Designation: F1383 - 20

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

This standard is issued under the fixed designation F1383; 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 resistance to permeation under the condition of intermittent contact of the protective clothing material with liquid or gaseous chemicals. Resistance to permeation and penetration under conditions of continuous contact should be determined by Test Methods F739 and F903, respectively. In certain situations, the permeation of liquids through protective clothing materials can be measured using a permeation cup following Test Method F1407. An undesirable change in the physical properties of protective clothing materials is called degradation. Methods for measuring the degradation of rubbers, plastics, and coated materials 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 intermittent 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 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.
- 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 hazard 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

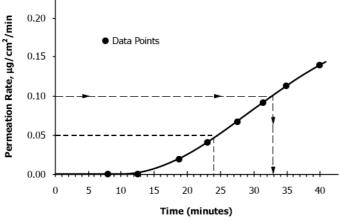
D1777 Test Method for Thickness of Textile Materials

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.

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<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.



Note 1—In an intermittent contact test, it is possible that the permeation rate will exceed, then go below, and then again exceed a permeation rate of  $0.1~\mu g/cm^2/min$ . If this occurs, the standardized breakthrough time is the first occurrence of the permeation rate exceeding  $0.1~\mu g/cm^2/min$ .

FIG. 1 The Breakthrough Detection Time for a Method Sensitivity of 0.05 μg/cm²/min is 24 min. The Standardized Breakthrough Detection Time is 33 min.

E171/E171M Practice for Conditioning and Testing Flexible Barrier Packaging

F739 Test Method for Permeation of Liquids and Gases Through Protective Clothing Materials Under Conditions of Continuous Contact

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

ASTMF1

F1407 Test Method for Resistance of Chemical Protective Clothing Materials to Liquid Permeation—Permeation Cup Method

F1494 Terminology Relating to Protective Clothing

# 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 procedures are often specific to individual chemical and collection medium combinations. Applicable techniques can 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. (See Fig. 1.)
- 3.1.2.1 *Discussion*—The breakthrough detection time is dependent on the sensitivity of the method. (See Fig. 1 and 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 *contact time, n—in an intermittent contact test,* the duration during each cycle that the test chemical chamber side of the permeation cell is filled with the test chemical.
- 3.1.6 *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.
- 3.1.6.1 *Discussion*—Quantification of cumulative permeation enables the comparison of permeation behaviors under different intermittent and continuous contact conditions.
- 3.1.7 *cycle time, n—in an intermittent contact test,* the interval of time from the start of one contact period to the start of the next contact period.
- 3.1.8 *degradation*, *n*—a deleterious change in one or more properties of a material.
- 3.1.8.1 *Discussion*—For protective clothing materials, changes in physical properties are typically of most interest.
- 3.1.9 *minimum detectable mass permeated, n*—the smallest mass of test chemical that is detectable with the complete permeation test system.
- 3.1.9.1 *Discussion*—This value is not necessarily the sensitivity of the analytical instrument.
- 3.1.10 *minimum detectable permeation rate, n*—the lowest rate of permeation that is measurable with the complete permeation test system.
- 3.1.10.1 *Discussion*—This value is not necessarily the sensitivity of the analytical instrument.
- 3.1.11 *normalized breakthrough time, n*—the time at which the permeation rate reaches 1.0 µg/cm<sup>2</sup>/min.
- 3.1.12 *open-loop*, *adj*—refers to a testing mode in which fresh collection medium flows continuously through the collection chamber of the test cell.
- 3.1.13 penetration, n—for chemical protective clothing, the movement of a substance through voids in protective clothing material or the protective clothing item on a non-molecular level.
- 3.1.13.1 *Discussion*—Voids include gaps, pores, holes, and imperfections in closures, seams, interfaces, and protective clothing materials. Penetration does not require a change of state; solid chemicals move through voids as solids, liquids as liquids, and gases as gases. Penetration is a distinctly different mechanism from permeation.
- 3.1.14 permeation, n—for chemical protective clothing, the movement of chemical(s) as molecules through protective clothing materials by the processes of: (1) absorption of the chemical into the contact surface of the material, (2) diffusion of the absorbed molecules throughout the material, and (3) desorption of the chemical from the opposite surface of the material.

- 3.1.14.1 *Discussion*—Permeation is a distinctly different mechanism from penetration.
- 3.1.15 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.
- 3.1.16 *purge time*, *n*—*in an intermittent contact test*, the time immediately following the termination of the contact time when the test chemical is removed from the test chemical chamber side and air or nitrogen is blown over the outside surface of the protective clothing material.
- 3.1.17 *seam*, *n*—a line along which two pieces of material are joined together in protective clothing.
- 3.1.17.1 *Discussion*—Common ways that seams are constructed include sewing with thread, welding with heat, taping, gluing, or combinations thereof.
- 3.1.18 *standardized breakthrough time, n*—the first time at which the permeation rate reaches 0.1 µg/cm<sup>2</sup>/min (see Fig. 1).
- 3.1.19 *test chemical*, *n*—the solid, liquid, gas or mixture thereof, used to evaluate the performance of a protective clothing material.
- 3.1.19.1 *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). To be tested with this method, a solid must be soluble in a liquid or have a vapor pressure greater than 1 mm Hg at 25 °C.
- 3.1.20 *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, and subsequent permeation rate through replicate specimens of the material intermittently contacted with the chemical.
- 4.2 In the permeation test apparatus, the protective clothing material specimen partitions the test chemical from the collection medium.
- 4.2.1 Contact of the test chemical with the clothing material's outside surface is made intermittent by periodically adding and removing the test chemical from the test chemical chamber side of the test cell.
- 4.2.2 The collection medium is analyzed quantitatively for its concentration of the test chemical and, thereby, the amount of that chemical that has permeated the protective clothing material specimen as a function of time after its initial contact with the material.
- 4.2.3 By either graphical representation or appropriate calculations, or both, the breakthrough detection time, the standardized breakthrough time, normalized breakthrough time, and the cumulative permeation of the test chemical are determined.

### 5. Significance and Use

- 5.1 This test method is used to measure chemical permeation through specimens of protective clothing under the condition of intermittent contact of a test chemical with the specimen. In many applications, protective clothing is contacted intermittently to chemicals, not continuously as is tested by Test Method F739.
- 5.2 This test method is normally used to evaluate flat specimens and seams from finished items of protective clothing and of materials that are candidates for items of protective clothing.
- 5.2.1 Finished items of protective clothing include gloves, sleeves, aprons, suits, coveralls, hoods, boots, respirators, and the like
- 5.2.2 The phrase "specimens from finished items" encompasses seams or other discontinuous regions as well as continuous regions of protective clothing items.
- 5.2.3 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.
- 5.3 In some cases, it may be of interest to compare permeation behaviors that occur under conditions of intermittent contact with those that occur during continuous contact. Test Method F739 is recommended for measuring permeation under the conditions of continuous contact of the test chemical with the protective clothing specimen.
- 5.4 The breakthrough detection time, standardized breakthrough time, and the cumulative permeation are key measures of the effectiveness of a clothing material to be 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 and standardized breakthrough times and low amounts of cumulative permeation are characteristics of more effective barrier materials than materials with higher permeation characteristics.

Note 1—At present, there is limited quantitative information 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.

- 5.4.1 The reporting of a standardized breakthrough time greater than a specific time period does not mean that no chemical permeated through the protective clothing material since the standard breakthrough time is determined based on the permeation rate reaching a level of 0.1  $\mu$ g/cm²/min. Some chemical had already permeated the specimen prior to the reported standardized breakthrough time.
- 5.4.2 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.





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

This information has potential value when there are known maximum permitted skin exposure doses for specific chemicals.

- 5.5 The sensitivity of the test method in detecting low permeation rates or amounts of the test chemical permeated is determined by the combination of: (1) the analytical technique and collection system selected, and (2) the ratio of material specimen area to collection medium volume or flow rate.
- 5.5.1 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/cm²/min.
- 5.5.2 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.
- 5.5.3 Higher ratios of material specimen area to collection medium volume or flow rate permit earlier detection of permeation because higher concentrations of the test chemical in the collection medium will develop in a given time period, relative to those that would occur at lower ratios.
- 5.5.4 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.
- 5.5.5 The sensitivity of a closed-loop system is characterized by its minimum detectable mass permeated.
- 5.6 Comparison of results of tests performed with different permeation test systems requires specific information on the test cell, procedures, contact and purge times, and analytical techniques. Results obtained from closed-loop and open-loop testing may not be directly comparable.
- 5.7 While this method specifies standardized breakthrough time as the time at which the permeation rate reaches 0.1  $\mu$ g/cm²/min, it is acceptable to continue the testing and also report a normalized breakthrough time at a permeation rate of 1.0  $\mu$ g/cm²/min.

- 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 A group of chemicals that is commonly used in permeation testing is given in Guide F1001.
- 5.9 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 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 normal 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.

Note 4—Select test cell based on the challenge chemical and most appropriate analytical method.

- 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 5—The standard closed and flow chambers are made of glass.<sup>3</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 6—The configuration can be of two closed chambers, two flow chambers, or one closed and one flow chamber.

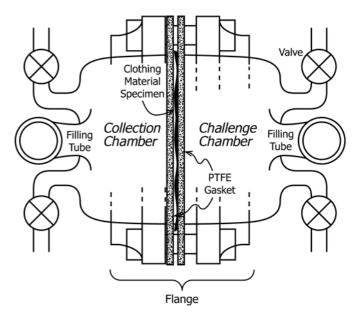
6.3.2.1 When the flow chamber contains the challenge chemical, the chemical is introduced through the longer stem that goes all the way to the end of the chamber. A shorter stem on the side of the test chamber provides the challenge chemical a means of exit from the test chamber. This mode of entry and 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 do not change over time.

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

6.3.3 Additional Information:

6.3.3.1 Make 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 by means of PTFE pressure-fit union connectors.

6.3.3.2 In closed-loop tests where increased analytical sensitivity is required, use a shorter length chamber to reduce the volume of the collection medium. This increases the sensitivity of the method by increasing the ratio of material specimen area



Note 1—The clothing material specimen is oriented such that its normally outside surface (as worn by a user) faces the test chemical chamber.

FIG. 3 ASTM Permeation Cell Configured for Intermittent Contact Testing (Top View)

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.

6.3.3.3 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.

6.3.3.4 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.

6.3.3.5 For a liquid collection medium that is not circulated, the test cell can consist of two test chambers clamped together, provided that the collection medium can be mixed, withdrawn, and replenished as needed during the test.

6.3.3.6 The test chemical side chamber may be modified to include an additional outlet port (with stopcock) positioned downward opposite the liquid chemical inlet port. Such a modification will facilitate the repeated addition and removal of liquid test chemicals.

6.4 Alternative Test Cell—Alternative permeation test cells may be used, provided that the results are reported as prescribed in Section 12. The cell and configuration described above and shown in Fig. 3, however, is the standard. If a different cell is used, it must be documented as described in Section 12.

6.5 Constant-Temperature Chamber or Bath—Used to maintain the test cell within  $\pm 1$  °C of the test temperature. The

<sup>&</sup>lt;sup>3</sup> The closed and flow chambers are available from Pesce Lab Sales, Inc. 355 N. Lincoln St, Kennett Square, PA 19348.

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, dilution, or both within the test cell.

Note 7—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. Hazards

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

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- 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 Considerations

- 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 8—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 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 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 5.5.1, 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, or when the protective clothing material is water sensitive.
- 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  $0.05 \,\mu\text{g/cm}^2/\text{min}$ . (See Appendix X1.)

- 8.7.4 In closed-loop testing, the system shall have a minimum sensitivity to detect a permeation rate of  $0.05 \,\mu\text{g/cm}^2/\text{min}$  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 cm³/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 cm³/min is required for a nominal 50 mm diameter cell.)
- 8.8.1 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. 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 chemical 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 %.
- 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  $27 \pm 2$  °C ( $81 \pm 4$  °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 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.
- Note 9—The weight per unit area, along with thickness, is a key characteristic of the material and is helpful when comparing permeation test results.
- 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. 3 and described in 6.3.1.
- 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 in a constant-temperature chamber or a water bath at 27 °C, the standard temperature for this method. Other temperatures shall be permitted to be used but must be noted in the report. The test cell must not be removed from the constant-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 on 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 testing 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 8.8.1).
- 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 permeation cell to establish the baseline values against which subsequent analytical data will be compared (see Note 10).

Note 10—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