

Designation: F739 – 20

# Standard Test Method for Permeation of Liquids and Gases Through Protective Clothing Materials Under Conditions of Continuous Contact<sup>1</sup>

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 ( $\varepsilon$ ) 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 appro-

priate 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:<sup>2</sup>
- 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

<sup>&</sup>lt;sup>1</sup> 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.

Current edition approved Nov. 1, 2020. Published November 2020. Originally approved in 1981. Last previous edition approved in 2012 as F739 – 12. DOI: 10.1520/F0739-20.

<sup>&</sup>lt;sup>2</sup> 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)<sup>3</sup>

## 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).

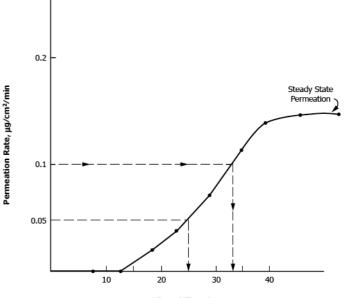
3.1.3 *closed-loop, adj*—refers to a testing mode in which there is no change in the volume of the collection medium except for sampling.

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.



Time (*T*), min

FIG. 1 The Breakthrough Detection Time for a Method Sensitivity of 0.05 μg/cm<sup>2</sup>/min is 25 min. The Standardized Breakthrough Detection Time is 33 min. The Steady-State Permeation Rate is Approximately 0.15 μg/cm<sup>2</sup>/min.

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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  $1.0 \,\mu\text{g/cm}^2/\text{min}$ .

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.

 $<sup>^{3}\,\</sup>text{The}$  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  $0.1 \,\mu\text{g/cm}^2/\text{min}$ .

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  $0.1 \,\mu\text{g/cm}^2/\text{min}$  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  $0.05 \,\mu\text{g/cm}^2/\text{min}$ , 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

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(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.STM F

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  $\mu$ g/cm<sup>2</sup>/min, it is acceptable to continue the testing and also report a normalized breakthrough time at a permeation rate of 1.0  $\mu$ g/cm<sup>2</sup>/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  $\pm 0.5$  mg, shall be used to determine weight per unit area of each test specimen.

6.3 *Test Cell*—The test apparatus consists of a twochambered 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.

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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.<sup>4</sup> 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.

<sup>4</sup> 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  $\pm 1.0$  °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.

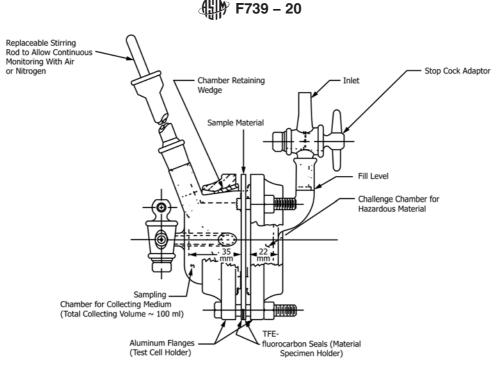
Note 10—If a circulating pump is used, care should be taken to avoid inducing pressure which may deform or damage the test specimen.

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.

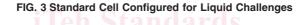
6.8 *Thermometer or Thermocouple*, used to measure the temperature of the constant-temperature chamber (or bath), or the collection chamber of the test cell, or both. A calibrated device accurate to  $\pm 0.5$  °C shall be used.

## 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.



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  $0.05 \text{ }\mu\text{g/cm}^2/\text{min}$ . (See Appendix X1.)

8.7.5 In closed-loop testing, the system shall have a minimum sensitivity to detect a permeation rate of  $0.05 \,\mu\text{g/cm}^2/\text{min}$  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  $100 \text{ cm}^3/\text{min}$ . Higher flow rates are preferred within the constraints imposed by analytical sensitivity, temperature control, and pressure gradients in the system.

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  $300 \text{ cm}^3/\text{min}$ . Higher flow rates are preferred within the constraints imposed by analytical sensitivity, temperature control, and pressure gradients in the system.

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  $27 \pm 2 \text{ °C} (81 \pm 4 \text{ °F})$  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 ( $\pm 2$  mg) and dividing by the area ( $\pm 0.4$  cm<sup>2</sup>), 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