



Standard Test Method for Permeation of Liquids and Gases through Protective Clothing Materials under Conditions of Continuous Contact¹

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 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.

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](#), Test Method [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 stated 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 and health practices and determine the applica-*

bility of regulatory limitations prior to use. Specific precautionary statements are given in Section 7.

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](#)
- [E171 Practice for Conditioning and Testing Flexible Barrier Packaging](#)
- [E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods](#)
- [E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)
- [F903 Test Method for Resistance of Materials Used in Protective Clothing to Penetration by Liquids](#)

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.

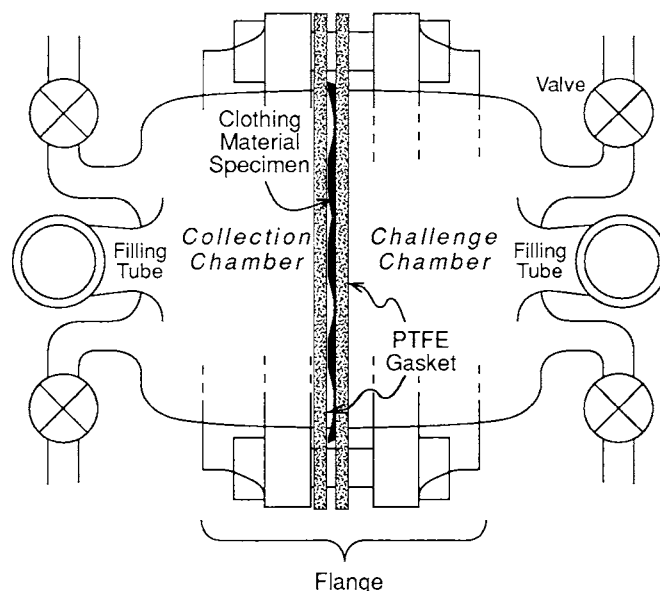


FIG. 1 ASTM Permeation Test Cell

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

2.2 ISO Standard:

ISO 6529 Protective Clothing—Determination of Resistance of Protective Clothing Materials to Permeation by Liquids and Gases³

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. 6.) 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.

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

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

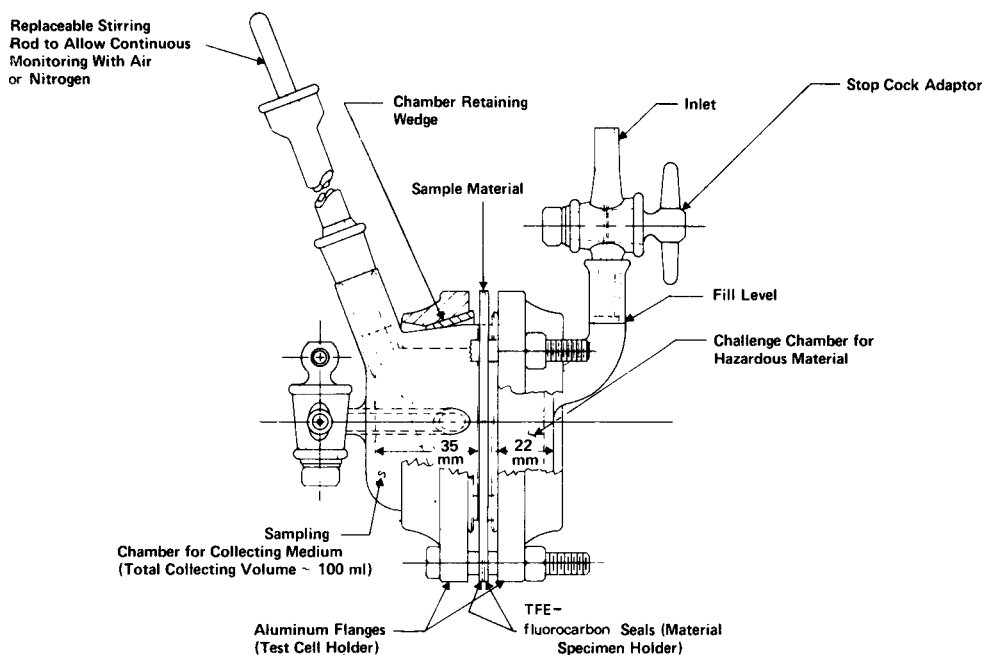


FIG. 2 Alternative Permeation Test Cell Design

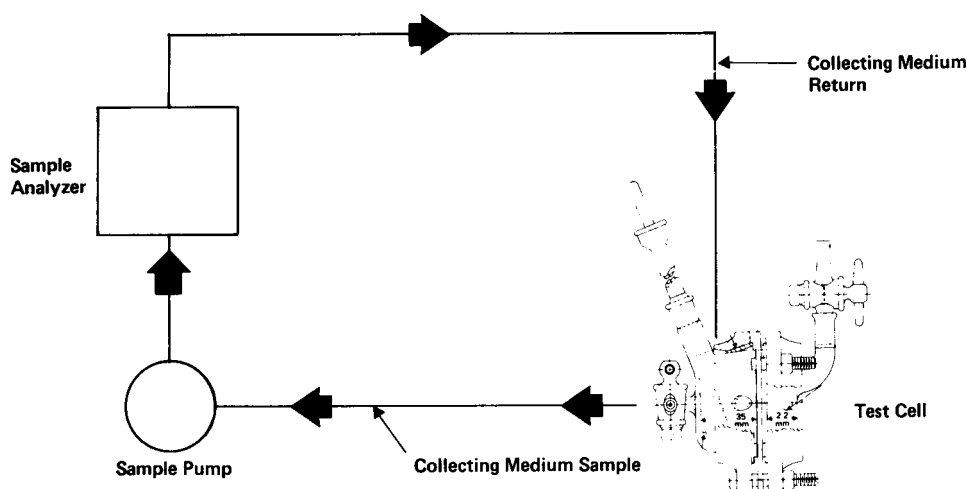


FIG. 3 Example Set-up for Continuous Collecting Medium Sample Withdrawal, Analysis, and Return

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

3.1.10.1 *Discussion*—Voids include gaps, pores, holes and imperfections in closures, seams, interfaces and protective clothing materials. Penetration does not require a change in 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.

3.1.11 *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.

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

3.1.12 *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.

3.1.13 *standardized breakthrough time, n*—the time at which the permeation rate reaches $0.1 \mu\text{g}/\text{cm}^2/\text{min}$.

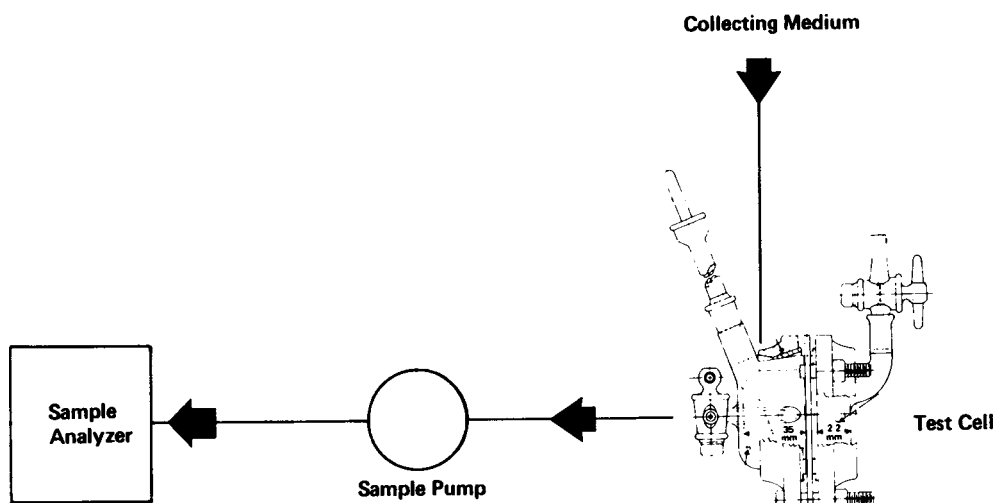


FIG. 4 Example Set-up for Continuous Flow of Fresh Collecting Medium

3.1.14 *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.

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

3.1.15.1 *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).

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, 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, and the permeation rate 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, arm shields, aprons, suits, hats, 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.

5.2 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 better barriers.

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.

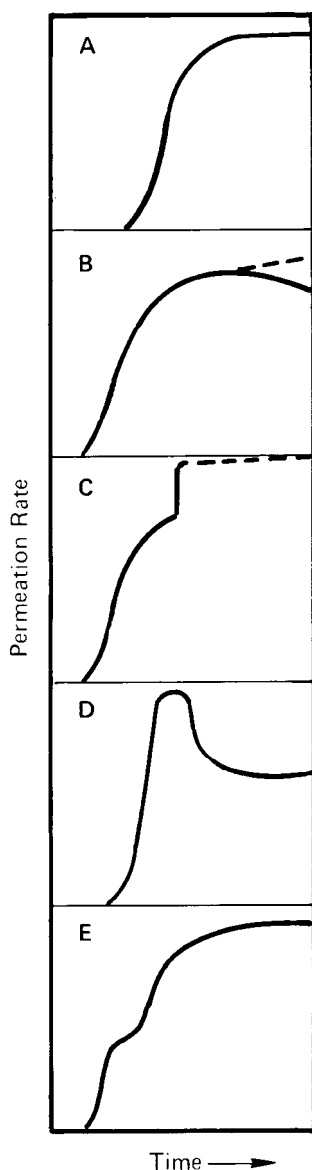
5.2.1 The reporting of a standardized breakthrough time greater than a specific time period means that the test chemical has not permeated the specimen at a rate exceeding $0.1 \mu\text{g}/\text{cm}^2/\text{min}$ in the designated time. Permeation may or may not have occurred at a lower rate during this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

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 should be capable of measuring the concentration of the test chemical in the collection medium at, or below, levels consistent with the standardized breakthrough time value specified in 3.1.13 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



NOTE 1—Fig. 5 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. 5 Five Types of Permeation Behavior

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.

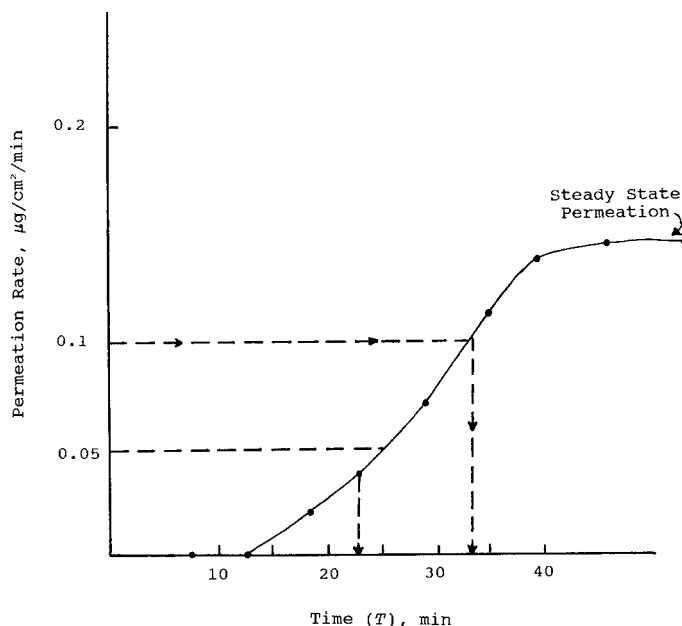


FIG. 6 The Breakthrough Detection Time for a method sensitivity of 0.05 $\mu\text{g}/\text{cm}^2/\text{min}$ is 23 minutes. The Standardized Breakthrough Detection Time is 33 minutes. The Steady State Permeation Rate is approximately 0.15 $\mu\text{g}/\text{cm}^2/\text{min}$.

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 These test procedures are also a part of ISO 6529. ISO 6529 provides a harmonized standard that also permits using some practices commonly followed in Europe for permeation testing, for example, using a breakthrough time normalized at a permeation rate of 1.0 $\mu\text{g}/\text{cm}^2/\text{min}$ instead of 0.1 $\mu\text{g}/\text{cm}^2/\text{min}$ as used in this method. For this reason, the reporting of all permeation data must include the method that is used in the testing. Guide [F1194](#) provides guidance on 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.

6.3.1 *Liquid Test Chemical*, for liquid chemicals, the test cell,⁴ shown in Fig. 1, is constructed of two sections of straight glass pipe, each nominally sized to a 25.4-mm (1.0-in.) diameter.⁵ Materials other than glass may be used. Such materials would be required for tests involving chemicals (for example, hydrofluoric acid) which are incompatible with glass. The section that is designated to contain the test chemical is 25.4 mm (1.0 in.) in length. The second section, which is designated to contain the collection medium, is 32 mm (1.2 in.) or less in length.

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

6.3.1.2 Inlet and outlet ports, with stopcock valves, if desired, are added to each chamber to enable the introduction and withdrawal of test chemical and collection medium, if appropriate. The collection medium inlet tube should direct the collection medium directly towards the center of the clothing material specimen. The inside diameter of tubing, ports, stopcocks, etc. should be at least 2 mm (0.08 in.) to prevent undesirable pressure differences in the system.

6.3.1.3 Each chamber may also be equipped with a straight bore, standard taper spout. This spout may be useful for adding and removing test chemical and collection medium. The spouts may also be used to introduce stirrers into the chambers.

6.3.1.4 Upon assembly, the clothing material is clamped between the two chambers by means of a yoke having at least three bolts.⁶ Two PTFE gaskets having smooth, rounded edges are used at the joint, with the clothing material between them.⁷

6.3.2 *Discussion*—The bolts shall be tightened with sufficient torque to prevent leakage of the test chemical or the collection medium but avoid damage to the clothing material or the test cell.

6.3.2.1 Leak-tight connections to the collection chamber inlet and outlet tube must be made. In addition, all tubing coming into contact with the test chemical should be made from material that does not absorb or react with the test chemical. Glass, PTFE or stainless steel can be used in most cases. Connections of external tubing to the glass inlet and outlet ports of the test cell chambers can be made via PTFE pressure-fit union connectors.⁸

6.3.2.2 In closed-loop tests where increased analytical sensitivity is required, a shorter length chamber may be used 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. In open-loop tests, lower collection medium flow rates will 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.

6.3.2.3 Liquid test chemicals that are mixtures must be stirred to minimize concentration gradients. Stirring may be effected by a stirring rod inserted through the fill spout or a magnetic stirrer. If there is not a good seal of the shaft of the rod and the spout, evaporation of the chemical can occur, reducing its volume and potentially changing its composition.

6.3.2.4 For a liquid collection medium that is not circulated, the two chambers of the test cell must permit the mixing, withdrawal and replenishment of the collection medium during the test.

6.3.3 For gaseous test chemicals, the test cell can consist of two chambers one of which allows the gaseous test chemical to be circulated from its reservoir. Flow must be such that the composition and concentration of the gas in the test chamber does not change with time, and the test gas in the chamber is well mixed.

6.4 *Alternative Test Cell*—Alternative permeation test cells may be used, provided that the results are reported as prescribed in Section 12. The cells and configuration described above and shown in Fig. 1, however, are the standards. If a different cell is used, it must be documented as described in Section 12. An alternative design that has been documented is shown in Fig. 2.

6.5 *Constant Temperature Chamber or Bath*, used to maintain the test cell within $\pm 1.0^{\circ}\text{C}$ of the test temperature. The standard temperature for this test is 27°C . Condition all test materials, including the test cells and chemicals, in the chamber(s) of bath(s) prior to testing.

6.6 *Circulating pump*, if appropriate, used to transport the collection medium, or test chemical, or both, through the test cell. All parts contacting the test chemical or fluid containing it 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.

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, may 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 $\pm 0.5^{\circ}\text{C}$ must 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.

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.

⁴ The test cell as shown is available from Pesce Lab Sales, P.O. Box 235, 226 Birch St., Kennett Square, PA 19348.

⁵ Sections of borosilicate glass pipe, available from Corning Glass, Catalog No. 72-0702 (1-in. length), or equivalent, are satisfactory for this purpose.

⁶ Flanges are available from Corning Glass, Catalog Nos. 72-9062 (aluminum) or 72-9654 (cast iron).

⁷ Gasket is available from Corning Glass, Catalog No. 72-9256.

⁸ PTFE union connectors suitable for making connections between external tubing and glass inlet/outlet tubes on the test cell are available from Berghoff America, 3773 NW 126th Ave, Building 1, Coral Springs, FL 33065, <http://www.berghoffusa.com/>.

7.1.2 For corrosive or otherwise hazardous chemicals, involved personnel should, as a minimum, 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 may consist of either a single layer or a composite of multiple layers that is representative of an 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 proposed design of an item 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 proposed design, seams are specified, 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.

8.2 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.

8.3 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 may be worn when handling specimens.

8.5 To avoid affecting permeation measurements, a 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 and/or the collection chamber should be measured. It may be necessary to pre-condition 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 measurement of the test chemical over the range of concentra-

tions that is consistent with 5.3.1, without exceeding the maximum concentration limits within the system as defined in 8.5.

8.7.1 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.

8.7.2 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.

8.7.3 In open-loop testing, the system shall have a sensitivity of at least $0.1\mu\text{g}/\text{cm}^2/\text{min}$. (See Appendix X1.)

8.7.4 In closed-loop testing, the system shall have a minimum sensitivity to detect a permeation rate of $0.1\mu\text{g}/\text{cm}^2/\text{min}$ over a five minute 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 $100\text{ cm}^3/\text{min}$. Higher flow rates are preferred within the constraints imposed by analytical sensitivity, temperature control, and pressure gradients in the system. (A minimum collection medium flow rate of $300\text{ cm}^3/\text{min}$ is required for a nominal 50-mm diameter cell.)

8.8.1 *Discussion*—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.

8.9 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 %. Over-pressurization 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.

8.10 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.

8.11 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 $27 \pm 2^\circ\text{C}$ ($81 \pm 4^\circ\text{F}$) and a relative humidity of 30 to 80 % as described in Specification E171.

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 centimetre by weighing the specimen on an analytical balance (± 2 mg) and dividing by the area (± 0.4 cm²), 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 specimen in the test cell and assemble as shown in Fig. 1 and described in 6.3.1 and 6.3.3.

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 to which the normally inside surface of the material specimen is exposed. 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. 3 and 4). The cell, along with the collection medium, should be maintained at the test temperature for at least 30 minutes 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 Discussion following 8.8.)

10.7 Initiate sampling of the collection medium, either continuously or discretely, and continue on a predetermined schedule throughout the test duration. Promptly complete

analysis of each sample for test chemical content. 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. (See Note 2.)

NOTE 2—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 minutes of contact.

10.10 Discontinue sampling and terminate the test after one or more of the following conditions is met (see Fig. 5):

10.10.1 Steady state permeation is reached (Fig. 5A and Fig. 5E).

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 five minutes 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 five minutes 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. 5C).

10.10.3 A maximum rate is reached and a steady-state permeation rate is not achieved within 30 minutes after the maximum rate occurs. (Fig. 5B and Fig. 5D).

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.

11. Calculations⁹

NOTE 3—Data from this method may be analyzed by following Practice F2815.

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

where:

- 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 , $\mu\text{g/L}$,
- 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 , $\mu\text{g/cm}^2/\text{min}$,
- $\bar{P}_i$ = average permeation rate for the interval T_{i-1} to T_i , $\mu\text{g/cm}^2/\text{min}$,
- 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 4—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 5—The following factors are useful in converting permeation rates: $1 \mu\text{g/cm}^2/\text{min} = 0.17 \text{ mg/m}^2/\text{s} = 10 \text{ mg/m}^2/\text{min}$.

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. 4.

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_i , is determined as follows. If the no test chemical is detected at any sampling time, T_i , the minimum detectable concentration of permeant should be utilized as C_i . C_0 is set to 0.

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

11.3 *Calculations for Closed Systems (Closed Loop)*—These calculations are applicable when any of the following conditions are 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. 3, 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 .

$$K_{Ti} = \frac{C_i V_t}{A}$$

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 ($C_0 = 0$).

⁹ Public domain software for making the calculations in this Section is available from the NIOSH National Personal Protective Technology Laboratory (NPPTL), 626 Cochran's Mill Road, P.O. Box 18070, Pittsburgh, PA, USA, 15236. Also on WWW at <http://www.cdc.gov/niosh/npptl/default.html>. USA telephone 412-386-6885

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

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 6—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 average 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, nominal thickness, and a general description of the location from which the specimen was taken (for example, palm or back of glove).

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, 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 ($^{\circ}\text{C}$).

12.6 Report the test duration (h).

12.7 For each protective clothing material tested, report the collection medium system (that is, open- or closed-loop), the collection medium, and the analytical technique used.

12.8 For each test in which chemical permeation was detected, report the standardized breakthrough detection time (that is, the time at which the permeation rate reaches $0.1 \mu\text{g}/\text{cm}^2/\text{min}$).

12.8.1 If permeation is first detected at a permeation rate $>0.1 \mu\text{g}/\text{cm}^2/\text{min}$, report the breakthrough time and the rate at which permeation was detected.

12.8.2 If permeation is first detected at a rate $<0.1 \mu\text{g}/\text{cm}^2/\text{min}$, the breakthrough detection time and rate at which permeation was detected may be reported.

12.8.3 If no permeation was detected or the permeation rate did not reach $0.1 \mu\text{g}/\text{cm}^2/\text{min}$, report the fact that no permeation was detected at either the minimum detectable permeation or $0.1 \mu\text{g}/\text{cm}^2/\text{min}$.

12.9 If the permeation process reached steady state, report the steady state permeation rate in $\mu\text{g}/\text{cm}^2/\text{min}$ for each material specimen. Also report the average of the steady-state permeation rates for each material type.

12.10 Report the maximum permeation rate in $\mu\text{g}/\text{cm}^2/\text{min}$. Also report the average of the maximum permeation rates for each material type.

12.11 Plot the permeation rate as a function of time for each clothing specimen tested. A copy of this graphical presentation for each of the triplicate tests should be included as part of the test report.

12.12 If a protective clothing material degrades rapidly after initial contact with the test chemical, such that no meaningful permeation data could be obtained, report this fact.

12.13 Report the cumulative permeation in micrograms per square centimetre at the time specified for the entire exposure period, if not specified.

12.14 In addition, the results may be presented according to the guidance provided in Guide F1194.

13. Precision¹⁰

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 #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.¹¹ 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 $\pm 2^{\circ}\text{C}$ 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 eight hours, 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:

¹⁰ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F23-1007.

¹¹ These materials may be obtained by contacting the chairman of ASTM Subcommittee F23.30.

TABLE 1 Statistical Results for acetone/Norprene permeation testing. SBT in minutes and SSPR in $\mu\text{g}/\text{cm}^2/\text{min}$

Quantity	n	Average	S_r	S_R	CV_r	$2.8 \cdot CV_r$	CV_R	$2.8 \cdot CV_R$
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
 CV_R = coefficient of variance among all laboratories

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 minutes ($n=27$). The average SSPR was $157 \mu\text{g}/\text{cm}^2/\text{min}$ ($n=27$). 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 #6 sampled the collection medium at five minute intervals. No acetone was detected at 5 minutes but at 10 minutes the permeation rate was $>>0.1 \mu\text{g}/\text{cm}^2/\text{min}$. In accordance with the method, Lab #6 reported SBT as five minutes. For such rapid permeation a five-minute interval was inadequate and for this reason, Lab #6's SBT was dropped from the analysis. Lab #2's results are considered suspect and were dropped from the analysis. With the acetone, Lab #2's SBT were the approximately twice the mean, and with ethanol/Norfoil Lab #2 reported SBT and SSPR were far different from all the other labs. Lab #2 did not provide calibration or other backup data for clarification of its findings.

13.2.4.2 For SSPR, Lab #1 and Lab #7's SSPR 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 #2 and Lab #6 resulted in a value of 8.6 minutes ($n=21$). Calculation of the average and precision of SSPR without the Lab #1, #2 and #7 results yields a value of $81.2 \mu\text{g}/\text{cm}^2/\text{min}$ ($n=18$). 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 $>0.1 \mu\text{g}/\text{cm}^2/\text{min}$ 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 #2's results, which as mentioned above were considered suspect, seven labs did not find a permeation rate $>0.1 \mu\text{g}/\text{cm}^2/\text{min}$ for the duration of their testing. Four of those labs tested for 480 minutes; two labs for 440 minutes; and one lab for 100 minutes. One lab reported an average SBT of 216 minutes

13.3.2.2 For ethanol/Norfoil, six labs reported not detecting any permeation. One lab reported a SSPR of $0.09 \mu\text{g}/\text{cm}^2/\text{min}$; and the lab that reported the 216-minute SBT found a SSPR of $0.30 \mu\text{g}/\text{cm}^2/\text{min}$.

14. Keywords

14.1 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)

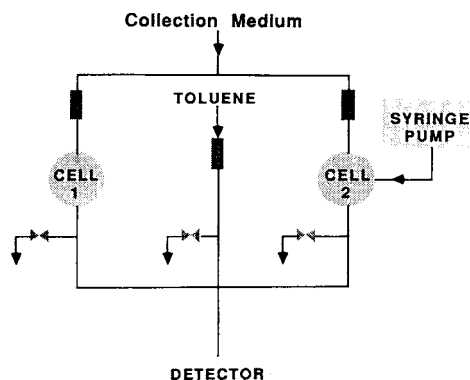


FIG. X1.1 Permeation System Set Up (for Sensitivity Determination)

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 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.

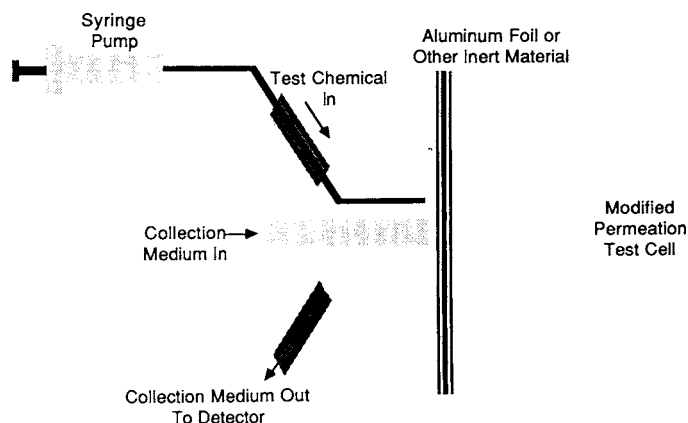


FIG. X1.2 Test Set Up to Determine Permeation System Sensitivity

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 flowrate, and the rate of test chemical introduction to the collection chamber, the theoretical concentration of the test chemical in the out-flowing collection medium can be calculated as follows:

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

where:

C = test chemical concentration, $\mu\text{g}/\text{cm}^3$,
 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, $\mu\text{g}/\text{min}$,
 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.

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