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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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/cm²/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/cm²/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/cm²/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/cm²/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/cm²/min or 1.0 μg/cm²/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/cm²/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 22863, 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**.
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 to 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.

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

NOTE 4  The pump may be either downstream 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.