Designation: F739 – 07

StandardTest Method for Permeation of Liquids and Gases through Protective Clothing Materials under Conditions of Continuous Contact[1]

This standard is issued under the fixed designation F739; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (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 permeation of liquids and gases through protective clothing materials under the conditions of continuous contact of the clothing material by the challenge chemical. Resistance to permeation under the condition of intermittent contact with the challenge chemical should be determined by Test Method F1383. In certain situations, the permeation of liquids through protective clothing materials can be measured using a permeation cup following Test Method F1407. Penetration of liquids should be determined by Test Method F903. An undesirable change in the physical properties of protective clothing materials is called degradation. Procedures for measuring the degradation of rubbers, plastics, and coated fabrics are found in Test Method D471, 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 continuous contact.

1.2 This test method is designed for use when the challenge 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 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Section 7.*

2. Referenced Documents

2.1 *ASTM Standards:*[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
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

[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.

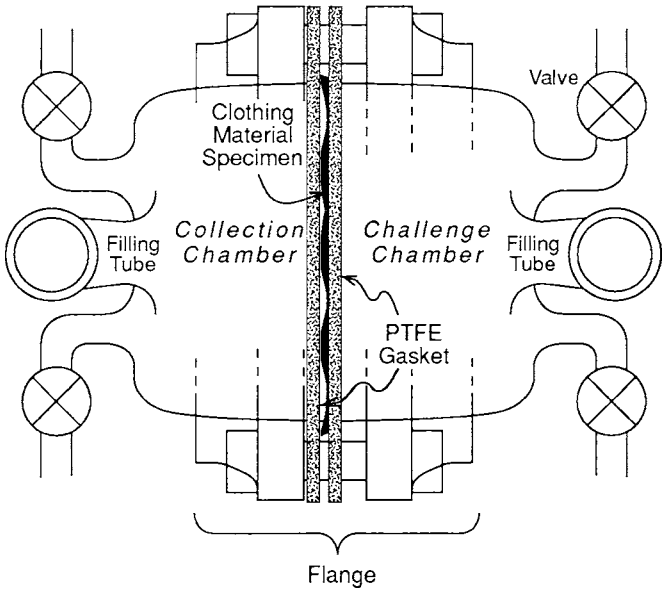


FIG. 1 ASTM Permeation Test Cell

F1194 Guide for Documenting the Results of Chemical Permeation Testing of Materials Used in Protective Clothing

F1383 Test Method for Permeation of Liquids and Gases through Protective Clothing Materials under Conditions of Intermittent Contact

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

F1494 Terminology Relating to Protective Clothing

2.2 *ISO Standard:*

ISO 6529 Protective Clothing—Determination of Resistance of Protective Clothing Materials to Permeation by Liquids and Gases[3]

3. Terminology

3.1 *Definitions:*

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

3.1.1.1 *Discussion*—These techniques are often specific to individual chemical and collection medium combinations. Applicable techniques include, but are not limited to, flame ionization, photo ionization, electro-chemical, ultraviolet and infrared spectrophotometry, gas and liquid chromatography, colorimetry, length-of-stain detector tubes, and radionuclide tagging/detection counting.

3.1.2 *breakthrough detection time, n*—the elapsed time measured from the start of the test to the sampling time that immediately precedes the sampling time at which the challenge chemical is first detected.

3.1.2.1 *Discussion*—(See Fig. 6.) The breakthrough detection time is dependent on the sensitivity of the method (see Appendix X1).

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

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

3.1.4 *closed-loop, adj*—refers to a testing mode in which the collection medium volume is fixed.

3.1.5 *collection medium, n*—a liquid, gas, or solid that absorbs, adsorbs, dissolves, suspends, or otherwise captures the challenge chemical and does not affect the measured permeation.

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

3.1.6.1 *Discussion*—For protective clothing materials, changes in physical properties are typically of most interest.

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

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

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

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

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

3.1.10 *penetration, n*—for a protective clothing material or item, the process by which a substance moves through

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

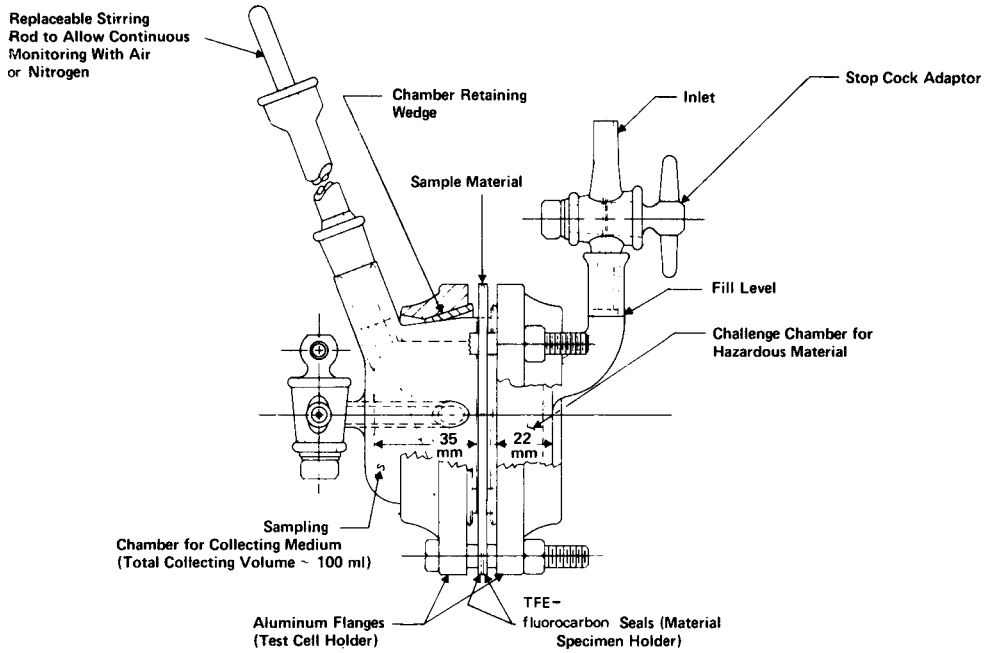


FIG. 2 Alternative Permeation Test Cell Design

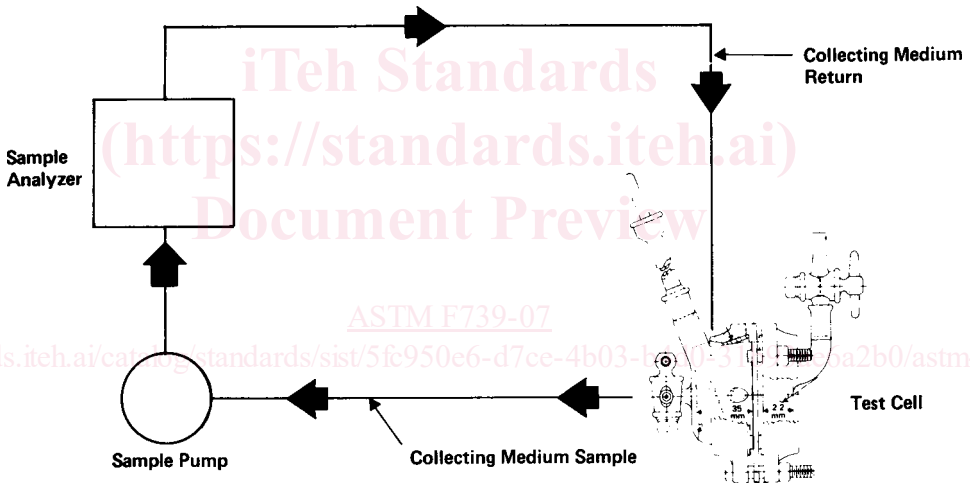


FIG. 3 Example Set-up for Continuous Collecting Medium Sample Withdrawal, Analysis, and Return

closures, seams, interstices, and pinholes or other imperfections on a non-molecular level.

3.1.10.1 *Discussion*—The substance may be a solid, liquid, gas, or biological species.

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

3.1.11.1 *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.

3.1.12 *protective clothing material, n*—any element, constituent, or substance from which protective clothing is composed or can be made.

3.1.13 *standardized breakthrough time, n*—the time at which the permeation rate reaches 0.1 μg/cm^2/min.

3.1.14 *steady-state permeation, n*—the constant rate of permeation that occurs after breakthrough when the chemical contact is continuous and all forces affecting permeation have reached equilibrium.

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.

4.2 In the permeation test apparatus, the protective clothing material specimen partitions the challenge chemical from the collection medium.

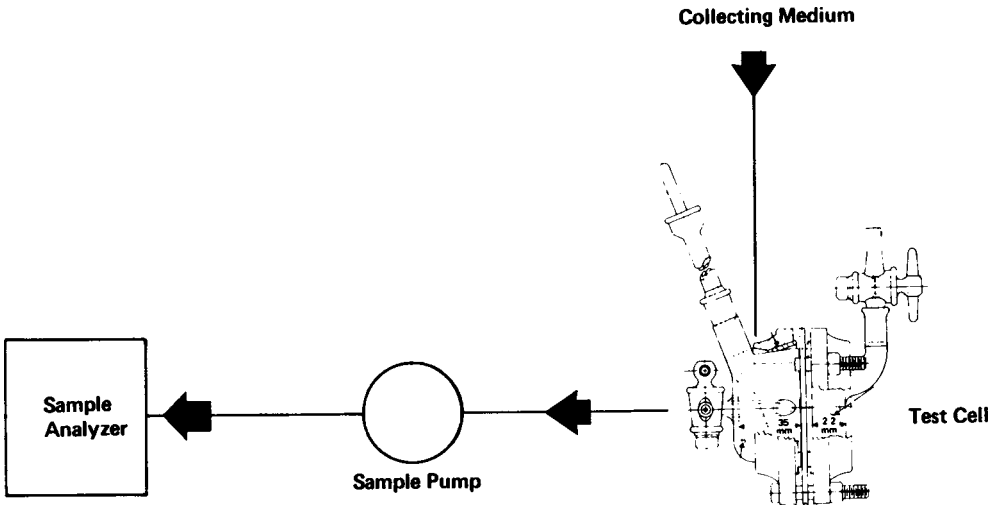


FIG. 4 Example Set-up for Continuous Flow of Fresh Collecting Medium

4.2.1 The collection medium is analyzed quantitatively for its concentration of the challenge chemical and thereby the amount of that chemical that has permeated the barrier as a function of time after its initial contact with the material.

4.2.2 By either graphical representation or appropriate calculations, or both, the breakthrough detection time, standardized breakthrough time, and the permeation rate of the challenge chemical are determined.

5. Significance and Use

5.1 This test method is normally used to evaluate flat specimens from finished items of protective clothing and from materials that are candidates for items of protective clothing.

5.1.1 Finished items of protective clothing include gloves, arm shields, aprons, suits, hats, boots, respirators, and the like.

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

5.2 The breakthrough detection time, standardized breakthrough time, and permeation rate are key measures of the effectiveness of a clothing material as a barrier to the challenge 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 detection times and low permeation rates are characteristics of better barriers.

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

5.3 The sensitivity of the test method in detecting low permeation rates or amounts of the challenge chemical that permeate is determined by the combination of the analytical technique and collection system selected, and the ratio of material specimen area to collection medium volume or flow rate.

5.3.1 The analytical technique employed should be capable of measuring the concentration of the challenge chemical in the collection medium at, or below, levels consistent with standardized breakthrough time value specified in 3.1.13 and at, or above, the steady-state permeation rate.

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

5.3.3 Higher ratios of material specimen area to collection medium volume or flow rate permit earlier detection of breakthrough and detection of lower permeation rates because higher concentrations of the challenge chemical in the collection medium will develop in a given time period, relative to those that would occur at lower ratios.

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

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

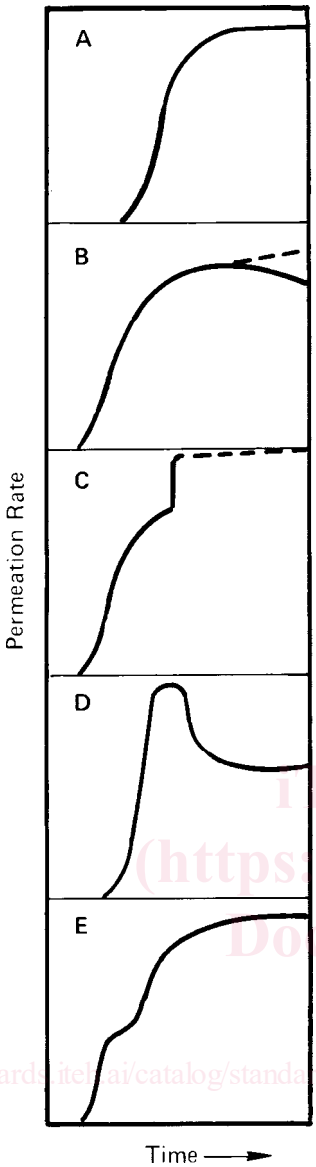
5.4.2 The sensitivity of a closed-loop system is characterized by its minimum detectable mass permeated.

5.5 A group of chemicals for use in permeation testing is given in Guide F1001.

5.6 These test procedures are also a part of ISO 6529. ISO 6529 provides a harmonized standard that also permits using some practices commonly followed in Europe for permeation testing, for example, using a breakthrough time normalized at a permeation rate of 1.0 μg/cm^2/min instead of 0.1μg/cm^2/min as used in this method. For this reason, the reporting of all permeation data must include the method that is used in the testing. Guide F1194 provides guidance on reporting permeation test results.

6. Apparatus

6.1 *Thickness Gauge,* suitable for measuring thicknesses to the nearest 0.02 mm (or the nearest 0.001 in.), as specified in



NOTE 1—Fig. 5 shows five types of permeation behavior. Type A, the most typical, where the permeation rate stabilizes at a "steady state" value. Type B behavior is due to the material specimen being structurally modified by the chemical resulting in an increase or decrease in permeation rate. Type C behavior occurs when the material specimen exhibits a sudden, very large increase in rate. Type D response happens when there is moderate to heavy swelling of the material specimen although the permeation rate eventually stabilizes. Type E response can occur when there is a high degree of swelling.

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FIG. 5 Five Types of Permeation Behavior

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 challenge chemical on

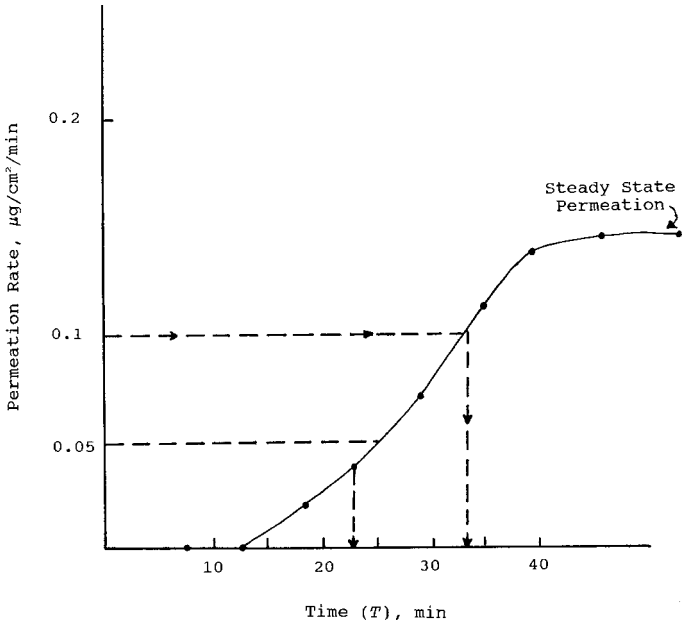

FIG. 6 The Breakthrough Detection Time for a method sensitivity of 0.05 µg/cm²/min is 23 minutes. The Standardized Breakthrough Detection Time is 33 minutes. The Steady State Permeation Rate is approximately 0.15 µg/cm²/min.

the specimen's normally outside surface and with a collection medium on the specimen's normally inside surface.

6.3.1 *Liquid Challenge Chemical,* for liquid chemicals, the test cell,[4] shown in Fig. 1, 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 challenge 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.

6.3.1.2 Inlet and outlet ports, with stopcock valves, if desired, are added to each chamber to enable the introduction and withdrawal of challenge 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 challenge 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

[4] The test cell as shown 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, are satisfactory for this purpose.

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 challenge 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 challenge chemical should be made from material that does not absorb or react with the challenge 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 via PTFE pressure-fit union connectors.[8]

6.3.2.2 In closed-loop tests where increased analytical sensitivity is required, a shorter length chamber 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 challenge 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 challenge chambers clamped together, provided that the collection medium can be mixed, withdrawn, and replenished as needed during the test.

6.3.3 For gaseous challenge chemicals, the test cell consists of two collection chambers clamped together. Thus, the gaseous challenge chemical can be circulated from its reservoir through the challenge-side chamber. Flow must be such that the composition and concentration of the gas in the test chamber does not change with time, and the test gas in the chamber is well mixed.

6.4 *Alternative Test Cell*—Alternative permeation test cells may be used, provided that the results are reported as prescribed in Section 12. The cells and configuration described above and shown in Fig. 1, however, are the standards. If a different cell is used, it must be documented as described in Section 12. An alternative design that has been documented is shown in Fig. 2.

6.5 *Constant Temperature Chamber or Bath,* used to maintain the test cell within ±1.0°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, or challenge chemical, or both, through the test cell. All parts contacting the challenge chemical or fluid containing it must be chemically inert and non-absorptive to the challenge chemical. The flow rate must be sufficiently high to provide adequate mixing, or dilution, or both 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), or the collection chamber of the test cell, or both. A calibrated device, accurate to ±0.5°C must be used.

7. Safety Precautions

7.1 Before this test method is carried out, safety precautions recommended for handling any potentially hazardous chemical should be identified and reviewed to provide full protection to all personnel.

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 Consideration

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 challenge 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 a minimum cross dimension of 43 mm (1.7 in.). A 51-mm (2-in.) diameter circle is convenient.

[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 Berghoff America, 3773 NW 126th Ave, Building 1, Coral Springs, FL 33065, http://www.berghofusa.com/.