Designation: F 739 – 99a

Standard Test Method for Resistance of Protective Clothing Materials to Permeation by Liquids or Gases Under Conditions of Continuous Contact[1]

This standard is issued under the fixed designation F 739; 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 resistance to permeation 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 F 1383. Resistance to penetration should be determined by Test Method F 903. Procedures for measuring the effects of liquid chemicals on the physical properties (that is, degradation) of rubbers, plastics, and coated fabrics are found in Test Method D 471, Test Method D 543, and Test Method D 751, respectively.

1. Scope

1.1 This test method measures the resistance of protective clothing materials to permeation by liquid or gaseous chemicals under the condition of continuous contact.

1.1.1 *Procedure A*—For use when the test chemical is a liquid that is either volatile or soluble in water.

1.1.2 *Procedure B*—For use when the test chemical is a gas.

1.2 *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 applicability of regulatory limitations prior to use. Specific precautionary statements are given in Section 7.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 471 Test Method for Rubber Property—Effect of Liquids[2]

D 543 Test Method for Evaluating the Resistance of Plastics to Chemical Reagents[3]

D 751 Test Methods for Coated Fabrics[4]

E 105 Practice for Probability Sampling of Materials[5]

E 171 Specification for Standard Atmospheres for Conditioning and Testing Flexible Barrier Materials[6]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[5]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

F 903 Test Method for Resistance of Materials Used In Protective Clothing Materials to Penetration by Liquids[7]

F 1001 Guide for Selection of Chemicals to Evaluate Protective Clothing Materials[7]

F 1194 Guide for Documenting the Results of Chemical Permeation Testing on Materials Used In Protective Clothing Materials[7]

F 1383 Test Method for Resistance of Protective Clothing Materials to Permeation by Liquids or Gases Under Conditions of Intermittent Contact[7]

2.2 *Federal Standard:*

Fed. Std. No. 191, Method 5030.2 Measurement of the

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

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[2] *Annual Book of ASTM Standards,* Vol 09.01.

[3] *Annual Book of ASTM Standards,* Vol 08.01.

[4] *Annual Book of ASTM Standards,* Vol 09.02.

[5] *Annual Book of ASTM Standards,* Vol 14.02.

[6] *Annual Book of ASTM Standards,* Vol 15.09.

[7] *Annual Book of ASTM Standards,* Vol 11.03.

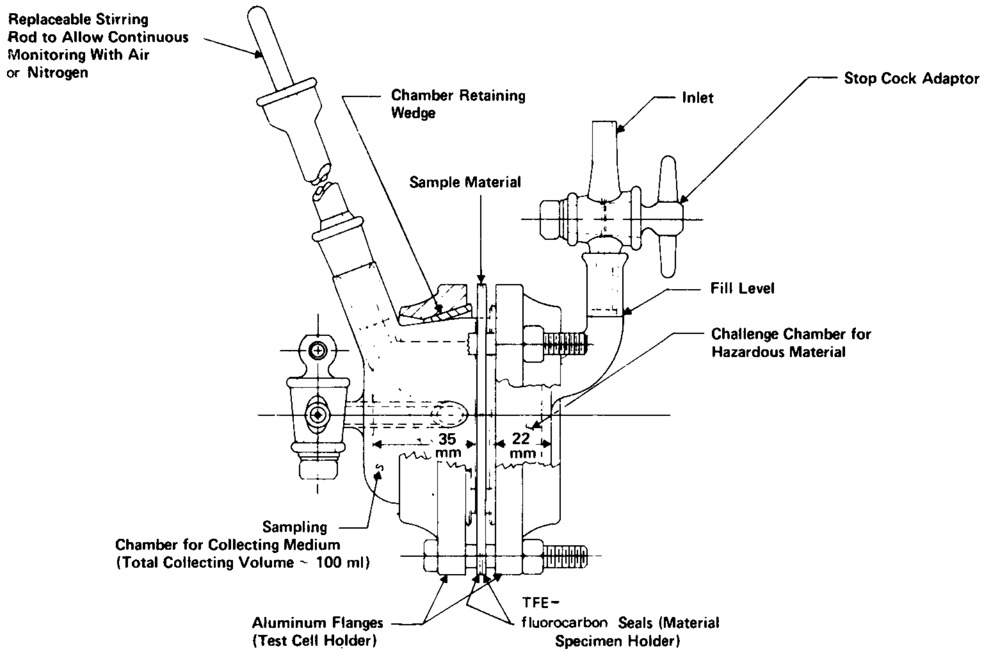


FIG. 1 ASTM Permeation Test Cell

Thickness of Materials[8]

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.

DISCUSSION—These procedures are often specific to individual chemical and collection medium combinations. Applicable techniques include UV (ultraviolet) and IR (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 start of the test to the sampling time that immediately precedes the sampling time at which the test chemical is first detected (see Fig. 5).

DISCUSSION—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 the collection medium volume is fixed.

3.1.4 *collection medium*, *n*—a liquid or gas that does not affect the measured permeation and in which the test chemical is freely soluble or adsorbed to a saturation concentration greater than 0.5 weight or volume percent.

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

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

DISCUSSION—This value is not necessarily the sensitivity of the analytical instrument.

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

DISCUSSION—This value is not necessarily the sensitivity of the analytical instrument.

3.1.8 *normalized breakthrough detection time*, *n*—in an open-loop test, the time at which the permeation rate reaches 0.1 $\mu g/cm^2$/min. In a closed-loop test, the time at which the mass of chemical permeated reaches 0.25 $\mu g/cm^2$.

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.

3.1.10 *penetration*, *n*—in a protective clothing material or item, the process by which a solid, liquid, or gas moves through closures, seams, interstices, and pinholes or other imperfections on a nonmolecular level.

3.1.11 *permeation*, *n*—the process by which a chemical moves through a protective clothing material on a molecular level.

DISCUSSION—Permeation involves the following: (*1*) sorption of molecules of the chemical into the contacted (challenge side) surface of the material, (*2*) diffusion of the sorbed molecules in the material, and (*3*) desorption of the molecules from the opposite (collection side) surface of the material.

[8] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

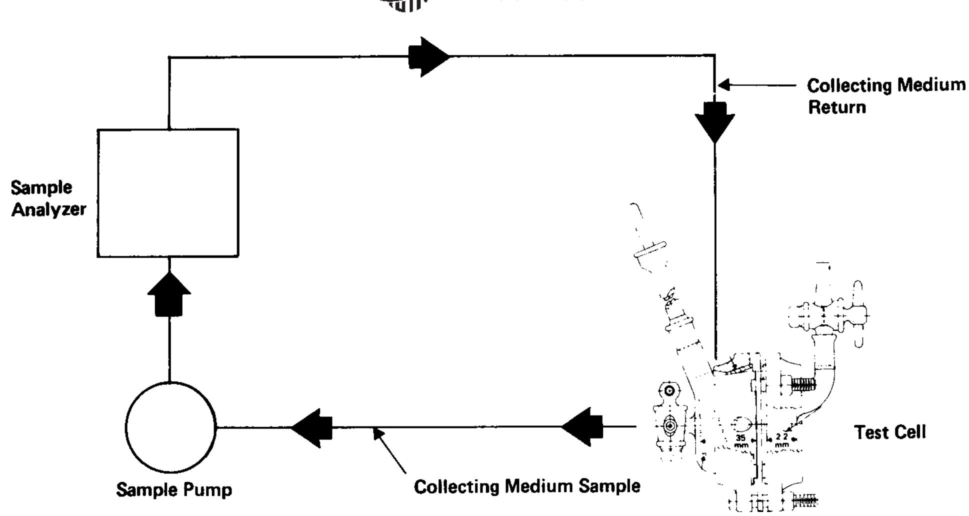


FIG. 2 Example Set-up for Continuous Collecting Medium Sample Withdrawal, Analysis, and Return

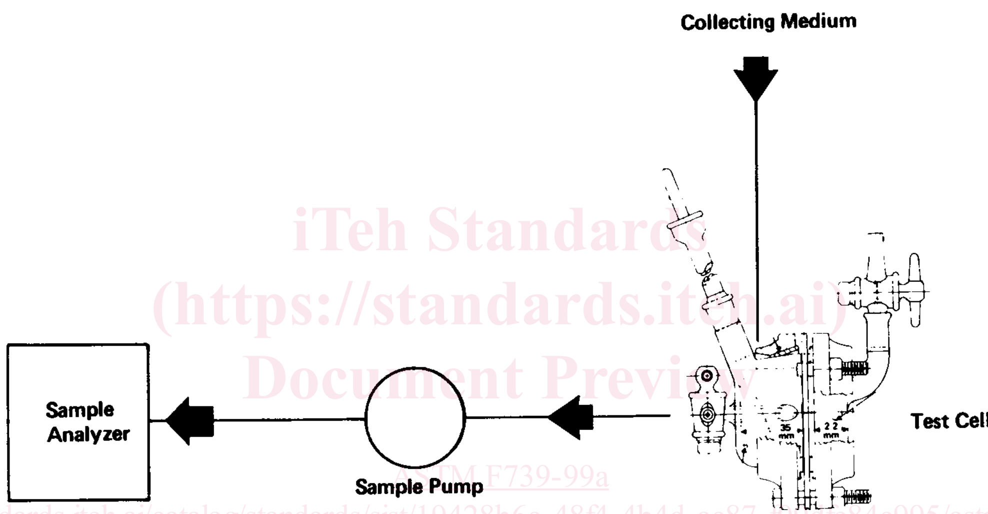


FIG. 3 Example Set-up for Continuous Flow of Fresh Collecting Medium

3.1.12 *protective clothing material*, *n*—any material or combination of materials used in an item of clothing for the purpose of isolating parts of the body from a potential hazard.

DISCUSSION—In this test method the particular hazard is permeation of a hazardous chemical.

3.1.13 *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.

3.1.14 *test chemical*, *n*—the liquid or gas that is used to challenge the protective clothing material specimen.

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 resistance of a protective clothing material to permeation by a test chemical is assessed by measuring the breakthrough detection time, normalized breakthrough detection time, and subsequent permeation rate 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.

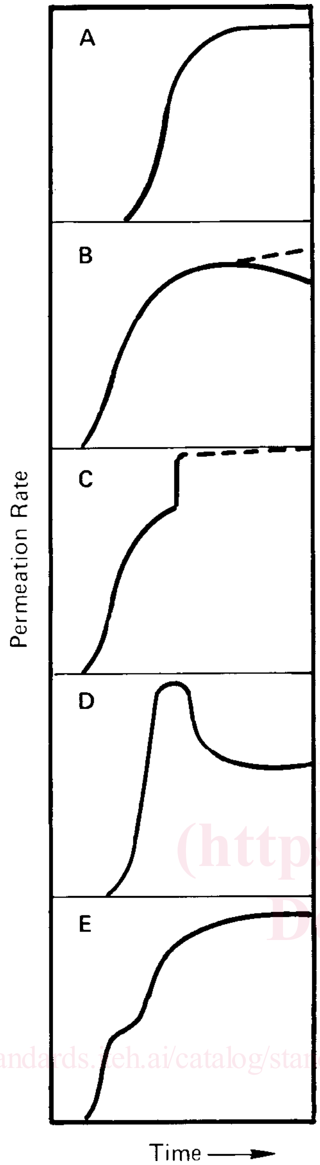
4.2.1 The collection medium, which may be liquid or gas, is analyzed quantitatively for its concentration of the chemical and thereby the amount of that chemical that has permeated the barrier as a function of time after its initial contact with the material.

4.2.2 By either graphical representation or appropriate calculations, or both, the breakthrough detection time, normalized breakthrough detection 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.



NOTE—Fig. 4 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. 4 Five Types of Permeation Behavior

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, normalized breakthrough time, and permeation rate 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 and normalized breakthrough detection times and low permeation rates are characteristics of better barriers.

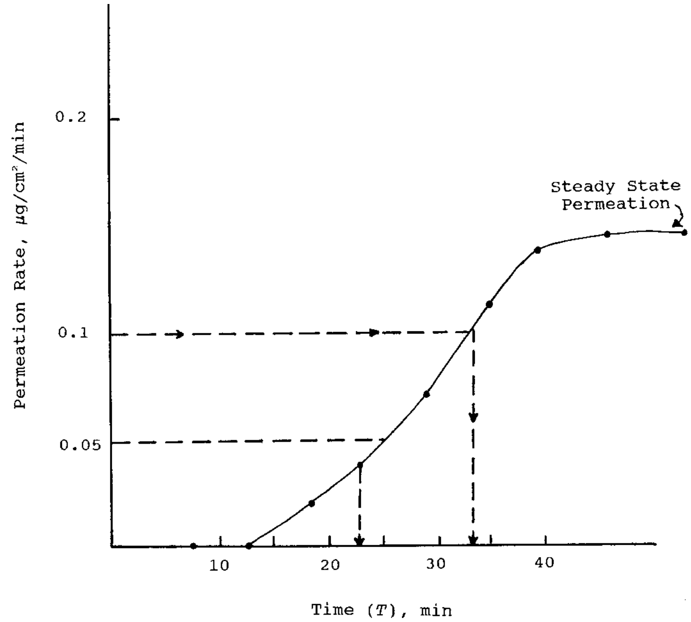

FIG. 5 The Breakthrough Detection Time for a method sensitivity of 0.05 μg/cm²/min is 23 minutes. The Normalized Breakthrough Detection Time is 33 minutes. The Steady State Permeation Rate is approximately 0.15 μg/cm²/min.

NOTE 1—At present, no quantitative information exists about acceptable levels of dermal contact. Therefore, the data obtained using this test method cannot be used to infer safe exposure levels.

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 A sensitive analytical technique permits quantitative detection of the test chemical in the collection medium at concentrations as low as parts per billion.

5.3.2 Higher ratios of material specimen area to collection medium volume or flow rate permit earlier detection of breakthrough and detection of lower permeation rates 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.

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

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

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

5.5 A group of chemicals for use in permeation testing is given in Guide F 1001.

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 Fed. Std. No. 191 Method 5030.2, 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,* the test cell,[9] as shown in Fig. 1, is constructed of two sections of straight glass pipe, each nominally sized to a 51-mm (2.0-in.) diameter.[10] 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 22 mm (0.85 in.) in length. The second section, which is designated to contain the collection medium, is 35 mm (1.4 in.) or less in length as described in 6.3.1.5.

6.3.1.1 One end of each glass section is sealed closed (for example, with a glass disk equivalent in quality to that of the glass of the original sections).

6.3.1.2 The opposite end of each glass section retains the as-manufactured flared end.

6.3.1.3 Inlet and outlet ports with appropriate stopcock valves are added to each glass section as shown.

6.3.1.4 When assembled, the two glass sections are joined horizontally by flanges.[11] A PTFE gasket is used at the joint.[12]

6.3.1.5 In closed-loop tests where increased analytical sensitivity is required, a shorter length of glass pipe may be used to contain the collection medium. This reduces the contained volume and increases 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.

6.3.2 *Gaseous Test Chemical,* the test cell is identical to the liquid test cell except that, with reference to Fig. 1, two collection medium sections are used. Thus, the gaseous chemical can be circulated from its reservoir through the challenge-side chamber. 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.

6.3.3 A specimen is positioned between the flared ends of the two glass sections that compose the test cell as shown in Fig. 1. When the specimen is in place, the test cell is divided into two chambers.

6.4 *Alternative Test Cell,* alternative test cells for conducting permeation determinations may be used, provided that the results are reported as prescribed in Section 13. The cells described above and shown in Fig. 1, however, are the standards.

6.5 *Constant Temperature Chamber or Bath,* used to maintain the test cell within ±1.0°C of the test temperature.

7. Safety Precautions

7.1 Before carrying out this test method, 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.

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. Test Specimen

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 permeation 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 68 mm (2.7 in.). A 76-mm (3-in.) diameter circle is convenient.

8.3 A minimum of three random specimens shall be tested for each material, composite, area (in the case of a heterogeneous design), or other condition. Random specimens shall be generated as described in Practice E 105.

9. Conditioning

9.1 Condition each protective clothing material specimen for a minimum of 24 h by exposure to a temperature of 21 ± 5°C (70 ± 9°F) and a relative humidity of 30 to 80 % as described in Specification E 171.

10. Procedure A: Liquid Test Chemical

10.1 Measure the thickness of each conditioned specimen to the nearest 0.02 mm (or nearest 0.001 in.) and record.

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

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

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

[12] Gasket is available from Corning Glass, Catalog No. 72-9256.

10.2 Determine specimen weight per unit area, ±10 %, and record. This value, along with thickness, is a key characteristic of the material and needed when comparing the results of permeation testing.

10.3 Mount the first specimen in the test cell and assemble as shown in Fig. 1 and described in 6.3.3.

10.4 If the test is to be carried out at a nonambient temperature, place the assembled test cell into a constant-temperature chamber or bath at the test temperature. The cell should be maintained at the nonambient temperature for at least 30 min before the testing proceeds further.

10.5 Charge the collection medium into the test-cell chamber to which the normally inside surface of the material specimen is exposed. Depending upon the combination of analytical technique and collection medium selected, attach peripheral devices as appropriate (see Figs. 2 and 3).

10.5.1 If the test is to be carried out at a nonambient temperature and the collection medium is a liquid, bring the medium to the test temperature before adding it to the test cell.

10.6 The combination of analytical technique and collection medium shall be selected to: maximize sensitivity for the detection of the test chemical, and represent actual occupational conditions as closely as possible.

10.6.1 Distilled water is preferred as a collection medium when simulating perspiration on the inside surface of the material specimen. Consider alternative liquids only when the test chemical does not meet the solubility requirements as described in 3.4.

10.6.2 Air, nitrogen, and helium are the preferred choices for the collection medium when simulating air on the inside surface of the material specimen. 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.

10.6.3 In open-loop testing, the system shall have a sensitivity of at least 0.1 µg/cm^2/min. (See Appendix X1.)

10.6.4 In closed-loop testing, the system shall have a minimum sensitivity of at least 0.25 µg/cm^2.

10.7 Stir, circulate, or flow the collection medium continuously. (See Note 2.)

10.7.1 In open-loop testing, the recommended minimum flow rate is 50 cm^3/min and the recommended maximum flow rate is 150 cm^3/min.

NOTE 2—The purpose of agitating the collection medium is twofold: to ensure that it is homogeneous for sampling and analytical purposes 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 an open-loop system (that is, collection medium flowing once through the cell, then being analyzed and discarded), interlaboratory data suggest that an adequate degree of mixing is produced when the flow rate is within the range specified in 10.7.1. Higher rates may be required for permeants with low solubilities in the collection medium or high permeation rates. Higher flow rates also result in better mixing in 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 closed loop systems with a liquid collection medium, adequate mixing levels can be determined by preliminary experiments, in which the rapidity of the dispersion of a dye is observed.

10.8 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 permeation cell to establish the baseline values against which subsequent analytical data will be compared. (See Note 3.)

NOTE 3—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 chemical on the walls of the test cell and associated tubing.

10.9 Expeditiously charge the liquid chemical into the chamber of the test cell to which the normally outside surface of the material specimen is facing. Hereafter, this will be referred to as the challenge chamber of the test cell. Fill the chamber to the fill mark on the stem. Begin timing the test when the addition of the liquid commences.

10.9.1 If the test is to be carried out at a nonambient temperature, bring the liquid to the test temperature before it is charged into the test cell.

10.10 In closed-loop systems with sample withdrawal, replenishment of the collection medium may be necessary in order to maintain a fixed ratio of collection medium volume to surface area of the test specimen in contact with the collection medium.

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

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

10.12 Discontinue sampling and terminate the test after one or more of the following conditions is met (see Fig. 4):

10.12.1 Steady state permeation is reached (Fig. 4A and 4E).

10.12.2 Permeation proceeds at an ever increasing rate (Fig. 4C).

10.12.3 A maximum rate is reached (Fig. 4B and 4D).

10.12.4 A prespecified time has passed.

10.13 Disassemble the test cell and thoroughly clean it.

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

11. Procedure B: Gaseous Test Chemical

11.1 Measure the thickness of each conditioned specimen to the nearest 0.02 mm (or nearest 0.001 in.) and record.

11.2 Determine specimen weight per unit area, ±10 %, and record. This value, along with the thickness, is a key characteristic of the material and needed when comparing the results of permeation testing.