



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

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 (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 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 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 fabrics are found in Test Methods [D471](#), Test Method [D543](#), and Test Method [D751](#), respectively. A starting point for selecting the chemicals to be used in assessing the chemical resistance of clothing materials is Guide [F1001](#).

1. Scope

1.1 This test method measures the permeation of liquids and gases through protective clothing materials under the condition of 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 appropriate safety and health practices and determine the applica-*

bility of regulatory limitations prior to use. Specific hazard statements are given in Section [7](#).

2. Referenced Documents

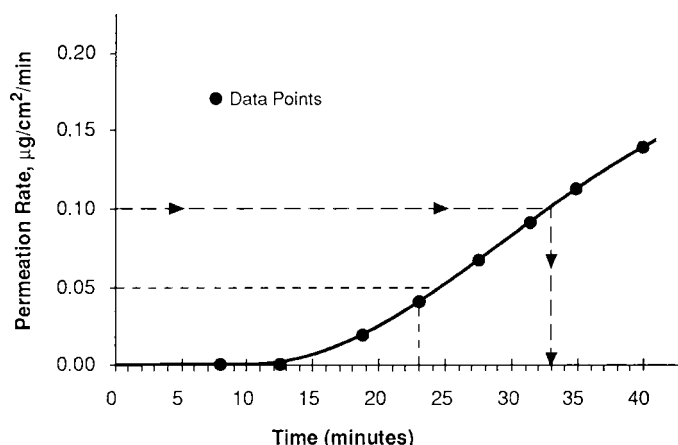
2.1 ASTM Standards:²

[D471](#) Test Method for Rubber Property—Effect of Liquids
[D543](#) Practices for Evaluating the Resistance of Plastics to Chemical Reagents
[D751](#) Test Methods for Coated Fabrics
[D1777](#) Test Method for Thickness of Textile Materials
[E105](#) Practice for Probability Sampling of Materials
[E171](#) Practice for Conditioning and Testing Flexible Barrier Packaging
[E177](#) Practice for Use of the Terms Precision and Bias in ASTM Test Methods
[E691](#) Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
[F739](#) Test Method for Permeation of Liquids and Gases through Protective Clothing Materials under Conditions of Continuous Contact

¹ This test method is under the jurisdiction of ASTM Committee [F23](#) on Personal Protective Clothing and Equipment and is the direct responsibility of Subcommittee [F23.30](#) on Chemicals.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



NOTE 1—In an intermittent contact test, it is possible that the permeation rate will exceed, go below, and then again exceed a permeation rate of 0.1 µg/cm²/min. If this occurs, the standardized breakthrough time is the first occurrence of the permeation rate exceeding 0.1 µg/cm²/min.

FIG. 1 The Breakthrough Detection Time for a Method Sensitivity of 0.05 µg/cm²/min is 23 min. The Standardized Breakthrough Detection Time is 33 min.

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

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

F1494 Terminology Relating to Protective Clothing

2.2 ISO Standard:

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

3. Terminology

3.1 Definitions:

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

3.1.1.1 *Discussion*—These 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 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 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 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 *minimum detectable mass permeated, n*—the smallest mass of test chemical that is detectable with the complete permeation test system.

3.1.8.1 *Discussion*—This value is not necessarily the sensitivity of the analytical instrument.

3.1.9 *minimum detectable permeation rate, n*—the lowest rate of permeation that is measurable 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 *open-loop, adj*—refers to a testing mode in which fresh collection medium flows continuously through the collection chamber of the test cell.

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

3.1.11.1 *Discussion*—Voids include gaps, pores, holes, and imperfections in closures, seams, interfaces, and protective clothing materials. Penetration does not require a change in state; solid chemicals move through voids in materials as solids, liquids as liquids, and gases as gases. Penetration is a distinctly different mechanism from permeation.

3.1.12 *permeation, n*—for chemical protective clothing, the movements of chemicals as molecules through protective clothing materials by the processes of: (1) absorption of the chemical into the contact surface of the materials, (2) diffusion of the absorbed molecules throughout the material, and (3) desorption of the chemical from the opposite surface of the material.

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

3.1.13 *protective clothing, n*—item of clothing that is specifically designed and constructed for the intended purpose of isolating all or part of the body from a potential hazard; or,

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

isolating the external environment from contamination by the wearer of the clothing.

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

3.1.15 *standardized breakthrough time, n*—the first time at which the permeation rate reaches $0.1 \mu\text{g}/\text{cm}^2/\text{min}$ (see Fig. 1).

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

3.1.16.1 *Discussion*—The liquid or gas may be either one component (for example, a neat liquid or gas) or have several components (for example, a mixture).

4. Summary of Test Method

4.1 The permeation of chemical(s) through a protective clothing material is assessed by measuring the breakthrough detection time, standardized breakthrough time, 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 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 barrier 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, 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 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 F739 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, standardized breakthrough 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 standardized breakthrough times and low cumulative permeation are characteristics of better barriers.

NOTE 1—At present, there is limited quantitative information exists about acceptable levels of dermal contact with most chemicals. Therefore, the data obtained using this test method cannot be used to infer safe exposure levels.

5.4.1 The reporting of a standardized breakthrough time greater than a specific time period does not mean that no chemical has permeated through the protective clothing material as the standard breakthrough time is determined based on the permeation rate reaching a level of $0.1 \mu\text{g}/\text{cm}^2 \text{ min}$, indicating that some chemical has already permeated the specimen prior to the reported standardized breakthrough time.

5.4.2 Cumulative permeation represents the mass that permeates through a protective clothing material over a specific period of time for a specific surface area of material. It is possible to use this information to model how much chemical can enter an item of protective clothing for a particular exposure based on a knowledge of the exposed surface area, the free volume inside the protective clothing item, and amount of air mixing or air exchange for the protective clothing item.

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 should be capable of measuring the concentration of the test chemical in the collection medium at, or below, levels consistent with standardized breakthrough time value specified in 3.1.15.

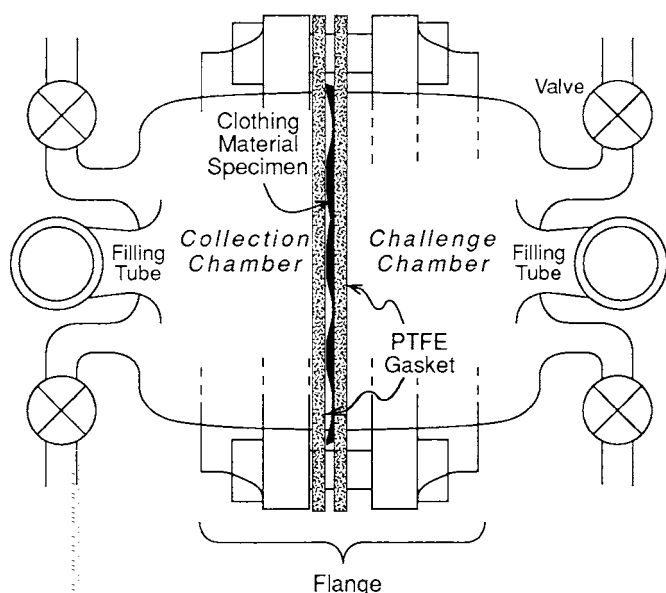
5.5.2 Often, permeation tests will require measurement of the test chemical over several orders of magnitude in concentration, requiring adjustments in either the sample collection volume or concentration/dilution, or the analytical instrument settings over the course of the test.

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



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

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 F1001.

6. Apparatus

6.1 *Thickness Gauge*, suitable for measuring thicknesses to the nearest 0.02 mm (or the nearest 0.001 in.), as specified in Test Method D1777, shall be used to determine the thickness of each protective clothing material specimen tested.

6.2 *Analytical Balance*, readable and reproducible to ± 0.5 mg shall be used to determine weight per unit area of each test specimen.

6.3 *Test Cell*—The test apparatus consists of a two-chambered cell for contacting the specimen with the test chemical on the specimen's normally outside surface and with a collection medium on the specimen's normal inside surface.

6.3.1 The test cell⁴, as shown in Fig. 2, is constructed of two sections of straight glass pipe, each nominally sized to a 25.4 mm (1.0 in.) diameter.⁵ Materials other than glass may be used. Such materials would be required for tests involving chemicals (for example, hydrofluoric acid), which are incompatible with glass. The section that is designated to contain the test chemical is 25.4 mm (1.0 in.) in length. The second section, which is designated to contain the collection medium, is 32 mm (1.2 in.) or less in length.

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

⁴ The test cell as shown in Fig. 2 is available from Pesce Lab Sales, P.O. Box 235, 226 Birch St., Kennett Square, PA 19348.

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

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

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

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

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

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

6.3.2.2 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. However, these approaches to increasing sensitivity must be achieved within the constraints of having sufficient volumes and mixing rates so as not to interfere with the permeation process.

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

6.3.2.4 For a liquid collection medium that is not circulated, the 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.

6.3.2.5 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

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

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

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

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. 2, 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 $\pm 1^\circ\text{C}$ of the test temperature. The standard temperature for this test is 27°C . Condition all test materials, including the test cells and chemicals, in the chamber(s) of bath(s) prior to testing.

6.6 Circulating pump, if appropriate, used to transport the collection medium and/or test chemical 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 and/or dilution within the test cell.

6.7 Flow meter, used to measure the flow rate of the collection medium through the collection chamber. A calibrated rotameter, or similarly accurate device, may be used. The flow rate shall be measured in-line with all system components in place at the start of each test.

6.8 Thermometer or thermocouple, used to measure the temperature of the constant-temperature chamber (or bath) and/or the collection chamber of the test cell. A calibrated device, accurate to $\pm 0.5^\circ\text{C}$ must 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 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. Testing and Analytical Technique Considerations

8.1 Each protective clothing material specimen may consist of either a single layer or a composite of multiple layers that is representative of an actual protective clothing construction with all layers arranged in proper order. In each test, the specimen's normally outer surface shall contact the test chemical.

8.1.1 If, in a proposed design of an item of protective clothing, different materials or thicknesses of materials are specified at different locations, specimens from each location shall be tested.

8.1.2 If, in a proposed design, seams are specified, additional specimens containing such seams shall be tested. Care must be taken to ensure that the test cell can be properly sealed when specimens of nonuniform thickness are tested.

8.2 Each material specimen to be tested shall have minimum cross dimension of 43 mm (1.7 in.). A51 mm (2 in.) diameter circle is convenient.

8.3 A minimum of three random specimens shall be tested. Random specimens shall be generated as described in Practice E105.

8.4 To avoid incidental contamination of exposed surfaces, clean gloves may be worn when handling specimens.

8.5 To avoid affecting permeation measurements, a collection medium should not interact with the test material, and must have adequate capacity for the permeant. To have adequate capacity for the permeant, the collection medium should not exceed 20 % of its saturation concentration from the permeant at any time during the test. For a liquid collection medium, saturation is the maximum solubility or miscibility of the permeant in the liquid at the test temperature. For a gaseous collection medium, saturation is determined by the vapor pressure of the permeant.

8.6 Under conditions in which the test chamber or bath is at a temperature significantly different from that of the test chemical or collection medium that is being introduced into the test cell, the temperature in the test chemical chamber and/or the collection chamber should be measured. It may be necessary to pre-condition the test chemical or collection medium before it enters into the test cell. Similarly, it may be necessary to maintain the temperature of the collection medium after it leaves the test cell to prevent condensation or precipitation.

8.7 The combination of system configuration, analytical technique, and collection medium shall be selected to allow measurement of the test chemical over the range of 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.

8.7.2 Air, nitrogen, and helium are the preferred choices for the collection medium for volatile test chemicals. Consider alternative gases only when these gases interfere with analytical detection of the test chemical. Regardless of the gas used, its purity must be sufficiently high so as not to interfere with the permeation process or the analytical procedure.

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

8.7.4 In closed-loop testing, the system shall have a minimum sensitivity to detect a permeation rate of $0.1\mu\text{g}/\text{cm}^2/\text{min}$ over a five minute sampling period.

8.8 With the nominal 25 mm diameter cell and in open-loop mode or in closed-loop mode with a circulating collection medium, the minimum flow rate for the collection medium is 100 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 *Discussion*—The purpose of agitating/mixing the collection medium is twofold: to ensure that it is homogeneous for sampling and analytical purpose and to prevent or minimize concentration boundary layers of permeant at the interface of the clothing material and the collection medium. The degree of agitation necessary to achieve these objectives is dependent on the permeation rate and the relative solubilities of the test chemical in the clothing material and the collection medium. At this time, sufficient data are not available to specify minimum agitation rates. However, as guidance, in any system in which the collection medium is flowing through the collection chamber, the minimum flow rate should be five chamber volumes per minute. Higher rates may be required for permeants with low solubilities in the collection medium or high permeation rates. High flow rates also result in better mixing in the chamber and consequently more uniform samples for analysis. 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 ± 2°C (81 ± 4°F) and a relative humidity of 30 to 80 % as described in Specification E171.

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 centimetre by weighing the specimen on an analytical balance (±2 mg) and dividing by the area (±0.4 cm²), and record. This value, along with thickness, is a key characteristic of the material and is needed when comparing the results of permeation testing.

10.3 Measure and record the inside diameter of the nominal 25 mm diameter opening of the PTFE gasket. Mount the first specimen in the test cell and assemble as shown in Fig. 2 and described in 6.3.1.

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 may be used, but must be noted in the report. The test cell must not be removed from the temperature chamber or bath for the duration of the test.

10.5 Charge the collection medium into the test-cell chamber to which the normally inside surface of the material specimen is exposed. The collection medium must be at the test temperature when it is introduced. Depending on the combination of analytical technique and collection medium selected, attach peripheral devices as appropriate (see Figs. 3 and 4). The cell, along with the collection medium, should be maintained at the test temperature for at least 30 minutes before the 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 Discussion following 8.8).

10.7 Initiate sampling of the collection medium, either continuously or discretely, and continue on a predetermined schedule throughout the test duration. Promptly complete analysis of each sample for test chemical content. Sampling is initiated before the test chemical is added to the permeation cell to establish the baseline values against which subsequent analytical data will be compared (see Note 2).

NOTE 2—The method chosen for collection medium withdrawal shall be based on the technique selected for analytical detection. For example, UV or IR spectroscopy is often used for continuous analysis of a sample stream (although compounding and curing agents often used in protective clothing materials can interfere) while gas chromatography requires the analysis of discrete samples. When sampling using open-loop techniques, the flow of collection medium should never be interrupted. This will minimize adsorption of permeated chemical on the walls of the test cell and associated tubing.

10.8 Add the test chemical into the test chemical chamber of the test cell.

10.8.1 The test chemical must be at the test temperature when it is added to the test cell.

10.8.2 For liquid test chemicals, the chemical can be introduced by pouring, syringe, cannula, etc. Fill the chamber to a level that indicates the liquid is covering the clothing material specimen, with no air bubbles. Begin timing the chemical contact time when upon the first addition of the liquid commences.

10.8.3 For gaseous test chemicals, begin the flow of gas into the test chamber. Begin timing the chemical contact time after

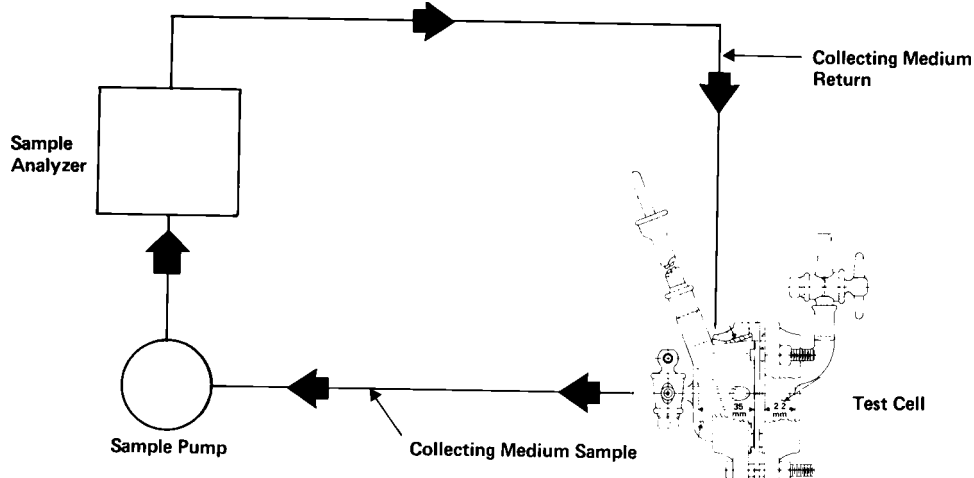


FIG. 3 Example Set-up for Continuous Collecting Medium Sample Withdrawal, Analysis, and Return

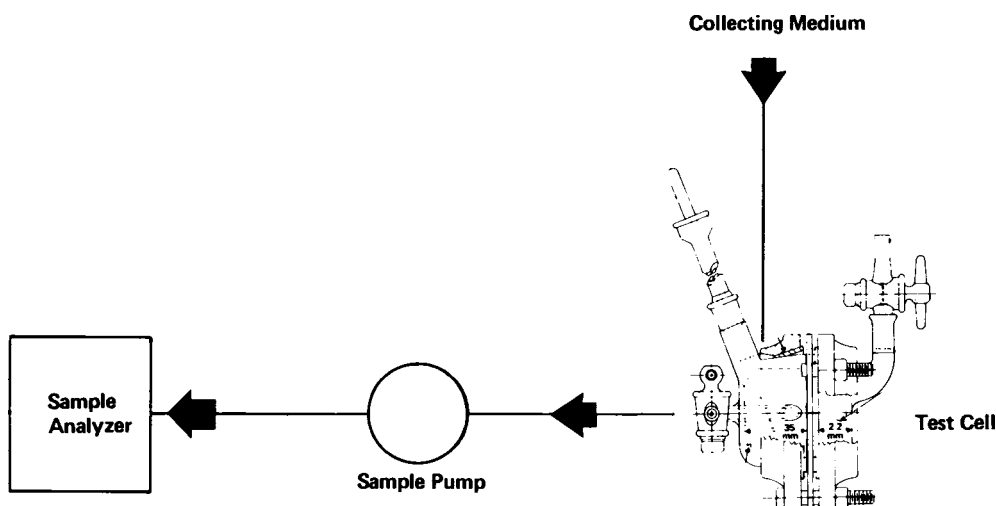


FIG. 4 Example Set-up for Continuous Flow of Fresh Collecting Medium

the equivalent of five chamber volumes of gas have passed through the chamber, as determined by means of a rotameter or other flow monitoring device placed in the outlet stream to the test chamber.

10.8.3.1 The five volumes of gas should be passed through the test 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 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 test chamber and withdrawal of the sample.

10.9.1 An analytical sample should be collected as soon as possible after contact of the specimen with the test chemical, but must be collected within the first 15 minutes of contact.

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

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

NOTE 3—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.

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

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

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

10.11.2 Care must be taken not to pressurize the test chemical 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.

10.11.3 Bring the purge gas to the test temperature before it enters the test cell.

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

10.13 Continue the test by repeating cycles of contact and purge in accordance with 10.8.1 through 10.12 until either a predetermined time or number of cycles has been completed.

10.13.1 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
D	User Defined	User Defined	User Defined

10.14 Remove the test cell from the controlled temperature environment. Remove the test chemical. Remove the collection medium. Disassemble the test cell and thoroughly clean it.

10.15 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 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,
- K_t = cumulative permeation determined at a given time, t ,
- n = number of measurement intervals between the start of the test and the time for which cumulative permeation is reported.
- M_i = cumulative amount permeated at T_i , $\mu\text{g/cm}^2$,
- P_i = permeation rate at T_i , $\mu\text{g/cm}^2/\text{min}$,
- $\bar{P}_i$ = average permeation rate for the interval T_{i-1} to T_i , $\mu\text{g/cm}^2/\text{min}$,
- T_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 4—The sample area exposed in the standard ASTM test cell (nominal 25 mm) is about 5.1 cm^2 , varying slightly due to the PTFE gasket.

NOTE 5—The following factors are useful in converting permeation: $1 \mu\text{g/cm}^2/\text{min} = 0.17 \text{ mg/m}^2/\text{s} = 10 \text{ mg/m}^2/\text{min}$.

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

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

$$P_i = C_i F / A$$

11.2.2 The cumulative permeation at a specific time, K_t , is determined as follows. If no test chemical is detected at any sampling time, T_i , the minimum detectable concentration of permeant should be utilized as C_j . C_0 is set to 0.

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

11.3 *Calculation for Closed Systems (Closed-Loop)*—This calculation is 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:

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

11.3.2 When conditions (1) through (4) of 11.3 are met, the cumulative permeation at T_i is determined as follows. If no test chemical is detected, the minimum detectable concentration of test chemical is substituted for value of C_i .

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

11.4 *Calculation for Closed Systems with Discrete Sampling*—This calculation is applicable when discrete samples of significant volume are removed from the collection medium.

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

$$P_i = \frac{C_i [V_T - (t-1)V_s] - C_{i-1} [V_T - (t-2)V_s]}{(T_i - T_{i-1}) A}$$

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

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

11.4.3 Cumulative permeation at time T_i for a closed system with discrete sampling is determined as follows. If no test

chemical is detected, the minimum detectable concentration of test chemical is substituted for value of C_i and C_j . There is no test chemical in the collection medium at time 0 ($C_0 = 0$).

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

11.5 When plotting average permeation rate or average cumulative permeation as a function of time, the time coordinate is the mid-point of the interval over which the average was obtained, and is calculated as follows:

$$T_p = \frac{T_i + T_{i-1}}{2}$$

12. Report

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

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

12.3 Report weight per unit area (g/m^2) of each specimen. Also, calculate and report the average weight per unit area of the specimens tested for each material type.

12.4 Report the name of the 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.

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

12.6 Report the test temperature ($^{\circ}\text{C}$).

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

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

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

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

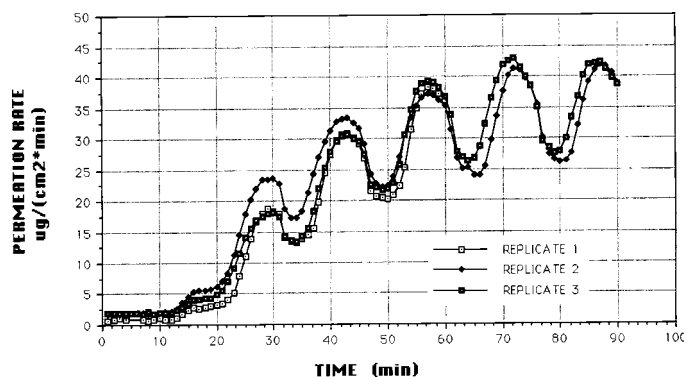


FIG. 5 Splash Test; Acetone/Neoprene 1 min Contact, 14 min Purge

12.8.3 If no permeation was detected or the permeation rate did not reach $0.1 \mu\text{g}/\text{cm}^2/\text{min}$, report the fact that no permeation was detected at either the minimum detectable permeation rate (see Appendix X1) or $0.1 \mu\text{g}/\text{cm}^2/\text{min}$.

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

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

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

12.12 In addition, the results may be presented in accordance with the guidance provided in Guide F1194.

13. Precision and Bias

13.1 A precision and bias analysis has not yet been completed for this procedure. Please refer to Test Method F739 for the results of an interlaboratory evaluation of Test Method F739, which is the same as this method except for contact is continuous rather than intermittent.

13.2 Committee F23 maintains two standard materials, Neoprene rubber and Norfoil film, for validating permeation test procedures.⁹

⁹ 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.

APPENDIX

(Nonmandatory Information)

X1. PROCEDURE FOR MEASURING THE SENSITIVITY OF OPEN-LOOP PERMEATION TEST SYSTEMS¹⁰

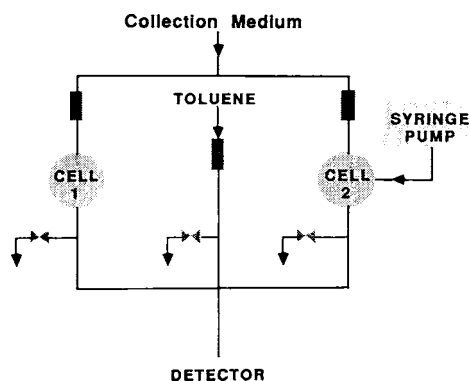


FIG. X1.1 Permeation System Set Up (for Sensitivity Determination)

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

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

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

X1.1.3 Detector response to the test chemical.

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

NOTE X1.1—A piece of aluminum foil, or other material known to be inert and impermeable to the test chemical, is placed in the permeation test cells in lieu of the protective clothing material. This arrangement allows collection medium and test chemical to mix in a manner simulating permeation of the chemical through the protective clothing material.

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

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

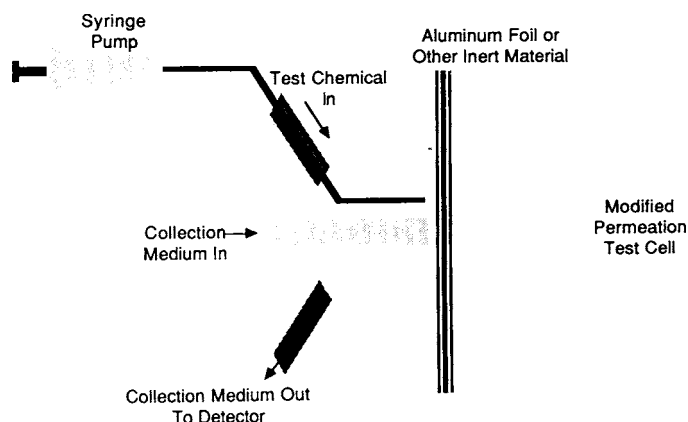


FIG. X1.2 Test Set Up to Determine Permeation System Sensitivity

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

X1.3.3 One port 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

¹⁰ Alternative procedures may be used.

response from responses for both the test chemical and the standard calibration chemical. The ratio of these adjusted detector responses can then be used to determine permeant

concentrations, when the calibration chemical is also used as an internal standard during the actual permeation test.

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