



Standard Test Method for Resistance of Protective Clothing Materials to Permeation by Liquids or Gases Under Conditions of Intermittent Contact¹

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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 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 F 739 and F 903, respectively. Methods for measuring the effects of the continuous contact of chemicals on the physical properties (that is, degradation) of rubbers, plastics, and coated fabrics are found in Test Methods D 471, Test Method D 543, and Test Method D 751, respectively.

1. Scope

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

NOTE 1—The liquid must be volatile or soluble in water.

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 hazard statements are given in Section 3.1.

2. Referenced Documents

2.1 ASTM Standards:

- D 471 Test Method for Rubber Property—Effect of Liquids²
- D 543 Practices for Evaluating the Resistance of Plastics to Chemical Reagents³

D 751 Test Methods for Coated Fabrics⁴

E 105 Practice for Probability Sampling of Materials⁵

E 171 Specification for Standard Atmospheres for Conditioning and Testing Flexible Barrier Materials⁶

F 739 Test Method for Resistance of Protective Clothing Materials to Permeation by Liquids or Gases Under Conditions of Continuous Contact⁷

F 903 Test Method for Resistance of Materials Used in Protective Clothing to Penetration by Liquids⁷

F 1001 Guide for Selection of Chemicals to Evaluate Protective Clothing Materials⁷

2.2 Federal Standard:

Fed. Std. No. 191, Method 5030.2 Measurement of the Thickness of Materials⁸

3. Terminology

3.1 Definitions:

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

¹ This test method is under the jurisdiction of ASTM Committee F-23 on Protective Clothing and is the direct responsibility of Subcommittee F23.30 on Chemicals.

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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.

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



3.1.1.1 *Discussion*—These procedures are often specific to individual chemical and collection medium combinations. Applicable techniques can include ultraviolet (UV) and infrared (IR) 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. 1).

3.1.2.1 *Discussion*—The breakthrough detection time is dependent on the sensitivity of the method.

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 %.

3.1.5 *contact time, n*—in an intermittent contact test, the duration during each cycle that the challenge side chamber of the permeation cell is filled with the test chemical.

3.1.6 *cumulative permeation, n*—the total mass of chemical that permeates during a specified time from when the material is first contacted.

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.

3.1.9 *minimum detectable mass permeated, n*—the smallest mass of test chemical that is detectable with the complete permeation test system. 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. This value is not necessarily the sensitivity of the analytical instrument.

3.1.11 *normalized breakthrough detection time, n*—in an open-loop system, the time at which the permeation rate reaches $0.1 \mu\text{g}/\text{cm}^2/\text{min}$ (see Fig. 1). In a closed-loop test, the time at which the mass of chemical permeated reaches $0.25 \mu\text{g}/\text{cm}^2$.

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

3.1.13 *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 non-molecular level.

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

3.1.14.1 *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.

3.1.15 *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.

3.1.15.1 *Discussion*—In this test method the particular hazard is permeation of a hazardous chemical.

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 challenge side chamber and air or nitrogen is blown over the outside surface of the protective clothing material.

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

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

4. Summary of Test Method

4.1 The resistance of a protective clothing material to a test chemical is measured in a permeation test in which replicate clothing specimens are 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 challenge chamber of the test cell.

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

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

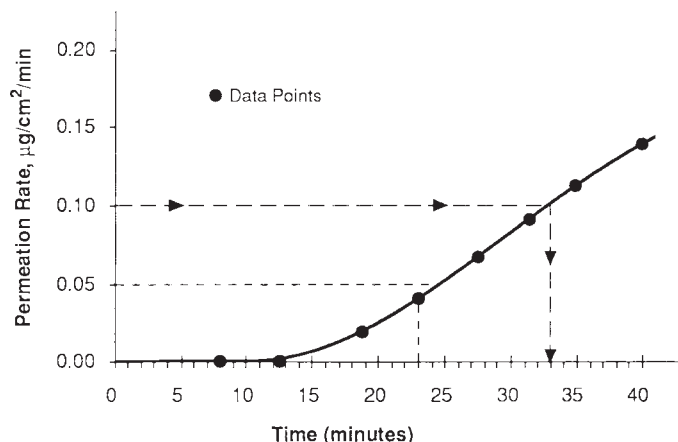


FIG. 1 The Breakthrough Detection Time for a Method Sensitivity of $0.05 \mu\text{g}/\text{cm}^2/\text{min}$ is 23 min. The Normalized Breakthrough Detection Time is 33 min.

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 F 739.

5.2 This test method is normally used to evaluate flat specimens 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, arm shields, aprons, suits, hats, boots, respirators, and the like.

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

5.3 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 F 739 is recommended for measuring permeation under the conditions of continuous contact of the test chemical with the clothing specimen.

5.4 The breakthrough detection time, normalized breakthrough detection time, and the 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 and normalized breakthrough detection times and low cumulative permeation are characteristics of better barriers.

NOTE 2—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.

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

5.5.2 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.

5.5.3 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.

5.5.4 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 A group of chemicals that is recommended for use in permeation testing is given in Guide F 1001.

6. Apparatus

6.1 *Thickness Gage*, 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 normal inside surface.

6.3.1 The test cell⁹, as shown in Fig. 2, 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. Each pipe section 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 by flanges.¹¹ A PTFE gasket is used at the joint.¹²

⁹ The test cell as shown in Fig. 2 is available from Pesce Lab Sales, P.O. Box 235, 226 Birch St., Kennet Square, PA 19348.

¹⁰ Sections of borosilicate glass pipe, available from Corning Glass, Catalog No. 72-0702 (1-in. length), or equivalent, have been found suitable for this purpose.

¹¹ Flanges, available from Corning Glass, Catalog Nos. 72-9062 (aluminum) or 72-9654 (cast iron), have been found suitable for this purpose.

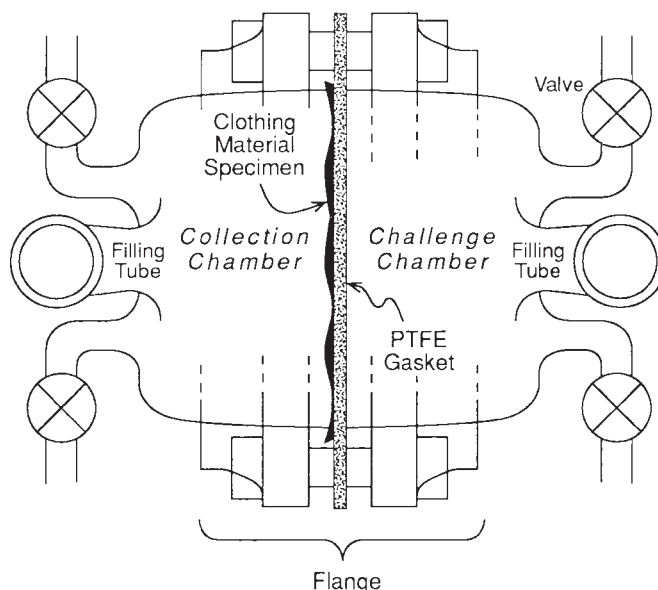


FIG. 2 ASTM Permeation Cell Configured for Intermittent Contact Testing (Top View)



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 The challenge-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.

6.3.3 The clothing material specimen is positioned between the flared ends of the two glass sections that compose the test cell as shown in Fig. 2. 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 also be used, provided that the results are reported as prescribed in Section 12. The cell described in 6.3.3 and shown in Fig. 2, however, is the standard.

6.5 *Constant Temperature Chamber or Bath*, used to maintain the test cell within $\pm 1^\circ\text{C}$ of the test temperature.

7. Hazards

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 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 $21 \pm 5^\circ\text{C}$ ($70 \pm 9^\circ\text{F}$) and a relative humidity of 30 to 80 % as described in Specification E 171.

10. Procedure

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

10.2 Determine specimen weight per unit area, $\pm 10\%$, 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 Mount the specimen in the test cell and assemble as described in 6.3.3.

10.4 If the test is to be carried out at a nonambient temperature, place the assembled test cell in a constant-temperature chamber or a water 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 on the combination of analytical technique and collection medium selected, attach peripheral devices as appropriate (see Figs. 3 and 4).

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 charging it to the test cell.

10.6 The combination of analytical technique and collection medium shall be selected to (1) maximize sensitivity for the detection of the chemical permeant, and (2) 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 preferred choices for 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 either the permeation process or the analytical procedure.

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

¹² Gasket, available from Corning Glass, Catalog No. 72-9256, has been found suitable for this purpose.

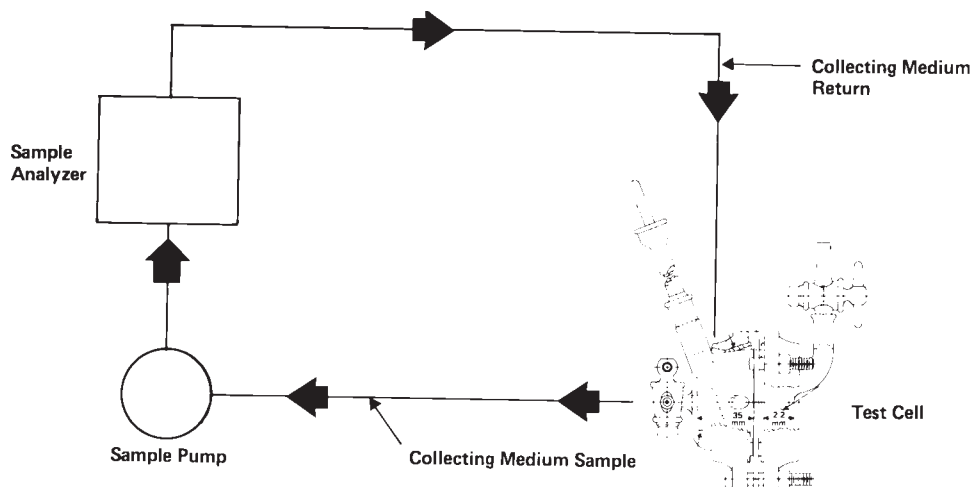


FIG. 3 Example Set-up for Continuous Collecting Medium Sample Withdrawal, Analysis, and Return

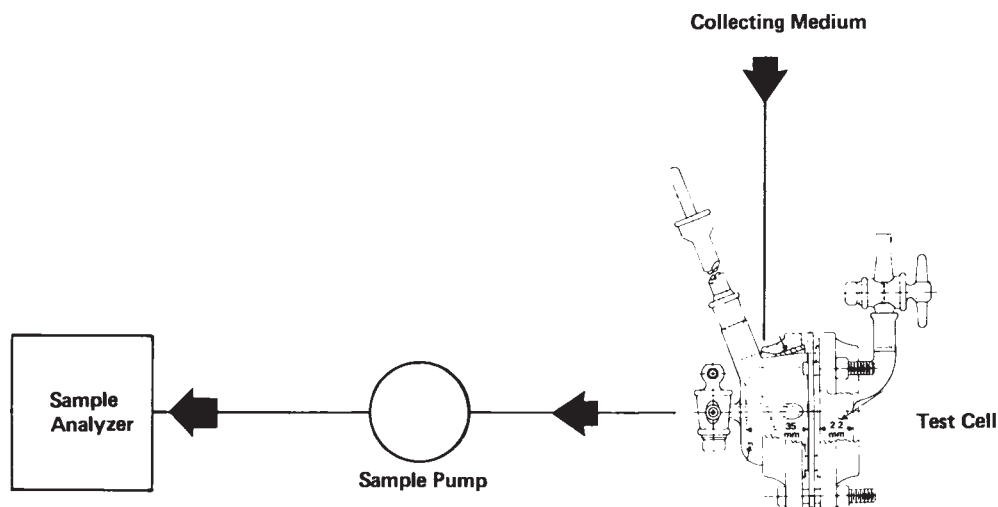


FIG. 4 Example Set-up for Continuous Flow of Fresh Collecting Medium

10.6.4 In closed-loop testing, the system shall have a sensitivity of at least $0.25 \mu\text{g}/\text{cm}^2$.

10.7 Stir, circulate, or flow the collection medium continuously (see Note 3).

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

NOTE 3—The purpose of agitating the collection medium is twofold: (1) to ensure that it is homogeneous for sampling and analytical purposes, and (2) to prevent or minimize concentration boundary layers of test chemical 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 permeant 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 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 liquid collecting media, adequate mixing levels can be determined by preliminary experiments, in which the rapidity of the dispersion of a dye is noted.

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 4).

NOTE 4—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 Expediently charge the test chemical into the challenge chamber of the test cell to which the normally outside surface of the material specimen is facing. Begin timing the test.

10.9.1 If the test is to be carried out at a nonambient temperature, bring the test chemical to the temperature before it enters the test cell.

10.9.2 For liquid test chemicals, fill the challenge chamber to the fill mark on the stem. Begin timing the chemical contact time when the addition of the liquid commences.

10.9.3 For gaseous test chemicals, begin the flow of gas into the challenge chamber. Begin timing the chemical contact time after the equivalent of five chamber volumes of gas have passed through the chamber, as determined by means of a rotameter or other flow monitoring device placed in the inlet stream to the chamber.

10.9.3.1 The five volumes of gas should be passed through the challenge chamber within 1 min. Following this initial period, the gas flow rate may be reduced. The flow, however, must be maintained at a rate that ensures that (1) the composition and concentration of the gas in the test chamber does not change with time, and (2) the test gas in the chamber is well mixed.

10.9.3.2 Care must be taken so as not to pressurize the challenge or the collection chamber. 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.

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 the test chemical was first added to the challenge chamber and withdrawal of the sample.

10.12 At the end of the predetermined contact time, remove the test chemical from the challenge chamber of the test cell. Begin timing the purge time.

10.12.1 For liquid chemicals, this may be accomplished by pouring or draining the test chemical from the challenge chamber through the inlet or outlet ports.

NOTE 5—Complete removal of viscous or tacky liquids may not be possible. Incomplete removal is acceptable since in the work environment complete removal of such chemicals is not likely.

10.12.2 For gaseous chemicals, this may be accomplished by stopping flow of the test chemical from its reservoir to the challenge chamber.

10.13 Flush the challenge chamber of the test cell for the predetermined purge time using air or an inert gas.

10.13.1 The purge gas should be flowed through the challenge chamber at a minimum rate equivalent to 10 challenge chamber volumes per minute.

10.13.2 Care must be taken not to pressurize the challenge chamber. Overly high pressures may develop at high gas flow rates or as a result of attachments to the chamber that restrict the flow of gas from the chamber.

10.13.3 If the test is to be carried out at a nonambient temperature, bring the purge gas to the test temperature before it enters the test cell.

10.14 At the end of the predetermined purge time, stop the flow of purge gas through the challenge chamber. This will complete the first cycle of the intermittent exposure test.

10.15 Continue the test by repeating cycles of contact and purge according to 10.9.1 through 10.14 until either a predetermined time or number of cycles has passed.

10.15.1 The contact time, purge time, and number of cycles composing a test are at the discretion of the tester. As a guide and to help in the comparison of data, one or more of the following sets of conditions are recommended:

Condition	Contact time, min	Purge time, min	Number of cycles
A	1	10	10
B	5	10	7
C	15	60	3

10.16 Disassemble the test cell and thoroughly clean it.

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

11. Calculation

11.1 *Symbols*—The following symbols are used in Eqs (1–7):

- A = area of the material specimen contacted, cm^2 ,
- C_i = concentration of test chemical in the collection medium at time T_i , $\mu\text{g/L}$,
- F = flow rate of fresh collection medium through the cell, L/min ,
- i = an indexing number assigned to indicate the specific concentration C_i that was measured at time T_i in volume V_i , starting with $i = 1$ for the first point,
- M_i = cumulative amount permeated at T_i , $\mu\text{g/cm}^2$,
- P_i = permeation rate at T_i , $\mu\text{cm}^2/\text{min}$,
- \bar{P}_i = average permeation rate for the interval T_{i-1} to T_i , $\mu\text{g/cm}^2/\text{min}$,
- T_i = time elapsed from the beginning with initial chemical contact and ending with the measurement of concentration C_i , min,
- T = time elapsed from the beginning of the first chemical contact, min,
- T_p = the elapsed time from the beginning of the first chemical contact to the mid-point of a sampling interval, min,
- V_t = total volume of the collection medium, L,
- V_s = volume of discrete sample removed from the collection medium, L, and
- V_i = volume of collection medium at T_i , L.



NOTE 6—The sample area exposed in the standard ASTM test cell is about 20.4 cm², varying slightly with the source of the PTFE gasket.

NOTE 7—The following factors are useful in converting permeation: 1 µg/cm²/min = 0.17 mg/m²/s = 10 mg/m²/min.

11.2 Calculations for Systems Using a Continuous Flow of Fresh Collection Medium (Open-Loop)—These calculations are applicable to a system where fresh collection medium transports the permeant from the cell to the analyzer as shown in Fig. 4.

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

$$P_i = C_i F/A \quad (1)$$

11.2.2 The cumulative amount permeated, M_i , at any time, T , is equivalent to the area under a curve obtained by plotting P_i versus T_i from time 0 to T . This amount will not include any chemical that may have permeated before the detection time; but was below the detection limit of the test method.

11.3 Calculations for Closed Systems (Closed-Loop)—These calculations are applicable when any of the following conditions are met: (1) samples are withdrawn, analyzed, and replaced prior to further sampling, (2) the volume of discrete samples is insignificant relative to the total volume (for example, microliter aliquots), (3) the collection medium is recirculated as in Fig. 3, or (4) the concentration of the test chemical is measured in the collection chamber without any sample removal.

11.3.1 The average permeation rate over the period $T_i - 1$ to T_i is calculated as follows:

$$\bar{P} = \frac{(C_i - C_{i-1}) V_i}{(T_i - T_{i-1}) A} \quad (2)$$

11.3.2 The cumulative amount permeated, M_i , for the total period up to T_i is calculated as follows:

$$M_i = \frac{C_i V_i}{A} \quad (3)$$

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

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

$$\bar{P}_i = \frac{(C_i - C_{i-1}) (V_t - [i - 1]V_s)}{(T_i - T_{i-1}) A} \quad (4)$$

11.4.2 Replenishment of the collection medium after each discrete sample changes the calculation to the following:

$$\bar{P}_i = \frac{\left[C_i - C_{i-1} \left(\frac{V_t - V_s}{V_t} \right) \right] V_t}{(T_i - T_{i-1}) A} \quad (5)$$

11.4.3 The cumulative amount permeated, M_i , for the total period up to T is calculated as follows:

$$M_i = \frac{C_i V_i}{A} + \sum_{i=1}^{i=i-1} C_i V_s \quad (6)$$

11.5 When plotting average permeation rate or average cumulative permeation as a function of time, the time coordi-

nate is the mid-point of the interval over which the average was obtained, and is calculated as follows:

$$T_p = \frac{T_i + T_{i-1}}{2} \quad (7)$$

12. Report

12.1 Report the following information:

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

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

12.1.3 Report weight per unit area (g/m²) of each specimen to ± 10 %. Also, calculate and report the average weight per unit area of the specimens tested for each material type.

12.1.4 Report the name of the test chemical, its physical state as tested (that is, liquid or gas) and if it is in a mixture, its concentration, and the other components.

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

12.1.6 Report the nominal test temperature (°C) and temperature range existent during the test.

12.1.7 Report the cycle time, contact time, purge time, number of cycles, and the total duration of the test.

12.1.8 For each open-loop test in which chemical permeation was detected, report the normalized breakthrough detection time (that is, the time at which the permeation rate reaches 0.1 µg/cm²/min).

12.1.8.1 If permeation is first detected at a permeation rate > 0.1 µg/cm²/min, report the breakthrough detection time and the rate at which permeation was detected.

12.1.8.2 If permeation is first detected at a rate < 0.1 µg/cm²/min, the breakthrough detection time and rate at which permeation was detected may be reported.

12.1.8.3 If no permeation was detected or the permeation rate did not reach 0.1 µg/cm²/min, report the fact that no permeation was detected at either the minimum detectable permeation rate (see Appendix X1) or 0.1 µg/cm²/min.

12.1.9 For each closed-loop test in which chemical permeation was detected, report the normalized breakthrough detection time (that is, the time at which chemical permeation reached 0.25 µg/cm²) for each material specimen.

12.1.9.1 If permeation is first detected at an amount > 0.25 µg/cm², report the breakthrough detection time and the amount at which permeation was detected.

12.1.9.2 If permeation is first detected at an amount < 0.25 µg/cm², the breakthrough detection time and the amount may be reported.

12.1.9.3 If no permeation was detected, report the duration of the test and the minimum detectable mass permeated in µg/cm².

12.1.10 Plot the permeation rate as a function of time for each specimen tested. Provide a copy of this graphical presentation for each of the triplicate tests (see Fig. 5).

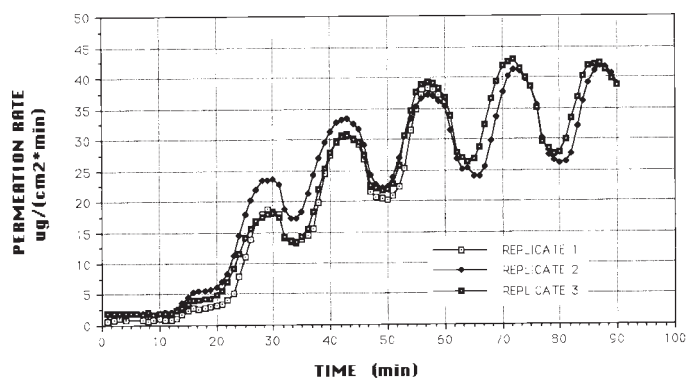


FIG. 5 Splash Test; Acetone/Neoprene 1 min Contact, 14 min Purge

12.1.11 Report the cumulative permeation in micrograms per square centimeter at four times equally spaced over the duration of the test. For example, if the test is one hour in duration, report the amounts at 15, 30, 45, and 60 min; if eight hours, at 2, 4, 6, and 8 h. Also, report the average of the three values of cumulative permeation for each time for each material type.

12.1.12 Plot the cumulative permeation as a function of time for each material tested. Provide a copy of this graphical presentation for each material type for each of the triplicate tests.

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

13. Precision and Bias

13.1 A precision and bias analysis has not yet been completed for this procedure.

13.2 The neoprene material used in the interlaboratory evaluation of Test Method F 739 is recommended as a standard reference material.¹³

¹³ Neoprene sheet, 0.016 in. (16 mil), Stock No. 5550, available from Fairprene Industrial Products Co., Inc., 85 Mill Plain Rd., Fairfield, CT 06430, (203-259-3351). Small samples are available through the Chairman of Subcommittee F23.30.

APPENDIXES

(Nonmandatory Information)

X1. PROCEDURE FOR MEASURING THE SENSITIVITY OF OPEN-LOOP PERMEATION TEST SYSTEMS¹⁴

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

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

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

X1.1.3 Detector response to the test chemical.

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

NOTE X1.1—A piece of aluminum foil, or other material known to be inert and impermeable to the test chemical, is placed in the permeation test

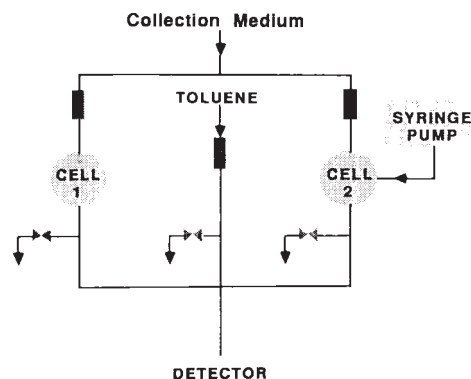


FIG. X1.1 Permeation System Set Up (for Sensitivity Determination)

cells in lieu of the protective clothing material. This arrangement allows collection medium and test chemical to mix in a manner simulating permeation of the chemical through the protective clothing material.

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

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

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

¹⁴ Alternative procedures may be used.

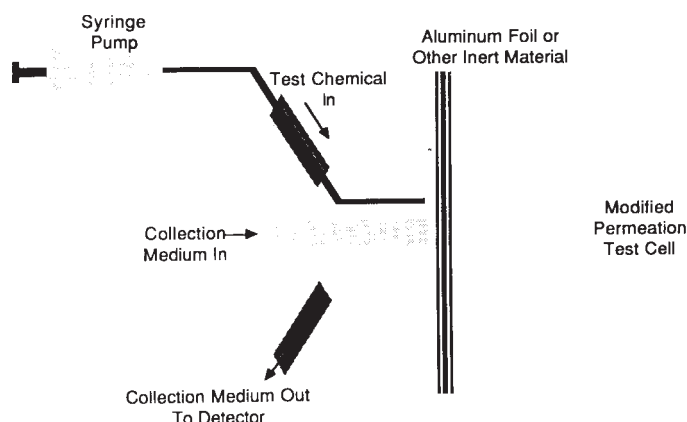


FIG. X1.2 Test Set Up to Determine Permeation System Sensitivity

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

X1.4 The test chemical can be delivered to the second permeation test cell using any method which can provide a controlled, measurable rate. A syringe pump may be employed for this purpose. The flow rate of the collection medium through both the blank cell and the test cell should be calibrated with a standardized flow meter at the outlet of the cell before beginning the test. With the collection medium flowrate and the rate of test chemical introduction into the

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

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

where:

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

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

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

X2. PERMEATION REFERENCE MATERIAL

X2.1 A standard reference material is available.¹³ It may be used to evaluate the performance of a permeation test system.

X2.2 The standard reference material was used in an interlaboratory evaluation of Procedure A of Test Method F 739 with acetone as the test chemical. The results are summarized in Table X2.1.

X2.3 For quality control and assurance requirements,

periodic evaluation of the permeation test system is recommended, using acetone, the standard neoprene, and Procedure A of Test Method F 739. Breakthrough detection time and steady state permeation rate are measured and the results compared to the interlaboratory results reported in Table X2.1. Results falling within ± 2 laboratory standard deviations (that is, ± 6 min) are indicative of satisfactory performance of the permeation test system.

**TABLE X2.1 Results of Interlaboratory Evaluation of Test Method F 739 Using Acetone as the Test Chemical and the Standard Neoprene Sheet as the Clothing Material Surrogate**

Laboratory	Number of Tests	Thickness, mm	Breakthrough Detection Time, min	Steady State Permeation Rate, $\mu\text{g}/\text{cm}^2/\text{min}$
1	3	0.43	...	2934 ± 441
2	3	0.43	...	no data
3	4	0.43	...	2134 ± 91
4	3	0.39	...	no data
5	4	0.43	...	1989 ± 379
6	3	0.42	...	1120 ± 191
7	3	0.43	...	2447 ± 83
8	3	no data	...	1188 ± 120
9	3	0.43	...	2826 ± 202
Average	2088 ± 202

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