Designation: F739 – 12$^{\varepsilon 1}$

Standard Test 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.

ε[1] NOTE—Editorially corrected 8.8.1 in February 2015.

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 test chemical. Resistance to permeation under the condition of intermittent contact with the test 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 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 Values states in SI units are to be regarded as standard. Values given in parentheses are not exact equivalents and are given for information only.

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

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[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

[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.

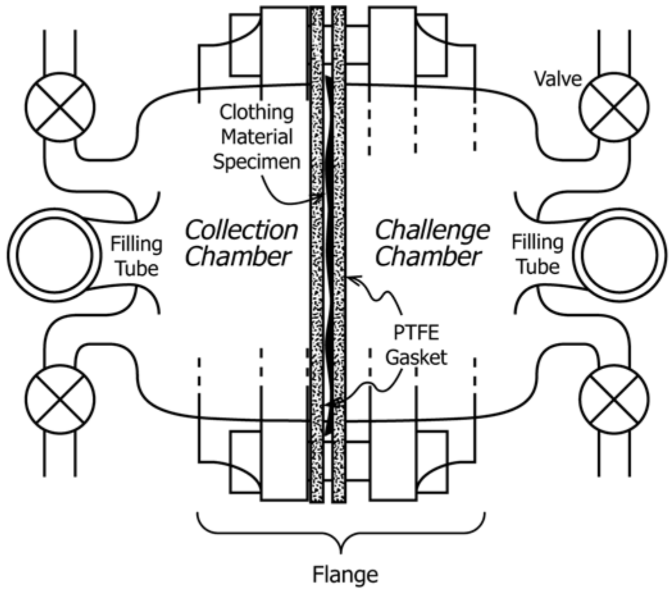


FIG. 1 ASTM Permeation Test Cell

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
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
F2815 Practice for Chemical Permeation through Protective Clothing Materials: Testing Data Analysis by Use of a Computer Program

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 test 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 initial exposure to the test chemical to the sampling time that immediately precedes the sampling time at which the test 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 *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 *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 *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 test chemical that is detectable with the complete permeation test system.

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

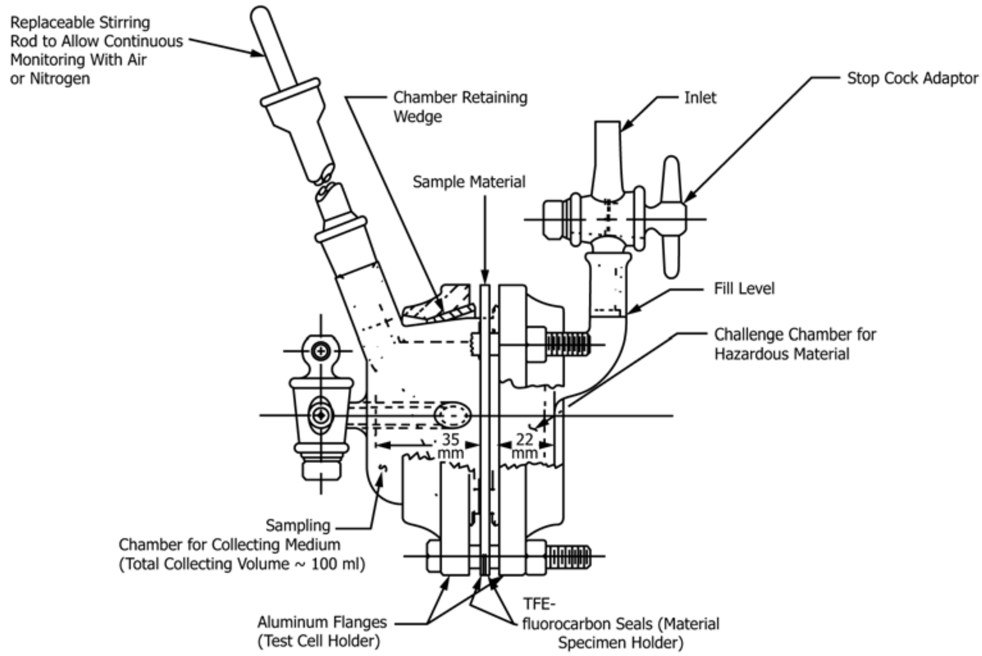


FIG. 2 Alternative Permeation Test Cell Design

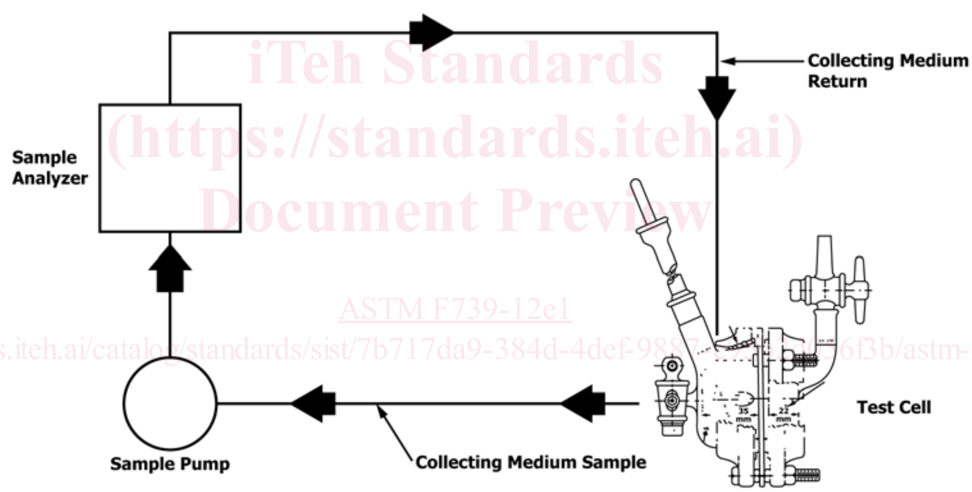


FIG. 3 Example Set-up for Continuous Collecting Medium Sample Withdrawal, Analysis, and Return

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 chemical protective clothing, the movement of substances through voids in protective clothing materials or items on a non-molecular level.

3.1.10.1 *Discussion*—Voids include gaps, pores, holes and imperfections in closures, seams, interfaces and protective clothing materials. Penetration does not require a change if 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.

3.1.11 *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.

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

3.1.12 *protective clothing, n*—item of clothing that is specifically designed and constructed for the intended purpose of

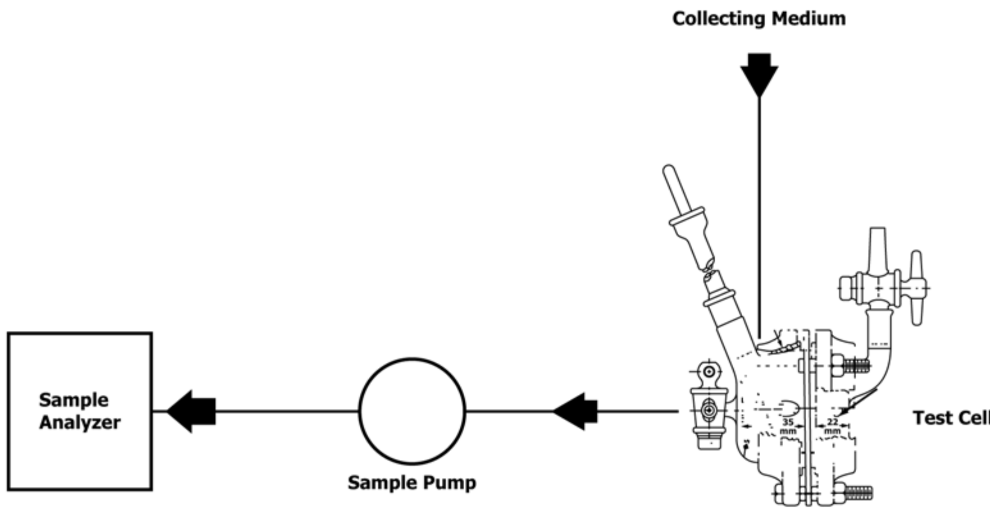


FIG. 4 Example Set-up for Continuous Flow of Fresh Collecting Medium

isolating all or part of the body from a potential hazard; or, isolating the external environment from contamination by the wearer of the clothing.

3.1.13 *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.

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

3.1.15.1 *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, subsequent permeation rate, and cumulative permeation over a period of time through replicate specimens of the material.

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

4.2.1 The collection medium 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.

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

5. Significance and Use

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

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

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, permeation rate, and 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, long standardized breakthrough detection times, low amounts of cumulative permeation, and low permeation rates are characteristics of better barriers.

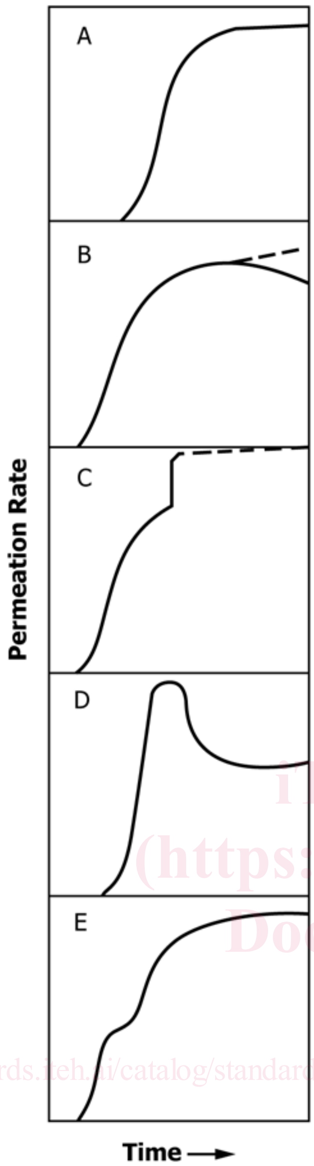
NOTE 1—At present, only limited quantitative information exists about acceptable levels of dermal contact with most chemicals. Therefore, the data obtained using this test method cannot be used to infer safe exposure levels.

5.2.1 The reporting of a standardized breakthrough time greater than a specific time period means that the test chemical has not permeated the specimen at a rate exceeding 0.1 µg/cm^2/min in the designated time. Permeation may or may not have occurred at a lower rate during this time interval.

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

5.3.1 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 the 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 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.



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

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 and levels of cumulative 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.

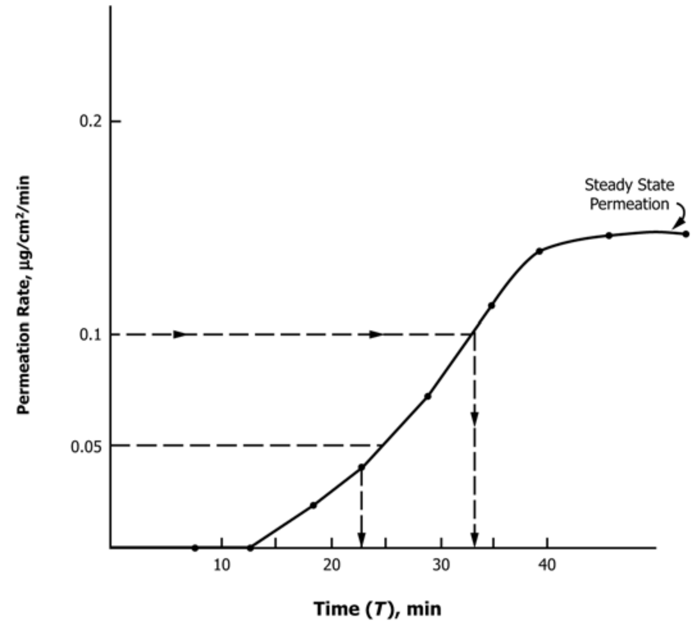

FIG. 6 The Breakthrough Detection Time for a method sensitivity of 0.05 µg/cm²/min is 23 minutes. The Standardized Breakthrough Detection Time is 33 minutes. The Steady State Permeation Rate is approximately 0.15 µg/cm²/min.

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/cm²/min instead of 0.1µg/cm²/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 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 normally inside surface.

6.3.1 *Liquid Test 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 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.

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 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 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 reduce the volume of the collection medium. This increases the sensitivity of the method by increasing 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 two chambers of the test cell must permit the mixing, withdrawal and replenishment of the collection medium during the test.

6.3.3 For gaseous test chemicals, the test cell can consist of two chambers one of which allows the gaseous test chemical to be circulated from its reservoir. 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 test chemical, or both, through the test cell. All parts contacting the test chemical or fluid containing it must be chemically inert and non-absorptive to the test chemical. The flow rate must be sufficiently high to provide adequate mixing, 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,

[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.

[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.