Designation: F739 – 20

Standard Test Method for Permeation of Liquids and Gases Through Protective Clothing Materials Under Conditions of Continuous Contact[1]

This standard is issued under the fixed designation F739; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Workers involved in the production, use, and transportation of liquid and gaseous chemicals can be exposed to numerous compounds capable of causing harm upon contact with the human body. The deleterious health effects of these chemicals can range from acute trauma such as skin irritation and burn, to chronic degenerative disease and mutagenic conditions, including 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.

This test method is used to measure the permeation of liquids and gases through protective clothing materials under the conditions of continuous contact of the clothing material by the test chemical. Resistance to permeation under the condition of intermittent contact with the test chemical should be determined by Test Method F1383. In certain situations, the permeation of liquids through protective clothing materials can be measured using a permeation cup following Test Method F1407. Penetration of liquids should be determined by Test Method F903. An undesirable change in the physical properties of protective clothing materials is called degradation. Procedures for measuring the degradation of rubbers, plastics, and coated fabrics are found in Test Method D471, Practice D543, and Test Method D751, respectively. A starting point for selecting the chemicals to be used in assessing the chemical resistance of clothing materials is Guide F1001.

1. Scope

1.1 This test method measures the permeation of liquids and gases through protective clothing materials under the condition of continuous contact.

1.2 This test method is designed for use when the test chemical is a gas or a liquid, where the liquid is either volatile (that is, having a vapor pressure greater than 1 mm Hg at 25 °C) or soluble in water or another liquid that does not interact with the clothing material.

1.3 Values states in SI units are to be regarded as standard. Values given in parentheses are not exact equivalents and are given for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 7.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D471 Test Method for Rubber Property—Effect of Liquids

D543 Practices for Evaluating the Resistance of Plastics to Chemical Reagents

D751 Test Methods for Coated Fabrics

D1777 Test Method for Thickness of Textile Materials

E105 Practice for Probability Sampling of Materials

[1] This test method is under the jurisdiction of ASTM Committee F23 on Personal Protective Clothing and Equipment and is the direct responsibility of Subcommittee F23.30 on Chemicals.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

E171/E171M Practice for Conditioning and Testing Flexible Barrier Packaging
F903 Test Method for Resistance of Materials Used in Protective Clothing to Penetration by Liquids
F1001 Guide for Selection of Chemicals to Evaluate Protective Clothing Materials
F1194 Guide for Documenting the Results of Chemical Permeation Testing of Materials Used in Protective Clothing
F1383 Test Method for Permeation of Liquids and Gases Through Protective Clothing Materials Under Conditions of Intermittent Contact
F1407 Test Method for Resistance of Chemical Protective Clothing Materials to Liquid Permeation—Permeation Cup Method
F1494 Terminology Relating to Protective Clothing
F2815 Practice for Chemical Permeation Through Protective Clothing Materials: Testing Data Analysis by Use of a Computer Program (Withdrawn 2019)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *analytical technique, n*—a procedure whereby the concentration of the test chemical in a collection medium is quantitatively determined.

3.1.1.1 *Discussion*—These techniques are often specific to individual chemical and collection medium combinations. Applicable techniques include, but are not limited to: flame ionization, photo ionization, electro-chemical, ultraviolet and infrared spectrophotometry, gas and liquid chromatography, colorimetry, length-of-stain detector tubes, and radionuclide tagging/detection counting.

3.1.2 *breakthrough detection time, n*—the elapsed time measured from the initial exposure to the test chemical to the sampling time that immediately precedes the sampling time at which the test chemical is first detected.

3.1.2.1 *Discussion*—(See Fig. 1.) The breakthrough detection time is dependent on the sensitivity of the method (see Appendix X1).

3.1.3 *closed-loop, adj*—refers to a testing mode in which there is no change in the volume of the collection medium except for sampling.

3.1.4 *collection medium, n*—a liquid, gas, or solid that absorbs, adsorbs, dissolves, suspends, or otherwise captures the test chemical and does not affect the measured permeation.

3.1.5 *cumulative permeation, n*—the total mass of chemical that permeates a specific area of protective clothing material during a specified time from when the material is first contacted by the test chemical.

3.1.6 *degradation, n*—a deleterious change in one or more properties of a material.

3.1.6.1 *Discussion*—For protective clothing materials, changes in physical properties are typically of most interest.

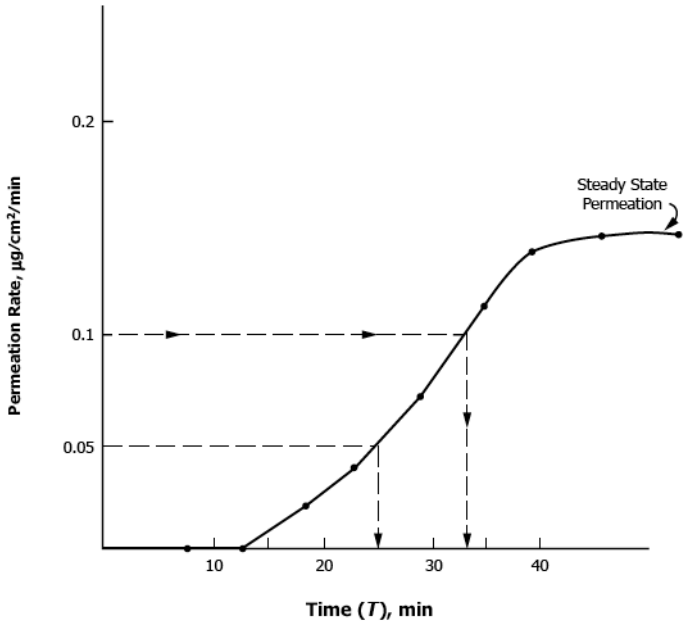


FIG. 1 The Breakthrough Detection Time for a Method Sensitivity of 0.05 µg/cm²/min is 25 min. The Standardized Breakthrough Detection Time is 33 min. The Steady-State Permeation Rate is Approximately 0.15 µg/cm²/min.

3.1.7 *minimum detectable mass permeated, n*—the smallest mass of test chemical that is detectable with the complete permeation test system.

3.1.7.1 *Discussion*—This value is not necessarily the sensitivity of the analytical instrument.

3.1.8 *minimum detectable permeation rate, n*—the lowest rate of permeation that is measurable with the complete permeation test system.

3.1.8.1 *Discussion*—This value is not necessarily the sensitivity of the analytical instrument.

3.1.9 *normalized breakthrough time, n*—the time at which the permeation rate reaches 1.0 µg/cm²/min.

3.1.10 *open-loop, adj*—refers to a testing mode in which fresh collection medium flows continuously through the collection chamber of the test cell.

3.1.11 *penetration, n*—for chemical protective clothing, the movement of substances through voids in protective clothing materials or items on a non-molecular level.

3.1.11.1 *Discussion*—Voids include gaps, pores, holes, and imperfections in closures, seams, interfaces, and protective clothing materials. Penetration does not require a change if state; solid chemicals move through voids in materials as solids, liquids as liquids, and gases as gases. Penetration is a distinctly different mechanism from permeation.

3.1.12 *permeation, n*—for chemical protective clothing, the movements of chemicals as molecules through protective clothing materials by the processes of (*1*) absorption of the chemical into the contact surface of the materials, (*2*) diffusion of the absorbed molecules throughout the material, and (*3*) desorption of the chemical from the opposite surface of the material.

[3] The last approved version of this historical standard is referenced on www.astm.org.

3.1.12.1 *Discussion*—Permeation is a distinctly different mechanism from penetration.

3.1.13 *protective clothing, n*—item of clothing that is specifically designed and constructed for the intended purpose of isolating all or part of the body from a potential hazard; or, isolating the external environment from contamination by the wearer of the clothing.

3.1.14 *seam, n*—a line along which two pieces of material are joined together in protective clothing.

3.1.14.1 *Discussion*—Common ways that seams are constructed include sewing with thread, welding with heat, taping, and gluing.

3.1.15 *standardized breakthrough time, n*—the time at which the permeation rate reaches 0.1 μg/cm^2/min.

3.1.16 *steady-state permeation, n*—the constant rate of permeation that occurs after breakthrough when the chemical contact is continuous and all forces affecting permeation have reached equilibrium.

3.1.17 *test chemical, n*—the solid, liquid, gas, or mixture thereof, used to evaluate the performance of a protective clothing material.

3.1.17.1 *Discussion*—The liquid or gas may be either one component (for example, a neat liquid or gas) or have several components (for example, a mixture).

3.1.18 *volatile liquid, n*—a liquid with a vapor pressure greater than 1 mm Hg at 25 °C.

3.2 For other protective clothing definitions, refer to Terminology F1494.

4. Summary of Test Method

4.1 The permeation of chemical(s) through a protective clothing material is assessed by measuring the breakthrough detection time, standardized breakthrough time, normalized breakthrough time, subsequent permeation rate, and cumulative permeation over a period of time through replicate specimens of the material.

4.2 In the permeation test apparatus, the protective clothing material specimen partitions the test chemical from the collection medium.

4.2.1 The collection medium is analyzed quantitatively for its concentration of the test chemical and, thereby, the amount of that chemical that has permeated the barrier as a function of time after its initial contact with the material.

4.2.2 By either graphical representation, appropriate calculations, or both, the breakthrough detection time, standardized breakthrough time, normalized breakthrough time, permeation rate, and cumulative permeation of the test chemical are determined.

5. Significance and Use

5.1 This test method is normally used to evaluate flat specimens from finished items of protective clothing and from materials that are candidates for items of protective clothing.

5.1.1 Finished items of protective clothing include gloves, sleeves, aprons, suits, coveralls, hoods, boots, respirators, and the like.

5.1.2 The phrase "specimens from finished items" encompasses seamed or other discontinuous regions as well as the usual continuous regions of protective clothing items.

5.1.3 Selected seams for testing are representative of seams used in the principal construction of the protective clothing item and typically include seams of both the base material and where the base material is joined with other types of materials.

5.2 The breakthrough detection time, standardized breakthrough time, permeation rate, and cumulative permeation are key measures of the effectiveness of a clothing material as a barrier to the test chemical. Such information is used in the comparison of clothing materials during the process of selecting clothing for protection from hazardous chemicals. Long breakthrough detection times, long standardized breakthrough detection times, low amounts of cumulative permeation, and low permeation rates are characteristics of more effective barrier materials than materials with higher permeation characteristics.

NOTE 1—At present, only limited quantitative information exists about acceptable levels of dermal contact with most chemicals. Therefore, the data obtained using this test method cannot be used to infer safe exposure levels.

5.2.1 The reporting of a standardized breakthrough time greater than a specific time period means that the test chemical has not permeated the specimen at a rate exceeding 0.1 μg/cm^2/min in the designated time. Permeation may or may not have occurred at a lower rate during this time interval.

5.2.2 The reporting of cumulative permeation over a specified test period is another means to report barrier performance of protective clothing for resistance to permeation. This measurement quantifies the total amount of chemical that passed through a known area of the material during the specified test period.

NOTE 2—It is possible to relate cumulative permeation test results to the total amount of chemical to which an individual wearer may be exposed by accounting for the exposed surface area and the underlying air layer. This information has value when there are known maximum permitted skin exposure doses for specific chemicals.

5.3 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 collection system selected, and the ratio of material specimen area to collection medium volume or flow rate.

5.3.1 The analytical technique employed shall be capable of measuring the concentration of the test chemical in the collection medium at or below 0.05 μg/cm^2/min, and at or above the steady-state permeation rate.

5.3.2 Often permeation tests will require measurement of the test chemical over several orders of magnitude in concentration, requiring adjustments in either the sample collection volume or concentration/dilution, or the analytical instrument settings over the course of the test.

5.3.3 Higher ratios of material specimen area to collection medium volume or flow rate permit earlier detection of breakthrough and detection of lower permeation rates and levels of cumulative permeation because higher concentrations

(a) (b)

NOTE 1—In each image, the closed chamber is on the right and the flow chamber is on the left of the assembly.

FIG. 2 Test Cell Setup—(a) 1 in. Diameter Cell; (b) 2 in. Diameter Cell

of the test chemical in the collection medium will develop in a given time period, relative to those that would occur at lower ratios.

5.4 Comparison of results requires specific information on the test cell, procedures, and analytical techniques. Results obtained from closed-loop and open-loop testing may not be directly comparable.

5.4.1 The sensitivity of an open-loop system is characterized by its minimum detectable permeation rate. A method for determining this value is presented in Appendix X1.

5.4.2 The sensitivity of a closed-loop system is characterized by its minimum detectable mass permeated.

5.5 A group of chemicals for use in permeation testing is given in Guide F1001.

5.6 While this method specifies standardized breakthrough time as the time at which the permeation rate reaches 0.1 μg/cm^2/min, it is acceptable to continue the testing and also report a normalized breakthrough time at a permeation rate of 1.0 μg/cm^2/min.

5.7 It is recommended that the test be continued for the measurement of maximum or steady-state permeation rate or for the duration specified for the determination of cumulative permeation.

5.7.1 It is permitted to terminate tests early if there is catastrophic permeation of the chemical through the protective clothing material and the rate of permeation could overwhelm the capability of the selected analytical technique.

5.8 Guide F1194 provides a recommended approach for reporting permeation test results.

6. Apparatus

6.1 *Thickness Gauge,* suitable for measuring thicknesses to the nearest 0.02 mm (or the nearest 0.001 in.), as specified in Test Method D1777, shall be used to determine the thickness of each protective clothing material specimen tested.

6.2 *Analytical Balance,* readable and reproducible to ±0.5 mg, shall be used to determine weight per unit area of each test specimen.

6.3 *Test Cell*—The test apparatus consists of a two-chambered cell for contacting the specimen with the test chemical on the specimen's normally outside surface and with a collection medium on the specimen's normally inside surface. See Fig. 2.

NOTE 3—Use of a 2 in. (50 mm) diameter cell (Fig. 2(b)) is preferred over a 1 in. (25 mm) diameter cell (Fig. 2(a)) due to higher ratios of material specimen surface area to collection medium volume.

6.3.1 The chambers are of two types:

6.3.1.1 *Closed Chamber*—The closed chamber contains a fixed volume of liquid and a straight bore, standard taper spout for adding challenge chemical or collection medium. Small volumes of collection medium may be removed with or without replacement for analysis. The 1 in. closed chamber is 23 mm (0.917 in.) in length and 25.3 mm (1.0 in.) internal diameter (see Fig. 2(a)). The internal volume of the closed chamber is 17.1 mL. The 2 in. closed chamber is 22.0 mm (0.87 in.) in length and 50 mm (2.0 in.) internal diameter (see Fig. 2(b)). The internal volume of the closed chamber is 48 mL.

6.3.1.2 *Flow Chamber*—The flow chamber has inlet and outlet ports with valves through which a challenge chemical or a collection medium flows during the test. The flow chamber is used for continuously passing a gaseous challenge over the normally outside surface of the test specimen, or continuously passing a gaseous or liquid collection medium over the normally inside surface of the test specimen. The 1 in. flow chamber is 31 mm (1.25 in.) in length and 25.3 mm (1.0 in.) internal diameter. The inlet and outlet ports have 4 mm

(0.19 in.) internal diameters (see Fig. 2(a)). The internal volume of the flow chamber is 17.8 mL. The 2 in. flow chamber is 35 mm (1.38 in.) in length and 50 mm (2.0 in.) internal diameter. The inlet and outlet ports have 4 mm (0.16 in.) internal diameters (see Fig. 2(b)). The internal volume of the flow chamber is 68.7 mL.

6.3.1.3 The open, circular end of each chamber is flared to create a flange that facilitates clamping the chambers together.

6.3.1.4 Use chemically inert and non-absorptive test cell parts that contact the test chemical.

NOTE 4—The standard closed and flow chambers are made of glass.[4] Test chemicals (for example, hydrofluoric acid) that are corrosive to glass require chambers constructed of alternative materials.

6.3.2 Select the test cell configuration based on the challenge chemical and most appropriate analytical method.

NOTE 5—The configuration can be of two closed chambers, two flow chambers, or one closed and one flow chamber.

6.3.2.1 When the flow chamber contains the challenge chemical, the chemical is introduced through the longer stem that goes all the way to the end of the chamber. A shorter stem on the side of the test chamber provides the challenge chemical a means of exit from the test chamber. This mode of entry and exit of the challenge chemical aids in mixing of the chemical inside the test chamber. Flow of the challenge chemical must be regulated such that its composition and the concentration does not change over time.

6.3.3 The test specimen is sandwiched between two PTFE or butyl gaskets and the assembly is clamped between the two chambers.

NOTE 6—Butyl gaskets can become contaminated and contaminate future tests.

NOTE 7—Adequate seal of elastomeric specimens may be achieved without use of gaskets.

6.3.4 *Additional Information:*

6.3.4.1 Make leak-tight connections to the collection chamber inlet and outlet tube. In addition, use tubing which is in contact with the test chemical that is made from material that does not absorb or react with the test chemical. Glass, PTFE, or stainless steel are appropriate choices in most cases. It is recommended to make connections of external tubing to the glass inlet and outlet ports of the test cell chambers via PTFE pressure-fit union connectors.

6.3.4.2 In non-flow tests where increased analytical sensitivity is required, use a closed chamber to reduce the volume of the collection medium. This increases the sensitivity of the method by increasing the ratio of material specimen area to the collection medium volume. Similarly, use a lower volume test chamber for a high hazardous chemical to minimize the amount of chemical being used for testing

6.3.4.3 In open-loop tests, lower collection medium flow rates increase the system sensitivity by lowering the minimum detectable permeation rate. However, these approaches to increasing sensitivity must be achieved within the constraints of having sufficient volumes and mixing rates so as not to interfere with the permeation process.

[4] The closed and flow chambers are available from Pesce Lab Sales, 355 N. Lincoln St, Kennett Square, PA 19348.

NOTE 8—A flow rate of 0.1 L/min has been found to achieve the required analytical sensitivity for minimum detectable permeation rate with an optimal mixing efficiency.

6.3.5 Special considerations with liquids that are mixtures:

6.3.5.1 In case of liquids that are mixtures and for liquid collections, minimize concentration gradients by mounting the test cell setup on a rocker table in a vertical orientation to ensure both surfaces of the specimen are fully contacted by liquids. Set the table rocker to be continuous with lowest speed sufficient to promote uniform mixing.

6.3.5.2 Alternatively, liquid test chemicals that are mixtures can be stirred to minimize concentration gradients. Use a stirring rod inserted through the fill spout or a magnetic stirrer.

6.3.5.3 If a stirrer is used, do not let it contact or damage the specimen.

NOTE 9—If there is a poor seal of the shaft of the rod with the spout, evaporation of the chemical can occur, reducing its volume and potentially changing its composition.

6.3.5.4 For a liquid collection medium that is not circulated, use a test cell design and permeation test setup that permits the mixing, withdrawal, and replenishment of the collection medium during the test.

6.4 *Alternative Test Cells*—Alternative permeation test cells shall be permitted to be used, provided the type of test cell used is reported as prescribed in Section 12. The cell and configuration described above and shown in Figs. 2 and 3, however, is the standard. If an alternate cell is used, the equivalence of the alternative test cell must be documented as described in Section 12.

6.5 *Constant-Temperature Chamber or Bath,* used to maintain the test cell within ±1.0 °C of the test temperature. The standard temperature is 27 °C. Condition all test materials, including the test cells and chemicals, in the chamber(s) or bath(s) prior to testing.

6.6 *Circulating Pump,* if appropriate, is used to transport the collection medium or test chemical, or both, through the test cell. All parts contacting the test chemical must be chemically inert and non-absorptive to the test chemical. The flow rate must be sufficiently high to provide adequate mixing or dilution, or both, within the test cell.

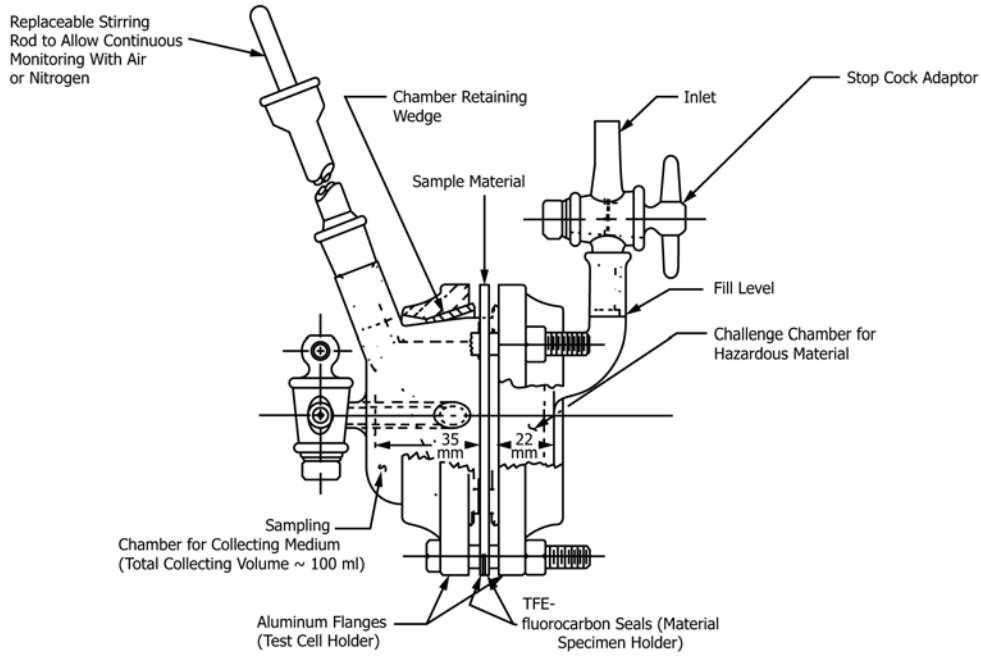
NOTE 10—If a circulating pump is used, care should be taken to avoid inducing pressure which may deform or damage the test specimen.

6.7 *Flow Meter,* used to measure the flow rate of the collection medium through the collection chamber. A calibrated rotameter, or similarly accurate device, shall be used. The flow rate shall be measured in-line with all system components in place at the start of each test.

6.8 *Thermometer or Thermocouple,* used to measure the temperature of the constant-temperature chamber (or bath), or the collection chamber of the test cell, or both. A calibrated device accurate to ±0.5 °C shall be used.

7. Safety Precautions

7.1 Before this test method is carried out, safety precautions recommended for handling any potentially hazardous chemical should be identified and reviewed to provide full protection to all personnel.



NOTE 1—Cell can be reconfigured for gas challenges. Collection chamber can be used in open- or closed-loop mode. Closed chamber is to right of sample material; flow chamber to left.

FIG. 3 Standard Cell Configured for Liquid Challenges

7.1.1 For carcinogenic, mutagenic, teratogenic, and other toxic (poisonous) chemicals, the work area should be isolated, well ventilated, and meticulously clean. Involved personnel should be outfitted with protective clothing and equipment.

7.1.2 For corrosive or otherwise hazardous chemicals, involved personnel should be outfitted with protective clothing and equipment.

7.2 Emergency equipment, such as a safety shower, eye wash, and self-contained breathing apparatus, should be readily accessible from the test area.

7.3 Appropriate procedures for the disposal of the chemicals should be followed.

8. Testing and Analytical Technique Consideration

8.1 Each protective clothing material specimen shall be permitted to consist of either a single layer or a composite of multiple layers that is representative of actual protective clothing construction, with all layers arranged in proper order. In each test, the specimen's normally outer surface shall contact the test chemical.

8.1.1 If in a design of protective clothing different materials or thicknesses of materials are specified at different locations, specimens from each location shall be tested.

8.1.2 If in a design of protective clothing seams are used, additional specimens containing such seams shall be tested. Care must be taken to ensure that the test cell can be properly sealed when specimens of nonuniform thickness are tested.

NOTE 11—Use of a 2 in. (50 mm) diameter cell is preferred over a 1 in. (25 mm) diameter cell for this reason.

8.2 Sample size is dependent on test cell dimensions.

8.2.1 For a 2 in. (50 mm) diameter cell, each material specimen to be tested shall have a minimum cross dimension of 68.6 mm (2.7 in.). A 76.2 mm (3 in.) diameter circle is convenient.

8.2.2 For a 1 in. (25 mm) diameter cell, each material specimen to be tested shall have a minimum cross dimension of 43 mm (1.7 in.). A 51 mm (2 in.) diameter circle is convenient.

8.2.3 Specimens are permitted to extend beyond the edge of the sealing surface if the larger specimen does not interfere with the ability to seal the test cell.

8.3 A minimum of three random specimens shall be tested. Random specimens shall be generated as described in Practice E105.

8.4 To avoid incidental contamination of exposed surfaces, clean gloves shall be worn when handling specimens.

8.5 To avoid affecting permeation quantification, the collection medium should not interact with the test material and must have adequate capacity for the permeant. To have adequate capacity for the permeant, the collection medium should not exceed 20 % of its saturation concentration from the permeant at any time during the test. For a liquid collection medium, saturation is the maximum solubility or miscibility of the permeant in the liquid at the test temperature. For a gaseous collection medium, saturation is determined by the vapor pressure of the permeant.

8.6 Under conditions in which the test chamber or bath is at a temperature significantly different from that of the test chemical or collection medium that is being introduced into the test cell, the temperature in the test chemical chamber or the collection chamber, or both, should be measured. It may be

necessary to precondition the test chemical or collection medium before it enters into the test cell. Similarly, it may be necessary to maintain the temperature of the collection medium after it leaves the test cell to prevent condensation or precipitation.

8.7 The combination of system configuration, analytical technique, and collection medium shall be selected to allow quantification of the test chemical over the range of concentrations that is consistent with 5.3.1, without exceeding the maximum concentration limits within the system as defined in 8.5.

8.7.1 The combination of system configuration, analytical technique, and collection medium shall be calibrated with the test chemical over the range of permeant concentrations consistent with 8.7.

8.7.2 Distilled water is preferred as a collection medium for non-volatile and semi-volatile test chemicals and non-water sensitive protective clothing materials. Consider alternative liquids only when the test chemical does not meet the solubility requirements as described in 8.5 or when the protective clothing material is water sensitive.

8.7.3 Air, nitrogen, and helium are the preferred choices for the collection medium for volatile test chemicals. Consider alternative gases only when these gases interfere with analytical detection of the test chemical. Regardless of the gas used, its purity must be sufficiently high so as not to interfere with the permeation process or the analytical procedure.

8.7.4 In open-loop testing, the system shall have a sensitivity of at least 0.05 μg/cm^2/min. (See Appendix X1.)

8.7.5 In closed-loop testing, the system shall have a minimum sensitivity to detect a permeation rate of 0.05 μg/cm^2/min over a 5-min sampling period.

8.8 With the nominal 25 mm diameter cell and in open-loop mode or in closed-loop mode with a circulating collection medium, the minimum flow rate for the collection medium is 100 cm^3/min. Higher flow rates are preferred within the constraints imposed by analytical sensitivity, temperature control, and pressure gradients in the system.

8.9 With the nominal 50 mm diameter cell and in open-loop mode or in closed-loop mode with a circulating collection medium, the minimum collection medium flow rate is 300 cm^3/min. Higher flow rates are preferred within the constraints imposed by analytical sensitivity, temperature control, and pressure gradients in the system.

8.9.1 The purpose of agitating/mixing the collection medium is twofold: to ensure that it is homogeneous for sampling and analytical purpose 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 clothing material and the collection medium. At this time, sufficient data are not available to specify minimum agitation rates. However, as guidance, in any system in which the collection medium is flowing through the collection chamber, the minimum flow rate should be five chamber volumes per minute. Higher rates may be required for permeants with low solubilities in the collection medium or high permeation rates. High flow rates also result in better mixing in the chamber and, consequently, more uniform samples for analysis. For these reasons, it is recommended that the condition of steady-state permeation be verified by measuring it at two different flow rates (see 10.9). Note, however, that higher flow rates will reduce the sensitivity of the system to the detection of breakthrough. For non-circulating collection medium systems, adequate mixing levels can be determined by preliminary experiments in which the rapidity of the dispersion of a dye is observed.

8.10 Care must be taken so as not to pressurize the test or collection chambers. Overly high pressures may develop at high gas flow rates or as a result of attachments that restrict the flow of gas from the chamber. Tightly packed activated carbon beds or highly restrictive sparger tubes are examples of such attachments. A differential pressure gauge can be used to measure pressures within the test or collection chamber over the range of expected flow rates by use of a modified chamber having an access port. As a rule of thumb, internal pressures should not exceed ambient pressure by more than 5 %. Overpressurization of either chamber of the test cells may result in distortion of the specimen, with concurrent increase in specimen surface area and decrease in specimen thickness.

8.11 In closed-loop systems with sample withdrawal, replenishment of the collection medium may be necessary to maintain a fixed ratio of collection medium volume to surface area of the test specimen in contact with the collection medium. See 11.4 for calculations related to this issue.

8.12 In cases where samples are withdrawn, analyzed, and returned to the test cell, no provision for volume maintenance is necessary.

9. Conditioning

9.1 Condition each protective clothing material specimen for a minimum of 24 h by exposure to a temperature of 27 ± 2 °C (81 ± 4 °F) and a relative humidity of 30 to 80 % as described in Practice E171/E171M.

9.2 Different types of specimen preconditions are permitted, including repeated flexing or abrasion, on samples from which permeation test specimens are removed. Describe any specific preconditions used in the test report.

10. Procedure

10.1 Measure the thickness of each conditioned specimen to the nearest 0.02 mm (or nearest 0.001 in.) at three locations within the area of the specimen that is to be exposed to the test chemical. Calculate the average thickness and record.

10.2 Determine specimen weight per unit area in grams per square centimeter by weighing the specimen on an analytical balance (±2 mg) and dividing by the area (±0.4 cm^2), and record. This value, along with thickness, is a key characteristic of the material and is needed when comparing the results of permeation testing.

10.3 Measure and record the inside diameter of the nominal 25 mm diameter opening of the PTFE gasket. Mount the first

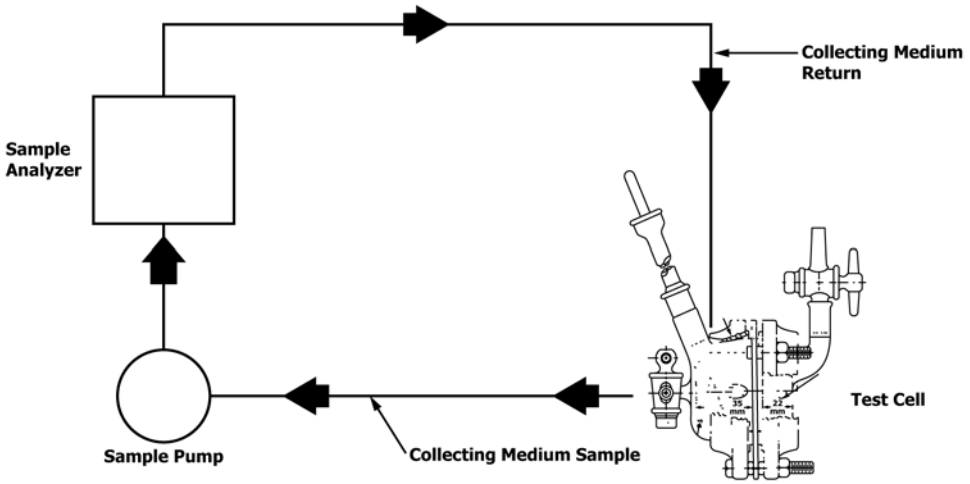


FIG. 4 Example Setup for Continuous Collecting Medium Sample Withdrawal, Analysis, and Return

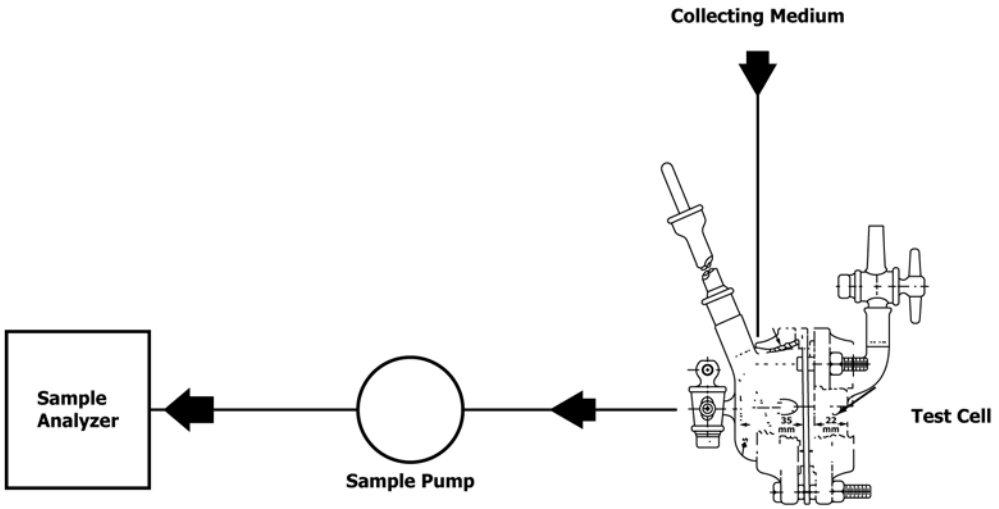


FIG. 5 Example Setup for Continuous Flow of Fresh Collecting Medium

specimen in the test cell and assemble as shown in Figs. 2 and 3 and described in 6.3.1 and 6.3.3.

10.3.1 Seal the test specimen in the test cell to prevent leakage but to avoid damage to the test specimen.

10.3.2 Special gasket materials are generally needed to seal specimens with uneven surfaces such as seams, which join two materials.

10.4 Place the assembled test cell into a constant-temperature chamber or bath at 27 °C, the standard temperature for this method. Other temperatures may be used but must be noted in the report. The test cell must not be removed from the temperature chamber or bath for the duration of the test.

10.5 Charge the collection medium into the test cell chamber that contacts the inside surface of the material specimen. The collection medium must be at the test temperature when it is introduced. Depending upon the combination of analytical technique and collection medium selected, attach peripheral devices as appropriate (see Figs. 4 and 5). The cell, along with the collection medium, should be maintained at the test temperature for at least 30 min before the test proceeds further. Temperature variances have significant effects on the results and reproducibility of the method.

10.6 Stir, circulate, or flow the collection medium continuously. (See 8.9.1.)

10.7 Initiate sampling of the collection medium, either continuously or discretely, and continue on a predetermined schedule throughout the test duration. Sampling is initiated before the test chemical is added to the test cell to establish the baseline values against which subsequent analytical data will be compared.

NOTE 12—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 using open-loop techniques, the flow of collection medium should never be interrupted. This will minimize adsorption of permeated test chemical on the walls of the test cell and associated tubing.

10.8 Add the test chemical into the test chemical chamber.

10.8.1 For liquid test chemicals, the chemical can be introduced by pouring, syringe, cannula, etc. Fill the chamber to a level that indicates the liquid is covering the clothing material specimen, with no air bubbles. Begin timing the test when the addition of the liquid commences.

10.8.2 For gaseous test chemicals, begin the flow of the gas into the test chemical chamber. Begin timing the test when the equivalent of five chamber volumes of gas have passed through the chamber as determined by means of a rotameter or other flow-monitoring device placed in the outlet stream of the test chemical chamber.

10.9 Record the concentration of the test chemical found in each sample and the associated time that has elapsed between the time that the challenge chemical was charged to the cell and the withdrawal of the sample.

10.9.1 An analytical sample should be collected as soon as possible after contact of the specimen with the test chemical, but must be collected within the first 15 min of contact.

10.10 Discontinue sampling and terminate the test after one or more of the following conditions is met (see Fig. 6):

10.10.1 Steady-state permeation is reached (Fig. 6A and Fig. 6E).

10.10.1.1 In an open-loop system, steady-state permeation can be defined as the point at which the average of four measurements of the permeation rate collected at intervals of at least 5 min shows less than a 5 % relative standard deviation (that is, the standard deviation divided by the average is less than 0.05). The average of the four measurements should be reported as the steady-state permeation rate.

10.10.1.2 It is recommended that the condition of steady-state permeation be verified by repeating the measurements and calculations described in 10.10.1.1 after doubling the flow rate through the collection chamber.

10.10.1.3 For a closed-loop system, steady-state permeation is achieved when the rate of increase in the concentration of the test chemical in the collection chamber is constant. This can be tested with four measurements collected at intervals at least 5 min apart. If the difference between the first two measurements is within 5 % of the difference between the last two measurements, then it can be concluded that steady-state has been achieved. The average of the four measurements can be used to calculate the steady-state permeation rate. (See 11.3 and 11.4.)

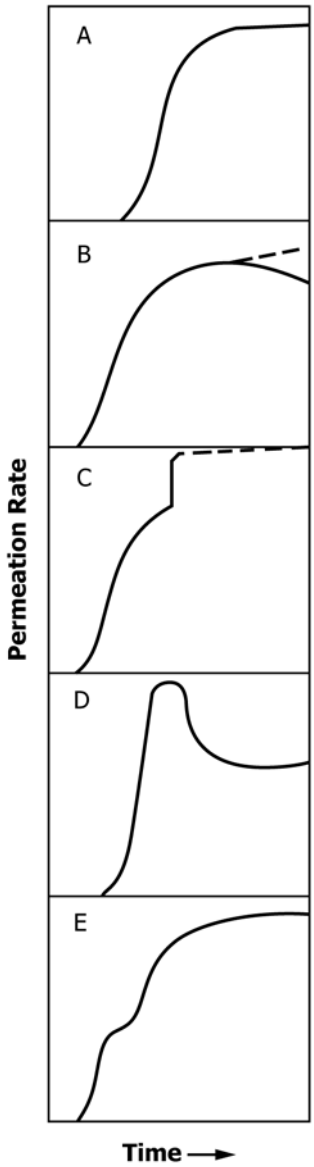
10.10.2 Permeation proceeds at an ever increasing rate (Fig. 6C).

10.10.3 A maximum rate is reached and a steady-state permeation rate is not achieved within 30 min after the maximum rate occurs. (Fig. 6B and Fig. 6D.)

10.10.4 A pre-specified time has passed.

10.11 Remove the test cell from the controlled-temperature environment. Remove the test chemical. Remove the collection medium. Disassemble the test cell and thoroughly clean it.

10.12 A minimum of three specimens per condition, as detailed in 8.1, shall be tested.



NOTE 1—Fig. 6 shows five types of permeation behavior. Type A, the most typical, where the permeation rate stabilizes at a "steady state" value. Type B behavior is due to the material specimen being structurally modified by the chemical, resulting in an increase or decrease in permeation rate. Type C behavior occurs when the material specimen exhibits a sudden, very large increase in rate. Type D response happens when there is moderate to heavy swelling of the material specimen, although the permeation rate eventually stabilizes. Type E response can occur when there is a high degree of swelling.

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FIG. 6 Five Types of Permeation Behavior

11. Calculations[5]

NOTE 13—Data from this method shall be permitted to be analyzed by following Practice F2815.

[5] Public domain software for making the calculations in this section is available from the NIOSH National Personal Protective Technology Laboratory (NPPTL), 626 Cochrans Mill Road, P.O. Box 18070, Pittsburgh, PA, 15236, USA. Also available at http://www.cdc.gov/niosh/npptl/default.html. USA telephone: 412-386-6885.

11.1 *Symbols*—The following symbols are used in the equations:

A = area of the material specimen contacted, cm^2,
C, C_i = concentration of test chemical in collection medium where C_i is the concentration at time T_i, µg/L,
F = flow rate of collection medium through the cell, L/min,
i = an indexing number assigned to indicate the specific concentration C_i that was measured at time T_i in volume V_i; starting with $i = 1$ for the first sample; the values of C_0 and T_0 are zero (0),
K_{Ti} = cumulative permeation determined at a given time, T_i,
n = number of measurement intervals between the start of the test and the time for which cumulative permeation is reported,
P, P_i = permeation rate where P_i is the permeation rate at T_i, $\mu g/cm^2/min$,
$\bar{P}_i$ = average permeation rate for the interval T_{i-1} to T_i, $\mu g/cm^2/min$,
T, T_i = time elapsed beginning with initial chemical contact where T_i is the time at which concentration in the collection medium was C_i, min,
T_p = the elapsed time from the beginning of the chemical contact to the mid-point of a sampling interval, min,
V_t = total volume of the collection medium, L,
V_s = volume of discrete sample removed from the collection medium, L, and
V_i = volume of collection medium at T_i, L.

NOTE 14—The sample area exposed in the standard ASTM test cell (nominal 25 mm diameter) is about 5.1 cm^2, varying slightly due to the PTFE gasket.

NOTE 15—The following factors are useful in converting permeation rates: 1 $\mu g/cm^2/min = 0.17\ mg/m^2/s = 10\ mg/m^2/min$.

11.2 *Calculations for Systems Using a Continuous Flow of Fresh Collection Medium (Open Loop)*—This calculation is applicable to a system where fresh collection medium transports the permeant from the cell to the analyzer as shown in Fig. 5.

11.2.1 The concentration, C_i, of the permeant in the collection medium at any time, T_i, is directly proportional to the permeation rate, P_i. Concentration is converted to permeation rate as follows:

$$P_i = C_i F/A$$

11.2.2 The cumulative permeation at a specific time, K_t, is determines as follows. If no test chemical is detected at any sampling time, T_j, the minimum detectable concentration of permeant should be utilized as C_i. C_0 is set to 0.

$$K_t = \sum_{i=1}^{n}\left[P_{i-1}(T_i - T_{i-1}) + \frac{1}{2}(P_i - PC_{i-1})(T_i - T_{i-1})\right]$$

11.3 *Calculations for Closed Systems (Closed Loop)*—These calculations are applicable when any of the following conditions is met:

(1) Samples are withdrawn, analyzed, and replaced prior to further sampling,

(2) The volume of discrete samples is insignificant relative to the total volume (for example, microlitre aliquots),

(3) The collection medium is recirculated as in Fig. 4, or

(4) The concentration of the test chemical in the collection chamber is measured without any sample removal.

11.3.1 The average permeation rate over the period T_{i-1} to T_i is calculated as follows:

$$\bar{P} = \frac{(C_i - C_{i-1})V_t}{(T_i - T_{i-1})A}$$

11.3.2 When conditions (*1*) through (*4*) of 11.3 are met, the cumulative permeation at T_i is determined as follows. If no test chemical is detected, the minimum detectable concentration of test chemical is substituted for value of C_i.

$$K_{T_i} = \frac{C_i V_T}{A}$$

11.4 *Calculations for Closed Systems with Discrete Sampling*—These calculations are applicable when discrete samples of significant volume are removed from the collection medium.

11.4.1 If the sample volume is not replaced, the average permeation rate over the period, T_{i-1} to T_i, is calculated as follows:

$$P_i = \frac{C_i[V_T - (i-1)V_s] - C_{i-1}[V_T - (i-2)V_s]}{(T_i - T_{i-1})A}$$

11.4.2 Replenishment with fresh collection medium after each discrete sample changes the calculation to:

$$\bar{P}_i = \frac{\left[C_i - C_{i-1}\left(\frac{V_t - V_s}{V_t}\right)\right]V_t}{(T_i - T_{i-1})A}$$

11.4.3 Cumulative permeation at time T_i for a closed system with discrete sampling is determined as follows. If no test chemical is detected, the minimum detectable concentration of test chemical is substituted for value of C_i and C_j. There is no test chemical in the collection medium at time 0 ($C_0 = 0$).

$$K_{T_i} = \frac{1}{A} \times \left(C_i V_t + \sum_{j=1}^{i-1} C_j V_s\right)$$

11.5 When plotting average permeation rate as a function of time, the time coordinate is the mid-point of the interval over which the average was obtained, and is calculated as:

$$T_p = \frac{T_i + T_{i-1}}{2}$$

NOTE 16—The interval used for determining the time coordinate is to be the start time of the analysis and the end time for the analysis, not the sampling period, as the analysis period may be significantly longer than the sampling period interval and therefore not representative of the calculated aver permeation rate.

11.6 When plotting cumulative permeation as a function of time, the time coordinate is the time of the measurement, T_i.

12. Report

12.1 Identify and report the material tested, including generic name, manufacturer, product name, and a general description of the location from which the specimen was taken (for example, palm or back of glove).

12.1.1 Report and describe if the material sample has been subjected to any preconditioning procedures, including but not

limited to: abrasion, flexing, laundering, sterilization, or different temperature or humidity environments.

12.2 Report the average thickness of each material specimen to the nearest 0.02 mm (or nearest 0.001 in.). Also calculate and report the average thickness of the specimens tested for each material type.

12.3 Report weight per unit area (g/m^2) of each specimen. Also calculate and report the average weight per unit area of the specimens tested for each material type.

12.4 Report the name of the challenge chemical and its physical state as tested (that is, liquid or gas). If the test chemical is in a mixture, report the identities and concentrations of the components.

12.5 Report the test temperature (°C).

12.6 Report the test duration (min).

12.7 Report the test cell type, cell size, collection medium system (that is, open or closed loop), the collection medium, and the analytical technique used.

12.7.1 Report the minimum detectable permeation rate and the minimum detectable mass permeated for the permeation test system.

12.7.2 For the selected collection medium system, collection medium, and analytical technique, describe the technique that was applied to demonstrate the ability of the collection system and medium to collect the test chemical(s) and the limit of detection of the overall permeation test system based on the test period to be run.

12.7.3 If discrete sampling is used, report intervals over which sampling of the collection medium occurred.

12.8 Report the standardized breakthrough detection time (that is, the time at which the permeation rate reached 0.1 $\mu g/cm^2$/min). Also report the average of the standardized breakthrough detection time for each material type.

12.8.1 If discrete sampling and analysis is used for the measurement of permeation, then the standardized breakthrough time is the time at the end of the preceding sampling interval where the measured permeation rate is still below 0.1 $\mu g/cm^2$/min.

NOTE 17—As an example, if sampling is conducted discretely every 4 min and the measured permeation rate exceeds 0.1 $\mu g/cm^2$/min during the 8 to 12 min sampling interval, but is below 0.1 $\mu g/cm^2$/min during the preceding 4 to 8 min sampling interval, the standardized breakthrough time is established at 8 min.

12.8.2 If no permeation was detected or the permeation rate did not reach 0.1 $\mu g/cm^2$/min over the test period, then report the standardized breakthrough time as greater than the duration of the overall test period.

NOTE 18—As an example, if no permeation is measured at or above 0.1 $\mu g/cm^2$/min for a 480-min test, the standardized breakthrough time is reported at >480 min.

12.8.3 Calculate and report the average standardized breakthrough time by dividing the sum of all of the determined standardized breakthrough times by the number of specimens tested with the following exception:

12.8.3.1 If no permeation is measured over the test time for one or more but not all specimens, then use the time for the length of the test as the individual standardized breakthrough time for purposes of calculating an average standardized breakthrough time.

12.8.3.2 If an individual result varies by more than 20 % from the average for that data set, then a second set of test specimens shall be prepared and testing shall be repeated.

12.9 It is permitted to also report breakthrough detection time based on the overall sensitivity of the permeation system that includes the collection medium system, collection medium, and analytical technique. If breakthrough detection time is also reported, report the breakthrough detection time and the average of all breakthrough detection times as follows:

12.9.1 If discrete sampling and analysis is used for the measurement of permeation, then the breakthrough detection time is the time at the end of the preceding sampling interval where the measured permeation rate is still below the minimum detectable permeation rate.

NOTE 19—As an example, if sampling is conducted discretely every 4 min and the measured permeation rate exceeds a minimum detectable permeation rate during the 4 to 8 min sampling interval, but is below the minimum detectable permeation rate during the 0 to 4 min sampling interval, the breakthrough detection time is established at 4 min.

12.9.2 If no permeation was detected over the test period, then report the breakthrough detection time as greater than duration of the overall test period.

NOTE 20—As an example, if no permeation is measured at or above the minimum detectable permeation rate for a 480-min test, the breakthrough detection time is reported at >480 min.

12.9.3 Calculate and report the average breakthrough detection time by dividing the sum of all of the determined breakthrough detection times by the number of specimens tested with the following exception:

12.9.3.1 If no permeation is measured over the test time for one or more but not all specimens, then use the time for the length of the test as the individual breakthrough detection time for purposes of calculating an average breakthrough detection time.

12.9.3.2 If an individual result varies by more than 20 % from the average for that data set, then a second set of test specimens shall be prepared and testing shall be repeated.

12.10 Report the greater of either the steady-state or maximum permeation rate, if any permeation is measured, for each tested specimen and as the average for all tested specimens in $\mu g/cm^2$/min.

12.10.1 Report a steady-state permeation rate if the permeation rate appears to level off or reach equilibrium for at least 10 % of the overall test time as defined by a percent change in the individual permeation rate being no more than 5 %.

12.10.1.1 If steady-state permeation is reached, indicate that the report permeation rate is a steady-state permeation rate.

12.10.2 Report the maximum permeation rate if steady-state permeation rate does not occur and indicated the reported permeation rate as a maximum permeation rate.

12.10.3 If no permeation is measured above a permeation rate of 0.1 $\mu g/cm^2$/min, then report the permeation rate for the specimen as <0.1 $\mu g/cm^2$/min.

12.10.4 If no permeation is measured above a permeation rate of 0.1 μg/cm^2/min over the test time for one or more but not all specimens, then use 0.1 μg/cm^2/min as the individual permeation rate for those samples for purposes of calculating an average permeation rate.

12.10.5 If breakthrough detection time is to be reported and if no permeation is measured, then report the permeation rate for the specimen as less than the minimum detectable permeation rate.

12.10.6 If no permeation is measured for one or more but not all specimens, and if breakthrough detection time is being reported, then use the minimum detectable permeation rate as the individual permeation rate for purposes of calculating an average permeation rate.

12.10.7 If an individual result varies by more than 20 % from the average for that data set, then a second set of test specimens shall be prepared and testing shall be repeated.

12.11 It is permitted to report the cumulative permeation in μg/cm^2 in addition to standardized breakthrough time and permeation rate.

12.11.1 Report cumulative permeation for each specimen and as the average for all tested specimens, along with the specific time interval for which it is measured.

12.11.2 If no cumulative permeation is measured over a specific time interval, report the cumulative permeation as less than the minimum detectable cumulative permeation.

12.11.3 If no cumulative permeation is measured for one or more but not all specimens, then use the minimum detectable cumulative permeation for the purposes of calculating an average cumulative permeation.

12.11.4 If an individual result varies by more than 20 % from the average for that data set, then a second set of test specimens shall be prepared and testing shall be repeated.

12.12 Plot the permeation rate as a function of time for each specimen tested. Provide a copy of this graphical presentation for each replicate

12.13 Report any observations of specimen degradation that occurs as the result of exposure during chemical permeation testing.

12.13.1 Report if a specimen degrades rapidly after initial contact with the test chemical, such that no meaningful permeation data could be obtained.

NOTE 21—Degradation is rapid if it occurs within 10 min of contact with the chemical.

12.13.2 Document the effects of specimen degradation by taking a photograph of affected specimens and include in the report.

12.14 In addition, it is acceptable to report the results according to the guidance provided in Guide F1194.

13. Precision[6]

13.1 *Background:*

13.1.1 Interlaboratory evaluation of this method was performed in 2005. Nine independent laboratories participated, each testing in triplicate, two test chemical/clothing material pairs. The test chemicals were acetone and ethanol. To avoid possible confounding effects from denaturants, the laboratories were directed to use undenatured, 95 %, reagent grade ethanol (Spectrum Chemicals, #E1029). Lab No. 6 reported having used 100 % denatured ethanol. The clothing materials were neoprene rubber and Norfoil film. Rolls of the neoprene and the film are owned by Committee F23 and maintained at a single location.[7] Specimens were cut from the rolls, randomized, and sent to each of the participating laboratories.

13.1.2 One-inch (2.54 cm) diameter test cells were used. The specified test temperature was 27 °C, and all labs reported being within ±2 °C of that specification. The method allows open or closed-loop testing, and for the tester to choose the collection medium and the analytical method. Most testing was performed open-loop with nitrogen as the collection medium and gas chromatography with flame ionization detection as the analytical method. Test duration was until a steady-state permeation rate (SSPR) could be measured or 8 h, whichever was shorter. Templates were provided for reporting data in a common format. In addition, to aid in the precision determination, each lab was asked to supply raw data and calibration curves.

13.1.3 Acetone was used with the neoprene; short standardized breakthrough times (SBT) and relatively large permeation rates were expected. Ethanol was used with the Norfoil; long standardized breakthrough times (if any) and low permeation rates (if any) were expected.

13.2 *Acetone/Neoprene:*

13.2.1 *Neoprene Thickness*—The thicknesses of three neoprene specimens were measured by each of the nine laboratories with the following results:

Average	0.44 mm
Minimum	0.42 mm
Maximum	0.47 mm

13.2.2 *Data Quality*—Precision calculations are presented for the complete data set (that is, the results from all nine labs) and for a subset of the data. Review of the data and supporting information led to the conclusion that the results from some of the labs may be flawed. The reduced data set included results from six or more labs.

13.2.3 *Full Data Set*—For acetone/neoprene, the average standardized breakthrough (SBT) was 8.8 min (n = 27). The average SSPR was 157 μg/cm^2/min (n = 27). Detailed statistics are summarized in Table 1.

13.2.4 *Reduced Data Set:*

13.2.4.1 Review of the data led to the conclusion that there may be deficiencies with the SBT data from two of the labs and that their results can be eliminated from the analysis. Lab No. 6 sampled the collection medium at 5-min intervals. No acetone was detected at 5 min, but at 10 min the permeation rate was >>0.1 μg/cm^2/min. In accordance with the method, Lab No. 6 reported SBT as 5 min. For such rapid permeation

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F23-1007. Contact ASTM Customer Service at service@astm.org.

[7] These materials may be obtained by contacting the Chair of ASTM Subcommittee F23.30.

TABLE 1 Statistical Results for Acetone/Neoprene Permeation Testing. SBT in min and SSPR in µg/cm²/min

Quantity	n	Average	S_r	S_R	CV_r	2.8*CV_r	CV_R	2.8*CV_R
SBT	27	8.8	1.0	3.1	0.1	0.3	0.4	1.0
SBT(red. data set)	21	8.7	0.8	2.4	0.1	0.2	0.3	0.8
SSPR	27	157	16	238	0.1	0.3	1.5	4.3
SSPR (red. data set)	18	81	9.2	60	0.1	0.3	0.7	2.1

In this table:

n = number of tests,
S_r = standard deviation within a laboratory,
S_R = standard deviation among all laboratories,
CV_r = coefficient of variance within a laboratory, and
CV_R = coefficient of variance among all laboratories.

a 5-min interval was inadequate and, for this reason, Lab No. 6's SBT was dropped from the analysis. Lab No. 2's results are considered suspect and were dropped from the analysis. With the acetone, Lab No. 2's SBTs were the approximately twice the mean, and with ethanol/Norfoil Lab No. 2 reported SBT and SSPR were far different from all the other labs. Lab No. 2 did not provide calibration or other backup data for clarification of its findings.

13.2.4.2 For SSPR, Lab No. 1 and Lab No. 7's SSPRs were considerably lower and higher, respectively, than those reported by the other labs. No calibration data or other backup information was provided for clarification of their findings, and consequently the data from these two labs was dropped from this analysis.

13.2.4.3 Calculation of the average SBT without the Lab No. 2 and Lab No. 6 resulted in a value of 8.6 min (n = 21). Calculation of the average and precision of SSPR without the Lab Nos. 1, 2, and 7 results yields a value of 81.2 µg/cm²/min (n = 18). Details are in Table 1.

13.3 *Ethanol/Norfoil Film:*

13.3.1 *Norfoil Thickness*—The thicknesses of three Norfoil film specimens were measured by each of eight laboratories with the following results:

Average	0.10 mm
Minimum	0.09 mm
Maximum	0.11 mm

13.3.2 *Permeation Test Results:*

13.3.2.1 For ethanol/Norfoil, most labs did not detect permeation >0.1 µg/cm²/min and therefore did not report a SBT. Similarly, most labs did not report a SSPR. Consequently, it is not possible to present a quantitative precision statement. Not including Lab No. 2's results, which as mentioned above were considered suspect, seven labs did not find a permeation rate >0.1 µg/cm²/min for the duration of their testing. Four of those labs tested for 480 min; two labs for 440 min; and one lab for 100 min. One lab reported an average SBT of 216 min.

13.3.2.2 For ethanol/Norfoil, six labs reported not detecting any permeation. One lab reported a SSPR of 0.09 µg/cm²/min; and the lab that reported the 216-min SBT found a SSPR of 0.30 µg/cm²/min.

14. Keywords

14.1 chemical protective clothing; permeation; protection clothing; protective clothing materials

APPENDIX

(Nonmandatory Information)

X1. PROCEDURE FOR MEASURING THE SENSITIVITY OF OPEN-LOOP PERMEATION TEST SYSTEMS (alternative procedures may be used)

X1.1 Any procedure for establishing the sensitivity of an open-loop system for measuring the permeation should involve the following considerations:

X1.1.1 Baseline response of the detector for a blank permeation cell (that is, a cell containing an inert and impermeable material such as aluminum foil between the collection and test chemical chambers) but no test chemical.

X1.1.2 Detector response to a known concentration of a standard calibration chemical in the collection medium.

X1.1.3 Detector response to the test chemical.

X1.2 Fig. X1.1 is a schematic drawing of one possible configuration of a system for measuring the system sensitivity as well as calibrating the system for the test chemical. The system uses a gaseous collection medium which is directed into two permeation cells operating in parallel. The first cell (Cell 1) is used as the blank to establish the baseline response of the detector. The second cell (Cell 2) is used to assess sensitivity to the test chemical. Each cell contains a piece of aluminum foil or other inert and impermeable material. (See Note X1.1.) A standard calibration gas (toluene) is also used. Flows of the collection media from each permeation cell and the standard calibration gas are selectively directed to the detector.

NOTE X1.1—A piece of aluminum foil, or other material known to be inert and impermeable to the test chemical, is placed in the permeation test cells in lieu of the protective clothing material. This arrangement allows collection medium and test chemical to mix in a manner simulating

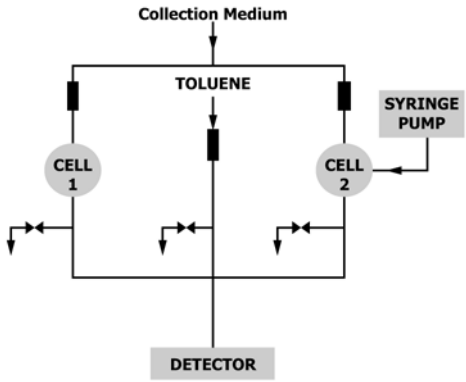

FIG. X1.1 Permeation System Setup (for Sensitivity Determination)

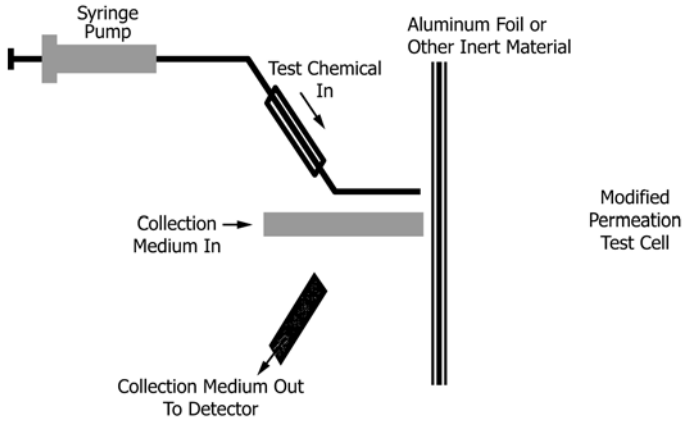

FIG. X1.2 Test Setup to Determine Permeation System Sensitivity

permeation of the chemical through the protective clothing material.

X1.3 Fig. X1.2 illustrates the second permeation cell and one approach for injecting the test chemical into the cell at a constant, measurable rate. This practice requires modification of the standard permeation cell to include three ports:

X1.3.1 One port is used for introduction of the collection medium near the surface of the inert material.

X1.3.2 One port is used for introduction of the test chemical near the surface of the inert material.

X1.3.3 One port for removal of the collection medium/mixture from the cell.

X1.4 The test chemical can be delivered to the second permeation test cell using any method which can provide a controlled, measurable rate. A syringe pump may be employed for this purpose. The flow rate of the collection medium through both the blank cell and the test cell should be calibrated with a standardized flowmeter at the outlet of the cell before beginning the test. With the collection medium flow rate, and the rate of test chemical introduction to the collection chamber, the theoretical concentration of the test chemical in the outflowing collection medium can be calculated as follows:

$$C = (d \times MV \times PR)/(MW \times F)$$

where:

C = test chemical concentration, μg/cm^3,
d = test chemical density (at the test temperature), g/cm^3,
MV = molar volume (at the test temperature), cm^3/mol,
PR = rate of delivery of the test chemical into the collection chamber, μg/min,
F = flow rate of collection medium, cm^3/min, and
MW = molecular weight, g/mol.

X1.5 Successive, discrete increases in the rate of test chemical introduction can be used to find the lower limit of detection for the permeation system. The lower detectable rate should be twice the baseline noise level of the system with the blank cell in place.

X1.6 Permeation system calibration factors and relative sensitivity may be determined by subtracting the baseline response from responses for both the test chemical and the standard calibration chemical. The ratio of these adjusted detector responses can then be used to determine permeant concentrations when the calibration chemical is also used as an internal standard during the actual permeation test.