significant changes to this standard have been made since the previous edition:

1 Restrictions have been placed on the volatility of the challenge chemical. This is because involatile chemicals will not evaporate into a gaseous collection medium to be carried to the detector

2 Restrictions have been placed on the solubility of the challenge chemical. This is because chemicals that are insoluble in a liquid collection medium will not be carried to the detector. Furthermore, chemicals that are only slightly soluble in a liquid collection medium might not sufficiently dissolve in order for detection levels to yield a breakthrough time or cumulative permeation data

3 A requirement has been made that if the above restrictions are not met, then another test method shall be used

4 A requirement has been added to ensure that, when using a gaseous collection medium, the pressure inside the collection side of the permeation test cell relative to the pressure in the challenge side of the permeation test cell shall not exceed a certain value. This is because it has been shown that excessive pressure causes the test specimen to deform, become thinner, and have an increased surface area. This renders the test specimen more susceptible to faster permeation

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# Introduction

Persons 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. Such protective clothing is typically designed to resist permeation and penetration of hazardous chemicals and degradation of the clothing that may be caused by such chemicals.

The test methods described in this document are intended to be used to evaluate the barrier effectiveness of materials used for protective clothing against ingress by liquid or gaseous chemicals.

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

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

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

## 1 Scope

This document 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 continuous contact. This test method is referred to in ISO 16602-3.

Method A is applicable to testing against liquid chemicals, either volatile (with a vapour pressure in excess of 133,332 Pa (1 mmHg) at 20 °C) or sufficiently soluble in water (such that detection limits are possible that allow breakthrough times and/or cumulative permeation parameters to be measured), 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.

These test methods assess the permeation resistance of the protective clothing material under laboratory conditions in terms of cumulative permeation, permeation rate and breakthrough time. 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 document.

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*

ISO 19918, *Protective clothing — Protection against chemicals — Measurement of cumulative permeation of chemicals with low vapour pressure through materials*

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ISO 2286-2:2016, *Rubber- or plastics-coated fabrics — Determination of roll characteristics — Part 2: Methods for determination of total mass per unit area, mass per unit area of coating and mass per unit area of substrate*

ISO 2286-3:2016, *Rubber- or plastics-coated fabrics — Determination of roll characteristics — Part 3: Method for determination of thickness*

ISO 11610:2023, *Protective clothing — Vocabulary*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 2859-1:1999, *Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11610 and the following apply (see also Figure 1).

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

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

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

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

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

Note 2 to entry: A chosen analytical technique may also qualify the chemical/s being tested. Particularly relevant if testing a mixture and identifying and then further quantifying the specific chosen chemical of interest (i.e. some chemicals in a mixture may be toxic and desired to be quantified, others may be ignored, but the method would therefore need to distinguish between them.)

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**
**BDT**
time elapsed from the start of the test to the sampling time at which the test chemical is first detected.

Note 1 to entry: See figure 1 at the end of clause 3.

Note 2 to entry: breakthrough detection time is related to MDPR (minimum detectable permeation rate) which is specific to the test, the test equipment and analytical technique.

**3.3**
**challenge chemical**
see test chemical

**3.4**
**collection medium**
liquid or gas on the inner side of the test sample in which any permeated chemical is collected

**3.5**
**cumulative permeation mass**
**CPM**
total amount of chemical that permeates during a specified period of time since the start of the test

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**3.6**
**cumulative permeation time**
**CPT**
time at which the total quantity of chemical that has permeated through each square centimetre of fabric has reached a predetermined mass

Note 1 to entry: The predetermined mass can vary and would typically be stipulated as per the performance standard. 3.7

**3.7**
**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.

**3.8**
**loop**

**3.8.1**
**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 as a consequence of 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.

**3.8.2**
**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

**3.9**
**minimum detectable mass permeated**
**MDMP**
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.

**3.10**
**minimum detectable permeation rate**
**MDPR**
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.

Note 2 to entry: This value defines the sensitivity of the test, which may be reflected in other values when calculated based on MDPR, for example cumulative permeated mass.

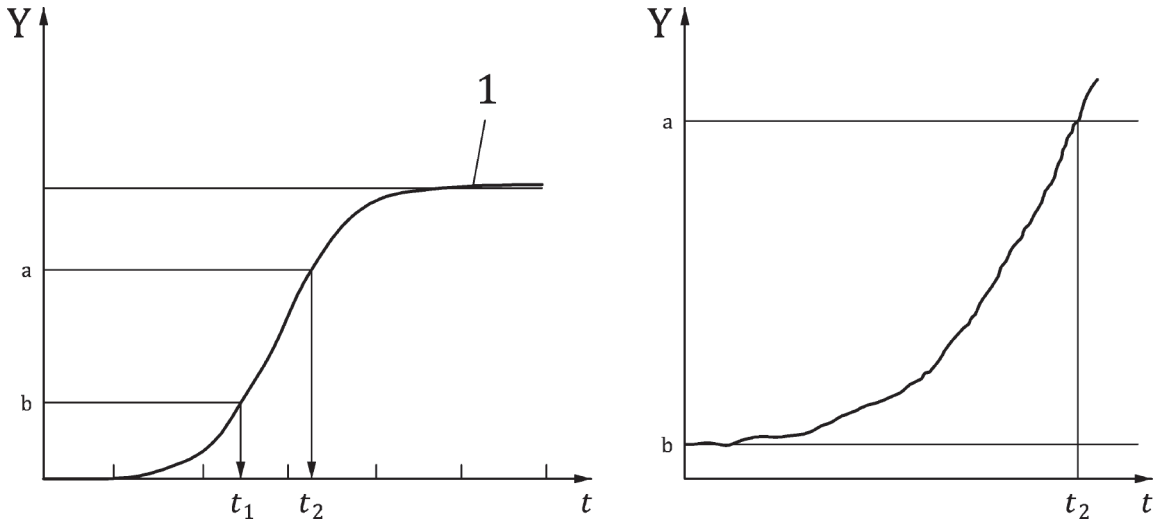
**3.11**
**normalization permeation rate**
**NPR**
permeation rate used for determining the normalized breakthrough detection time

Note 1 to entry: This test method provides two normalisation 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.

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**3.12**
**normalized breakthrough (detection) time**
**NBT**
time at which the permeation rate reaches the normalization permeation rate




**Key**

1 steady-state permeation rate (SSPR)
Y permeation rate (µg/cm$^2$/min)
*a* normalized permeation rate (NPR)
*b* minimum detectable permeation rate (MDPR)
*t* time (min)
$t_2$ normalized breakthrough time (NBT)

**Figure 1 — Schematic permeation graph showing normalised breakthrough times and cumulative permeation mass**

Note 1 to entry: The breakthrough detection time is dependent on the sensitivity of the method and the frequency of sampling of the collection medium.

Note 2 to entry: The cumulative permeated mass over a $t_2$ period is equal to the area of the shaded region under the graph.

**3.13**
**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

**3.14**
**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.

**3.15**
**permeation mass**
quantity of test chemical that passes through the protective clothing material within a given time

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**3.16**
**permeation rate**
mass of test chemical permeating a unit area of the test specimen per unit time

Note 1 to entry: Permeation rate is usually expressed in the units micrograms per square centimetre per minute (µg/cm2/min).

**3.17**
**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.

**3.18**
**response time**
time between the actual arrival of the challenge chemical on the collecting side of the specimen and the time when the analytical instrumentation responds to it

**3.19**
**steady-state permeation rate**
**SSPR**
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.

**3.20**
**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. In the case of liquids, they shall be either volatile (with a vapour pressure in excess of 133,332 Pa (1 mmHg) at 20 °C) or sufficiently soluble in water to determine breakthrough times and/or cumulative permeation limits as defined in this standard. The challenge chemical shall be in continuous contact with the protective clothing material.

The collection medium, which may be liquid or gas, is (either periodically or continuously – for example using an analog detector and chart recorder for continuous measurement) 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.

This test method can also be used with a number of pre-conditioning conditions such as flexing, abrasion, or practical performance tests.

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# 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. The collection medium shall be at the temperature defined for the permeation test as per 10.4.2.

This test method is only applicable to testing against liquid chemicals, either volatile (with a vapour pressure in excess of 133,332 Pa (1 mmHg) at 20 °C) or soluble in water, expected to be in continuous contact with the protective clothing material. If this condition cannot be met, then ISO 19918 (Measurement of cumulative permeation of chemicals with low vapour pressure through materials) shall be used.

NOTE 1 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.

This test method allows for the use of either gaseous or liquid collection media. In some instances, it might be possible to test against the same challenge chemical using either collection medium. Experience has shown that the use of different collection media does not always yield the same results. Where there is a choice, a gaseous collection medium shall be used for testing clothing materials, and an aqueous collection medium shall be used when testing gloves or footwear.

NOTE 2 Gaseous collection media are to be preferred when testing clothing fabrics because, when in use, the inside of the garment is usually in contact with air inside the clothing.

NOTE 3 Aqueous collection media are to be preferred when testing footwear or gloves because, when in use, the inside of these items of PPE is usually in contact either directly with the skin, or in contact with the wearer's perspiration.

## 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 Water grade 3 (ISO 3696) or distilled water is often used.

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### 5.4 Limitation due to collection media

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.

NOTE 1 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.

NOTE 2 Water soluble test specimen (e.g. PVAL-polyvinyl alcohol), a non-aqueous collecting medium needs to be used.

### 5.5 Other collection media

Other collection media such as solid sorbents may be used according to ISO 19918.

## 6 Apparatus

**6.1 Thickness gauge,** shall be suitable for measuring thickness to the nearest 0,02 mm, as specified in ISO 2286-3, 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**, shall be 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 0,000 1 g

**6.3 Permeation test cell**, shall consist 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).

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 difference in pressure between the two surfaces of the test sample shall not exceed 2 500 Pa. This has been found to be critically important.

— If measurement of the hydrostatic pressure of the liquid challenge chemical is not possible for any reason, an estimate shall be reported according to liquid depth and density. If an estimate is used, this shall be reported.

— 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 (except where a solid sorbent is used as per 5.5 where a smaller size facilitates easier removal and replacement with a fresh sorbent collection piece; if using such a sorbent material, this smaller collection area shall be used for all results and subsequent calculations).

— 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.

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— 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 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.

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 1 Gaskets are used to get a good seal. The choice of gasket is very important due to the chemical resistance of different materials. Gaskets may become permeated and need to be replaced to avoid cross contamination from one test to the next.

NOTE 2 Annex B contains a list of suppliers of permeation test cells.

NOTE 3 Undue pressure may cause the fabric to stretch thereby rendering it thinner and less resistant to permeation. It is required that the difference in pressure between the two test surfaces not exceed 2 500 Pa in order to avoid the majority of issues that can arise from this, although the stretching is material dependent and can still occur with this small pressure difference for certain material types. Measurement of the hydrostatic pressure exerted by the challenge liquid may be facilitated by a standard probe with a protective sheath, for example. If measurement is not possible for any reason, estimations may be used according to liquid depth and density.

NOTE 4 For open-loop testing, a larger test cell is preferred because the larger area of fabric exposed to the challenge chemical increases sensitivity. For closed-loop testing, a smaller test cell is preferred in order to reduce the volume of the collection medium and thereby increase the sensitivity of detection. Smaller test cells may nonetheless be used in the open-loop configuration if the challenge chemical is particularly hazardous or expensive and it is necessary to limit the volume used.

NOTE 5 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.

NOTE 6 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.

NOTE 7 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 (including piping, tubing, etc.) 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 Stopwatch, or electronic timer** shall be capable of measuring to the nearest second.

NOTE 1 Computer calculation based on their own internal clock are source of deviation (e.g 1 second is actually only 0,99 seconds).

**6.5 Constant temperature chamber**, bath or room, shall be used to maintain the permeation test cell within ±1,0 °C of the nominal test temperature (See 10.4.2).

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.

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The use of water baths shall be restricted to the testing of PPE (protective clothing, gloves and footwear) material without textile liner, except when demonstrably adequate measures are taken to prevent the migration of water into either side of the test cell or the wicking of water into any internal or external textile component of the test specimen, e.g. by enclosing the permeation cell in a waterproof bag.

# 7 System configuration

## 7.1 General

The apparatus shall be arranged in either the open-loop or closed-loop configuration. The collection medium flow rate shall be induced upstream of the cell either through a pump or other method. Open and closed loop systems do not necessarily provide comparable results.

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.

## 7.2 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 medium 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. The sensitivity is dependent on the minimum detectable rate. 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.

NOTE 3 ASTM F 739-20 Appendix provides a method for checking sensitivity for an open loop system.

The system shall be configured so as to ensure that any molecules of challenge chemical permeating through the test fabric are rapidly mixed into the collection medium. This may be achieved by either or both of:

1. Ensuring that the flow of collection medium through the collection side of the test cell is sufficiently turbulent that mixing is efficient.

NOTE 4 3x cell collection has been found to be sufficient but individual system designs should be independently validated, taking into account in particular system pressure as per 6.3.

2. In such cases mixing may be deemed sufficient if a further increase in flow rate, subject to the system pressure requirement in 6.3 does not alter the measured cumulative permeation, breakthrough time or steady-state permeation rate. At the same time too high a flow rate will dilute the concentration and therefore impact the available detection limit.

3. The addition of some means of stirring or agitating the collection medium in the collection side of the test cell. Such stirring may include, but is not limited to:

    a. the use of a shaft-driven stirrer (it must not create a push or pull effect on the test sample),

    b. the use of a magnetic flea stirrer

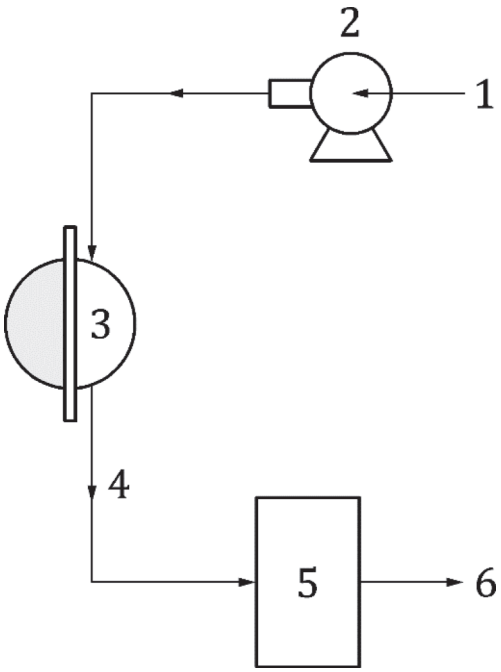
NOTE 5 For liquid collection media, adequate mixing levels can be determined by preliminary experiments in which the rapidity of the dispersion of a dye is observed.

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NOTE 6 It has previously been found that, for some designs of test cell a gaseous flow rate equal to five (±0,5) times the volume of the collection side of the test cell per minute can result in sufficient mixing.

NOTE 7 Lower flow-rates result in lower dilution of permeated chemical and thereby increase minimum detection levels.




**Key**

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

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

NOTE 8 It is advisable that the pump is positioned up-stream of the permeation test cell to avoid negative pressure in the collection chamber drawing chemical through porous materials.

## 7.3 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. The sensitivity is dependent on the minimum detectable mass permeation, which means increased analytical sensitivity is required. 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, however cell design is also important as larger contact areas with limited volume in the collection chamber may be feasible.

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

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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 system shall be configured so as to ensure that any molecules of challenge chemical permeating through the test fabric are rapidly mixed into the collection medium. Such stirring, agitation or pumping shall be constant to create less than ±10 % variation in flow rate. This may be achieved either or both of:

1. Ensuring that the flow of collection medium through the collection side of the test cell is sufficiently turbulent that mixing is efficient. In such cases mixing may be deemed sufficient if a further increase in flow rate does not alter the measured breakthrough time or steady-state permeation rate.

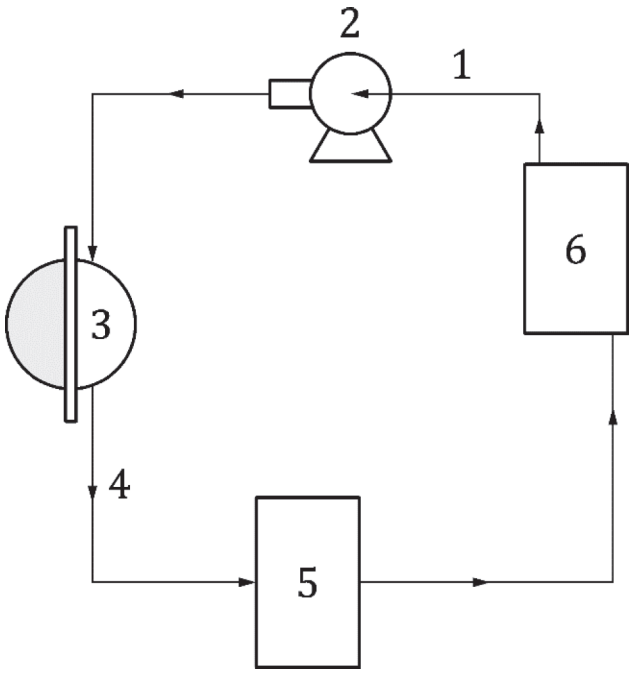
2. The addition of some means of stirring or agitating the collection medium in the collection side of the test cell. Such stirring may include, but is not limited to:

   a. the use of a shaft-driven stirrer (Provided that it does not create a push or pull effect on the test sample),

   b. the use of a magnetic flea stirrer

NOTE 3 It has previously been found that, for some designs of test cell a gaseous flow rate equal to five (±0,5) times the volume of the collection side of the test cell per minute can result in sufficient mixing.

NOTE 4 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 pump
3 sampling side for collection medium
4 collection medium flow path
5 sample analyser (non-destructive)
6 collection medium reservoir

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

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NOTE 5 The pump is up-stream of the permeation test cell in this example, but this is not mandatory. Design of the test setup should consider any impact of pump pressure (positive, negative, or oscillating if using a peristaltic pump for example) on the collection chamber of the test cell, and whether this carries a risk for the specific material type being tested

# 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 shall be as listed in Table 1. The test shall not routinely be run beyond 480 min, but the uncertainty of measurement may require that the test the run for a few minutes longer than 480 minutes.

**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 | Every 11 min |

NOTE 1 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.

NOTE 2 More frequent sampling is permissible

When a test is carried out for the first time on a PPE barrier material, the final result is clearly not known. In such cases the necessary frequency of analysis may be achieved by one of the following means:

1. Sampling at least every 75 s for the first 10 minutes of the test, at least every 150 seconds between the 10th and 60th minutes, at least every 6 minutes between the 60th and 240th minutes, etc.

2. Sampling at least every 75 seconds throughout the test

The lag time as determined from the experimental design shall be measured (to the nearest 15 seconds) and accounted for in reported results.

**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 needs 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 least at 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 120 s.

**8.1.4** Interpolation of data points is allowed if sampling is every 2 minutes or less. If sampling times are longer, then interpolation shall not be used. Table 1 only provides minimum sampling rates. For interpolation to be meaningful it is important to minimise the error associated with it. 2 minute sampling achieves this.

**8.1.5** In some unusual cases it may not be possible or cost-effective to sample the collection medium as frequently as prescribed in Table 1. For example, the quantitative analysis of the collection may take several hours and/or may be particularly expensive. In such cases this shall be noted in the report as a deviation

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to the test method along with a brief explanation of why the prescribed sampling frequency could not be achieved. The report shall also note that a more favourable and/or more accurate result may have been possible had the sampling frequency been greater.

**8.1.6** In cases in which cumulative permeation is to be measured by the open loop technique, in every time interval in which no permeation is measured it shall be assumed that the rate of permeation was actually just less than the minimum detectable permeation rate.

## 8.2 Analytical methods

Any analytical method shall meet the following requirements:

— it is suitably sensitive to the test chemical;

— it is either

  — not significantly sensitive to the collection medium, or

  — any such sensitivity is 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 preferable that the analytical method also provides 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 closed 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.

If an analytical method providing confirmation of the identity of the substance permeating into the collection medium is not available, other detection methods involving the partial or complete destruction of the detected molecule can be used. 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.

## 8.3 Anomalous data points

It is not uncommon for some otherwise reliable analytical systems to give isolated anomalous data points over the course of a permeation test. For example, the permeation rate could be zero for the first 60 minutes of a test, and zero from the 62$^{nd}$ minute until the end of the test after 8 hours. However, the data-point at the 61$^{st}$ minute could indicate a permeation rate of 10 µg/cm$^2$/min. This is clearly an anomalous result because it is not physically possible for the rate of permeation to rise that much and then fall back to zero again in just 2 minutes. Such anomalous data points shall be identified in the report and any graphical representations of the results, but shall be disregarded for the purposes of calculating the final test result. The laboratory shall

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give a brief justification for disregarding such data points. Reasons for such anomalies may include, but are not limited to:

— Transitory fluctuations in the electricity supply to the laboratory

— Minor mechanical disturbance of the detection instrument caused by a slamming door or a minor seismic event

— Momentary disruption of the supply of collection medium

— A recognised minor fault with the instrument that is known to arise from time to time but does not otherwise impact on its analytical performance

### 8.4 Detection of chemical mixtures

When testing a chemical mixture, it shall be determined whether there is more than one chemical of interest to be detected (this will depend on customer requirements, and for example is likely not to be the case with a typical aqueous chemical such as hydrochloric acid, where the acid itself is likely the only chemical requiring quantification). If two or more chemicals of interest are to be detected (for example different solvents used in a cleaning product), then each chemical shall be quantified individually (for example via separation prior to detection using GCMS, LCMS/MS or other applicable techniques) if possible. If this is not feasible, and the chemical is to be detected as a single entity, assuming equal stoichiometric ratio upon detection as is present in the challenge chamber, then only estimated permeation rates and associated breakthrough times, as well as cumulative permeation quantities shall be given, and this shall be clearly noted for each individual entry in the test report. If a mixture is being treated as a single entity in this way (for example separation is not possible) then the calibration procedure and justification shall be clearly documented in the test report. See also section 8.2.

## 9 Sampling procedure

### 9.1 Choice of sample

#### 9.1.1 Clothing samples shall be taken depending on the product standard requirements, if not they 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 10.8. If desired, random specimens may be taken as described in ISO 2859-1.

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**9.1.2** Boot samples shall be taken depending on the product standard requirements, if not they 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 shall be taken depending on the product standard requirements, if not they 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 Size and cutting of specimen

The specimen size shall be such that a good seal is achieved through out the test (for example least 1 mm larger than the circumference of the clamping mechanism of the test cell).

### 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 per unit area of each sample shall be measured to the nearest 1 g/m$^2$.

The thickness shall be measured according to ISO 2286-3 using a pressure of 24 kPa and a 9 mm foot, and the mass per unit area shall be measured according to ISO 2286-2.

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

### 9.4 Conditioning 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 (see 10.4.2).

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.

## 10 Test Procedure

### 10.1 Pre-screening test

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.

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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 It has been found that protective clothing materials which are not resistant to liquid penetration under pressure can nonetheless attain reasonable results in the permeation test. However, it has also been observed that such fabrics often exhibit wide inter-sample variation in the permeation test, hence a larger number of replicate tests is required.

## 10.2 Calibration

The response of the analytical detection system to the test chemical shall be calibrated to determine the analytical sensitivity, MDPR and response time. The calibration shall be carried out at the temperature defined for the permeation test as per 10.4.2 (i.e. the collection medium and associated apparatus shall be at this temperature for the calibration).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.

The maximum quantification limit shall be equivalent to a rate of permeation of ≥ 1,2x for the chosen breakthrough time criterion. For cumulative permeation, the maximum quantification limit shall be equivalent to a rate of ≥ 1,2x the maximum permeation rate observed during the test.

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 suitable to cover at least 1,2x the calibration for the highest permeation measurement taken during the course of the test (for example, if the highest individual measurement is equal to 500 µg/cm$^2$, the calibration shall encompass the required range up to at least 600 µg/cm$^2$.

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 using a fabric that consistently provides a result with repeatability of the permeation profile including the maximum permeation rate observed and steady state permeation rate (the degree of variance may be material-specific, so a suitable tolerance shall be decided by the laboratory according to the chosen material where applicable). 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. (see also calibration)

If changing a condition (such as flow rate for example), a separate validation shall be conducted accordingly to ensure there is no appreciable change (outside of normal expected variance) to the results of the known system.

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 PPE barrier 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

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medium. The clamping mechanism shall be devised such as to, or the specimen shall be prepared such that when sealing of the specimen occurs it:

— is closed with sufficient torque to prevent leakage

— does not over-stretch the sample

— does not deform or pull a thread of the fabric of the specimen, or create folds

**10.4.2** Unless otherwise required, 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. A minimum of 1 measurement for every 60 minutes throughout the test shall be measured and reported.

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.

**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, experimental data suggests that for a typical GC-based open-loop system, a flow rate of 100 mL/min may give sufficient agitation and cell clearance, while meeting the system pressure differential requirement in 6.3. However, it is important to consider individual system limitations and design aspects (including cell design) on a case-by-case basis.

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 according to the chosen method, and shall be continued on a predetermined schedule according to the requirements of 8.1.

Sampling shall be 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.

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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 Permeation

### 10.5.1 General

This standard describes two methods: liquid chemicals with continuous contact and gaseous chemicals with continuous contact.

For the purpose of this test method, the steady state of the permeation rate is reached:

— Open-loop: at the point at which the average of four consecutive measurements at intervals of at least five minutes is within a 5 % relative standard deviation (or the standard deviation divided by the average is less than 0,05). The average of those four measurements shall be reported as the steady-state permeation rate.

— Closed-loop: at the point at which the rate of increase in the concentration of the test chemical in the collection chamber is constant. This is calculated using the average of four consecutive measurements with intervals at least five minutes apart. If the difference between the first two measurements is within 5 % of the difference between the last two measurements, then steady-state is achieved. Is this really needed?

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

For breakthrough time determination:

— a normalized breakthrough time has been recorded;

— permeation reaches a steady-state permeation rate (i.e. breakthrough rate will not be reached);

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

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

NOTE 1 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

— severe degradation of the test specimen has occurred risking saturation of the detector or damage to the test equipment.

For cumulative permeation time (to a specified mass) determination:

— the specified cumulative mass has been reached

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

NOTE 2 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

— severe degradation of the test specimen has occurred risking saturation of the detector or damage to the test equipment.

For cumulative permeation mass (to a specified time) determination:

— the pre-specified time has passed

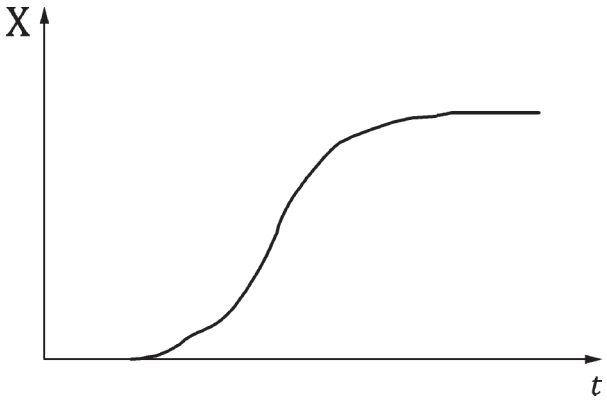
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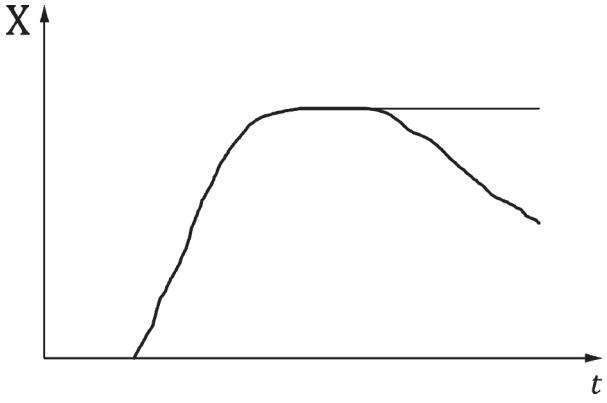
— the rate of permeation is such that the test chemical might overload the detection system or severely contaminate the collection or test apparatus

— severe degradation of the test specimen has occurred risking saturation of the detector or damage to the test equipment.

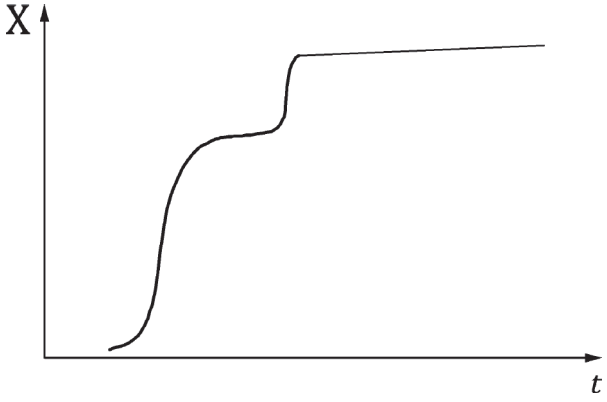
NOTE 3 The end point of the test may typically be instructed by the end user directly and/or the relevant product standard requirements.




**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**

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