



**Designation: E2945 – 14**

# Standard Test Method for Film Permeability Determination Using Static Permeability Cells<sup>1</sup>

This standard is issued under the fixed designation E2945; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the measurement of the transmission of a gas through plastic membranes, sheeting, films, and fabric materials using a static sealed diffusion chamber. The test method monitors gas diffusion across a film membrane and provides measurements of (1) gas concentrations on each side of the film membrane and (2) estimates of the mass transfer coefficient (MTC) for the tested gas and film material. The MTC represents the film permeability and is independent of the concentration gradient used during testing, which simplifies some aspects of the experimental design.

1.2 This test method permits the loading of mixed vapors and simultaneous determination of the permeability of one film to various gases.

1.3 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this 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 applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

D618 Practice for Conditioning Plastics for Testing

D1898 Practice for Sampling of Plastics (Withdrawn 1998)<sup>3</sup>

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

<sup>1</sup>This test method is under the jurisdiction of ASTM Committee E35 on Pesticides, Antimicrobials, and Alternative Control Agents and is the direct responsibility of Subcommittee E35.22 on Pesticide Formulations and Delivery Systems.

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<sup>2</sup>For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup>The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

## 3. Terminology

### 3.1 Definitions:

3.1.1 *concentration, C, n*—chemical mass divided by the chamber volume.

3.1.1.1 *Discussion*— $C_o$  is the initial ( $t = 0$ ) concentration in the source chamber. The SI unit of concentration is  $\mu\text{g}/\text{cm}^3$ .

3.1.2 *concentration gradient, n*—difference in the concentration of gases across the film membrane divided by the transport distance between the source and collection chambers (for example, usually considered to be the film thickness).

3.1.2.1 *Discussion*—The SI unit of the concentration gradient is  $\mu\text{g}/\text{cm}^3\text{-cm}$ .

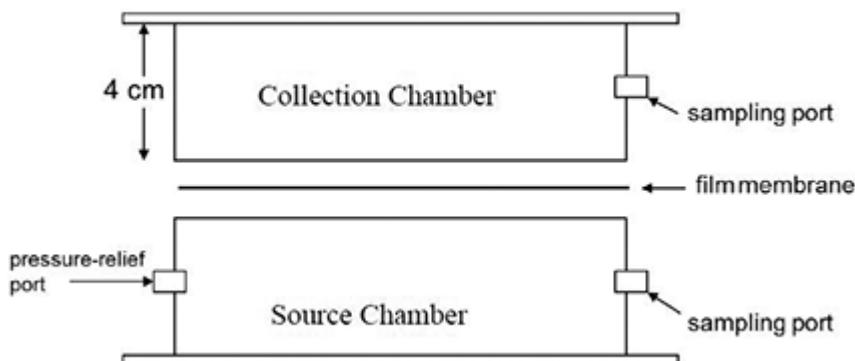
3.1.3 *mass transfer coefficient, MTC, n*—gas diffusion rate constant that relates the mass transfer rate, distance, and concentration gradient as the driving force through a film membrane under the test conditions.

3.1.3.1 *Discussion*—The SI unit of the MTC is  $\text{cm}/\text{hour}$ . The MTC expresses the ease of transmission of a gas through a membrane under test conditions. The test conditions shall be stated, which include the ambient temperature, relative humidity, film conditioning, sampling, and handling.

3.1.4 *mass transfer rate, J, n*—mass transfer rate, or flux density, of a gas diffusing through a film membrane is the mass of gas passing through a unit area (for example,  $1 \text{ cm}^2$ ) of film membrane per unit time interval (for example,  $1 \text{ h}$ ). The SI unit of  $J$  is  $\mu\text{g}/\text{cm}^2 \text{ hour}$ .

## 4. Summary of Test Method

4.1 This test method uses a static sealed apparatus consisting of two chambers separated by the test-film membrane. The test chemical in the vapor phase is added to the chamber on one side of the film and the apparatus is incubated at constant temperature during which the chemical diffuses through the test membrane. This test method requires determination of the relative chemical concentrations on both sides of the membrane at several time points during the incubation. Concentrations are monitored until equilibrium is reached or some other practical stoppage time. For permeable films, more frequent sampling is necessary because equilibrium may be reached within minutes or hours. For films with very low permeability, longer incubation times (weeks) may be necessary to reach



**FIG. 1 Schematic of Static Film Permeability Apparatus Consisting of Two Parts: A Source and Collection Chamber with a Film Membrane between Them**

equilibrium. Linear regression of data may be used to calculate the mass transfer coefficient (MTC). Alternatively, an analytical solution to a mathematical model may be used to calculate MTC (see [Appendix XI](#)) for which a nonlinear least-square algorithm is available to fit concentrations derived from the mathematical model to the observed concentrations. See Papiernik et al<sup>4,5</sup> for additional details.

## 5. Significance and Use

5.1 This test method provides a simple approach for determining the transmission properties of film membranes and sheeting over a range of permeability exceeding four orders of magnitude. This test method is described here to measure the permeability of films used in soil fumigation, but it is also appropriate for other gases and membranes if the analytical methods are appropriately modified.

5.2 This test method can be used for single or mixed compounds. This test method uses small quantities of test chemicals in vapor form, and microgram to milligram quantities of each chemical may produce a sufficient amount of vapor for each test depending on the analytical methods.

5.3 Interlaboratory testing showed that the MTC estimated by this test method is relatively insensitive to the laboratory procedures. The interlaboratory testing involved measuring the MTC for several soil fumigant compounds and a wide range of film permeability. Analysts with prior experience handling and analyzing gaseous fumigant compounds had lower coefficients of variation (10 to 20 %) compared to less experienced analysts (20 to 50 %) based on triplicate tests. The coefficient of variation between laboratories was higher for less permeable film materials than for films with high MTC. This was attributed to the additional length of the experiments and potential for increased leakage from the apparatus and was most pronounced for less experienced analysts.

## 6. Apparatus

6.1 A sealed apparatus is constructed of inert and impermeable material (for example, stainless steel) such that a sample of test membrane is held between the two chambers in a closed system. The selection of material is dependent on the gases being considered. The apparatus (see [Fig. 1](#)) enables sampling of the time rate of change in the gas concentration in each chamber and the mass transfer coefficient. The apparatus is configured as shown in [Fig. 1](#).

6.1.1 *Permeability Apparatus*—Stainless steel pipe (for example, 0.3 to 0.6 cm thick, 10- to 15-cm diameter) is cut to form cylinders with height 2 to 6 cm. The volume of the chamber affects the time to reach equilibrium; therefore, taller cylinders are appropriate for testing permeable films, shorter cylinders for less permeable films. The ends of the pipe are trued and the mating surfaces smoothed. Each cylinder is welded to a flat steel plate (for example, 0.3 cm thick) at one end, as shown in [Fig. 2](#).

6.1.2 *Sampling Ports*—Holes are drilled and threaded on the side of each cylinder to allow installation of sampling ports. The holes should be located near the mid-point height of the cylinder ([Figs. 1](#) and [2](#)).

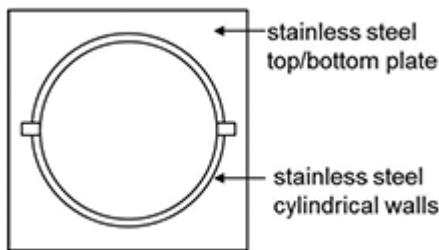
6.1.3 The purpose of the ports is to allow access to the inside of the chamber for spiking and sampling. During other times, ports should be sealed to prevent leakage. This can be accomplished using a septum port or sampling valve as described in [6.1.3.1](#) and [6.1.3.2](#).

6.1.3.1 *Septum Port*—A 1.6-mm steel (or brass) union connector is installed in each hole. Before installation, the threads of the union are coated with epoxy to ensure a gastight seal. One port is installed in the collection chamber and two ports (on opposite sides of the cylinder) are installed in the source chamber. The second port is used to vent the source chamber during spiking. A septum and threaded nut are installed onto the 1.6-mm union and the union threads coated with epoxy. The threaded nut is covered by a Swagelok<sup>6</sup> cap and a septum ([Fig. 3A](#)). Samples are collected with a syringe

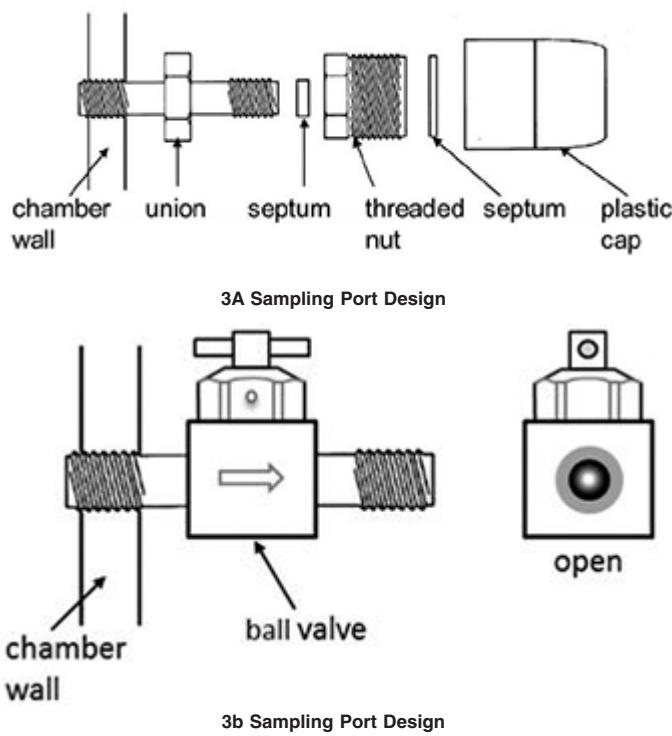
<sup>4</sup> Papiernik, S. K., Yates, S. R., and Gan, J., "An approach for estimating the permeability of agricultural films," *Environmental Science and Technology*, Vol 35, 2001, pp. 1240-1246.

<sup>5</sup> Papiernik, S. K., Ernst, F. F., and Yates, S. R., "An apparatus for measuring the gas permeability of films," *Journal of Environmental Quality*, Vol 31, 2002, pp. 358-361.

<sup>6</sup> Swagelok is a registered trademark of the Swagelok Company, Cleveland, Ohio.



**FIG. 2 Top View of the Source Chamber—A Stainless Steel Cylinder Is Welded to the Stainless Steel Bottom Plate Leaving One End of the Cylinder Open**



**FIG. 3 Sampling Port Design**

by removing the outer septum and cap and piercing through the septum behind the threaded nut (Fig. 3A). Between sampling, the nonpunctured septum and cap should be tightened over the threaded nut to prevent leakage from the pierced septum between sampling times.

**6.1.3.2 Sampling Valve Port**—A gastight sampling valve is screwed onto the union (Fig. Fig. 3A) or directly into the chamber wall and the threads sealed with epoxy (Fig. Fig. 3B). One valve is installed in the collection chamber and one valve is installed in the source chamber. The valve shall be made of inert and impermeable material and produce a gastight connection to the cylinder wall. A polytetrafluoroethylene stopcock screwed onto the union allows sample introduction or removal. A stainless steel two-way valve (1.6 mm) screwed directly into

the drilled hole could also be used to allow sample introduction or removal (Fig. Fig. 3B). The air volume within the valve should be minimized.

**NOTE 1**—Other configurations for the chamber access ports are possible, but design criteria and testing should demonstrate that they: (1) are constructed of inert materials, (2) are non-leaking between sampling times, (3) minimize leaking during sampling, and (4) maintain integrity during routine laboratory handling.

## 7. Materials

**7.1** The apparatus can be used to measure diffusion of an arbitrary gas through a film membrane. The specifics of the methodology described in the following relate to fumigant

gases and fumigation films, but the test method can be modified to allow measuring the MTC for other gases and other membranes.

**7.2 Fumigant Chemicals**—Iodomethane, 1,3-dichloropropene (mixture of cis and trans isomers), dimethyl disulfide, methyl isothiocyanate (transformation product of metam sodium or dazomet during fumigation), chloropicrin, methyl bromide, and sulfuryl fluoride.

**7.3 Gas-Mixing Chamber**—Gastight 1-L glass container with valves on both ends and a side sampling port. Other types of gastight containers with sampling ports may be used. If a clear glass container is used, it is recommended that the glass container be wrapped with aluminum foil to protect the fumigants from light. Some fumigants are photodegradable.

**7.4** A constant-temperature environmental chamber is used to maintain constant temperature during testing. Since the temperature is known to affect the MTC value, the variation in the temperature set point should be no more than  $\pm 2^{\circ}\text{C}$ .

**7.5 Miscellaneous**—An assortment of gastight syringes (for example, 10- $\mu\text{L}$  to 100-mL capacity), Tedlar bag with sampling port (for example, 0.6-L capacity), gas chromatograph autosampler vials, caps that are inert to the test gas, crimpers, timers, epoxy glue, aluminum adhesive tape.

**7.6 Gas Chromatograph/Mass Spectrometer Equipped with Appropriate Capillary Column**—A gas chromatograph (GC) with electron capture detector (ECD) can also be used for analysis of halogenated fumigants, such as methyl bromide, iodomethane, chloropicrin, 1,3-dichloropropene, and sulfuryl fluoride. Equipment that includes an autosampler provides added convenience.

**7.7 Other Gases**, appropriate sampling and detection equipment as needed.

## 8. Potential Hazards

**8.1 General**—Appropriate laboratory and chemical safety procedures should be followed and materials and gases should be used in accordance with information provided on product labels, safety data sheets, and established laboratory safety guidelines.

**8.2 Gases under Pressure**—When using gases stored under high pressure, the dispensing equipment should be appropriate for the intended use. The equipment should be rated for the gas cylinder or gas-line pressures, or both, and pressure-reducing valves and regulators used where needed.

**8.3 Fumigation gases** are a class of chemicals that pose significant health hazards. They generally are irritants and toxic. Adverse human health effects include harm if inhaled, swallowed, or absorbed through the skin; appropriate safety procedures should be used.

## 9. Sampling, Test Specimens, and Test Units

**9.1** Test specimens should be sampled in accordance with Practice D1898. Tested samples should be representative of the bulk material; free of wrinkles, stretches, pinholes, other

imperfections; and of uniform thickness. Surface condition and differences in materials or construction of each side of the film shall be reported.

**9.2** Cut the film test specimens into approximately 15- by 15-cm pieces.

**9.3** Information concerning the film composition (for example, thickness, presence of ultraviolet [UV] stabilizers, barrier polymers and additives, and so forth) and manufacturing should be reported, when available.

## 10. Preparation of Apparatus

**10.1** Mix together a small amount of the epoxy resin and hardener. Spread a thin layer of the well-mixed epoxy glue over the exposed rim of the open edge of the source chamber side of the permeability apparatus using a flat stainless steel spatula. Place the test film onto the edge containing the glue. Make sure the film is spread flat and evenly (not stretched and with no crevices). Spread a thin layer of well-mixed epoxy glue over the exposed rim of the collection chamber of the permeability apparatus. Carefully place the rim of collection chamber over the film and mate the two halves of apparatus by aligning and joining them together to form a gastight seal. Care should be taken to place the film and mate the two chambers with minimal movement after contact.

**10.2** After the glue is cured (usually overnight), trim the excess film with a razor blade. Apply aluminum tape to the outside of the apparatus over the seam between chambers and burnish to provide additional support and sealing of the apparatus. Place the constructed apparatus inside a temperature-controlled environment set at the target temperature and equilibrate for a minimum of 60 min before introducing the fumigants.

NOTE 2—The time needed to reach temperature equilibrium is dependent on the materials and quantities used for the apparatus. A preliminary study should be conducted to determine the equilibrium time for a particular test apparatus, and the measured equilibrium time should be used during testing.

**10.3 Replication**—In general, triplicate permeability apparatuses are constructed for each test film and the MTC is calculated for each replicate. The average and standard deviation of the triplicates should be reported.

## 11. Calibration and Standardization

**11.1 Quantitation**—Determine instrument response for each fumigant by injecting fumigant mixtures at varying concentrations into the instrument and creating a calibration curve. Using the same procedure as in 13.3.1, transfer aliquots (for example, 5, 10, 20, 50, 100, and 500  $\mu\text{L}$ ) of the test vapor from the 1-L mixing chamber (13.1) into vials. The fumigant concentrations in the vials are estimated using the values from 13.1.1.4 (Table 1 as an example) and the volume of the standard mixture placed in the vial.

**11.1.1** This method of preparing standards is suggested because absolute concentrations are not required for these tests. Other methods of constructing calibration curves that result in more exact determination of chemical concentration are acceptable so long as they conform to the standards of analytical chemistry.

**TABLE 1 Estimated Concentrations of Fumigants Present in the 1-L Mixing Chamber Based on the Suggested Procedures in 13.1.1.1 – 13.1.1.3**

Fumigant	State	Gas or Liquid Density (mg/mL)	Amount of Pure Material Added to Mixing Chamber	Calculated Concentration in Mixing Chamber (µg/mL of air)
Sulfuryl fluoride	gas	4.24	30 mL of gas (from Tedlar bag)	127 µg/mL
Methyl bromide	gas	3.97	30 mL of gas (from Tedlar bag)	119 µg/mL
1,3-dichloropropylene <sup>A</sup>	gas	1.22	40 µL	24.4 µg/mL <sup>A</sup>
Iodomethane	liquid	2.28	20 µL	45.6 µg/mL
Chloropicrin	liquid	1.69	20 µL	33.8 µg/mL
Dimethyl disulfide	liquid	1.06	20 µL	21.2 µg/mL
Methyl isothiocyanate	solid	–	20 mg	20 µg/mL

<sup>A</sup> 1,3-dichloropropene has two isomers (cis, trans) in an approximately 50:50 mixture. The calculated concentration of each isomer in the mixing chamber is 24.4 µg/mL.

11.2 The concentration of fumigant in each chamber of each apparatus during a test is determined by comparing the instrument response for each sample against the instrument calibration curve.

11.3 *Alternative Measurements*—The methodology used to calculate the MTC uses the ratio  $C/C_o$  in the source and collection chambers. Alternative ratios, for example, peak area divided by peak area in source chamber at the start of the test, can also be used if the instrument response is linear and provides identical results.

## 12. Conditioning

12.1 *Standard Conditioning*—In accordance with Practice D618 Procedure A for films with thickness less than 7 mm, condition all test specimens in a laboratory at standard conditions (that is,  $23 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  relative humidity) for 40 h or more before attaching the film membrane to the permeability apparatus and sealing with aluminum tape.

12.2 *Other Temperatures*—When tests are required at other temperatures, the film should be conditioned at the test temperature.

12.3 *Other Relative Humidity*—When tests are required at nonstandard relative humidity, the film and constructed apparatus should be conditioned at the test relative humidity in accordance with Practice D618. The conditioning and relative humidity of the collection and source chambers shall be reported.

12.4 *In-Situ Conditioning*—Prepare apparatus as in Section 10 and then sweep air at standard conditions through the assembled apparatus for 40 min or more before initiating a test.

## 13. Procedure

### 13.1 Preparation of Test Vapor:

#### 13.1.1 Fumigant Mixture Preparation:

13.1.1.1 *Solids*—Transfer a small amount of solid fumigant (for example, methyl isothiocyanate) (about 20 to 50 mg) into the 1-L glass chamber.

13.1.1.2 *Liquids*—Transfer about 20 to 50 µL of each liquid fumigant standard into the 1-L glass mixing chamber using a pipette or syringe.

13.1.1.3 *Gases*—In a fume hood, transfer a small amount (about 100- to 500-mL volume) of each gas (for example, methyl bromide and sulfuryl fluoride) from a compressed gas cylinder into a Tedlar bag, for example, using a small piece of

copper tubing, a step-down regulator, and a short piece of flexible tubing attached to a syringe needle. Using a gastight syringe, transfer about 30 mL of the collected gaseous compounds from the Tedlar bag to the 1-L mixing chamber.

NOTE 3—The fumigants should be left in the mixing chamber for a minimum of 30 min to allow equilibration of the concentration inside the mixing chamber before use. The mixing chamber may be placed in a warm place (for example, up to  $40^\circ\text{C}$  oven) to facilitate the vaporization of the fumigants. Methyl bromide and sulfuryl fluoride diffuse through the Tedlar bag and degrade over time and, therefore, cannot be stored in a Tedlar bag for long periods. Also, some fumigants, such as methyl iodide, chloropicrin, 1,3-dichloropropene, and methyl isothiocyanate are photo-sensitive and degrade quickly when exposed to light. Exposure of the containers containing fumigants to light should be minimized.

13.1.1.4 The estimated concentration of each fumigant, expressed as µg/mL, in the 1-L glass chamber can be estimated based on the assumption that the entire amount of each fumigant has completely evaporated in the chamber and the resultant gases are well mixed. Assuming complete vaporization, the estimated concentration of each fumigant in the vapor phase of the mixing chamber is calculated based on the amount added (for example, mass) of each compound divided by the chamber volume (1 L). Since complete vaporization and mixing within the chamber cannot be verified, the calculated chamber concentrations should be considered estimates. Table 1 summarizes the approximate concentrations for the stated amounts using 13.1.1.1 – 13.1.1.3.

13.1.1.5 The amount of each fumigant transferred to the mixing chamber and the subsequent transferring of gas to the apparatus can vary, as long as a sufficient quantity of gas is present in the apparatus for instrumental analysis. Therefore, an excessive quantity may be transferred to the mixing chamber to provide a saturated vapor. After establishing the linear range of the analytical instrument, quantitative transferring of a given quantity of fumigant vapor to the apparatus is not required because the use of concentration ratios, that is,  $C/C_o$  or equivalent, is sufficient.

13.1.1.2 *Mixture Preparation for Other Gases*—The procedure in 13.1.1 can be modified to enable estimation of the MTC for gases other than fumigants.

13.2 *Adding Test Gas to Apparatus*—Temporarily move the apparatus from the temperature chamber to a fume hood. Close the collection chamber port, open the source chamber port, and then withdraw approximately 30- to 40-mL volume of the vapor from the 1-L mixing chamber using a gastight syringe. Inject the vapor into the source chamber (typically the bottom

chamber) of the permeability apparatus and immediately close all valves/ports. Start timer to track incubation time. Return apparatus to the temperature chamber for incubation.

**NOTE 4**—If the apparatus includes a septum/cap (6.1.3.1), the venting valve should be opened before injection to avoid pressurizing the chambers. This can be accomplished by inserting a small-diameter needle through the inside septum of the venting port. If an on/off valve is used (6.1.3.2), the excess air/vapor will escape around the needle and no venting valve is needed.<sup>7</sup> The amount of the vapor injected into the source chamber of the permeability apparatus may be adjusted to obtain a sufficient amount of compound(s) to be analyzed depending on instrument sensitivity.

### 13.3 Sampling Gas from Apparatus:

13.3.1 At the appropriate sampling interval, use gastight syringes to withdraw equal and fixed volume of gas samples (for example, 250 µL) from both the collection and source chambers of each permeability apparatus. Note the exact sampling time for each replication. Follow one of the extraction procedures in 13.3.1.1 or 13.3.1.2 depending on the method of instrumental analysis.

**NOTE 5**—Dedicated syringes should be used for sampling the source and collection chambers as a good laboratory practice. Dedicated syringes are essential for pesticides (for example, chloropicrin) that tend to adhere to the glass inside of the syringe. Using the same syringe to sample both chambers could lead to contamination of the low-concentration sample if the high-concentration sample (for example, source chamber) is collected first or if the syringe is not completely cleaned between sample collection times (see 13.3.3).

13.3.1.1 *Extraction Procedure A*—With the vial cap held askew on top of the vial, inject gas sample into the bottom of a 10-mL headspace autosampler vial. Close the vial immediately using aluminum crimp caps with polytetrafluoroethylene-faced butyl rubber septa.

13.3.1.2 *Extraction Procedure B*—Inject gas sample into the bottom of a GC vial or 10-mL headspace vial filled with approximately 2 mL of solvent. Close the vials immediately using aluminum crimp caps with polytetrafluoroethylene-faced butyl rubber septa.

13.3.2 If samples will not be analyzed immediately, store them in a manner that preserves sample integrity (for example, in the dark at -20°C for fumigants).

13.3.3 Flush each gastight syringe with air to reduce carry-over between samples.

13.3.4 If the design of the apparatus includes septum ports, sample collection using side-port needles may help avoid needle plugging as a result of coring the septum material during sampling.

**NOTE 6**—After each sampling, ensure that air flows freely through the needle. If a gastight syringe is used that incorporates an on/off valve, the needle can be tested by drawing air into the syringe, closing the valve, and confirming that the contents are pressurized when depressing the plunger with the valve closed (if the needle is open, pressure inside the syringe will increase and the plunger cannot be completely depressed).

13.3.5 *Suggested Sampling Times*—Periodic sampling typically begins 5 min after introduction of the fumigants to the source chamber with subsequent sampling dependent on film

**TABLE 2 Ions Monitored in the GC/MS Analysis**

Fumigant	Retention Time (min)	Primary Ion (m/z)	Secondary Ions (m/z)
Sulfuryl fluoride	1.5	102	83, 67
Methyl bromide	2.3	94	96, 79
Methyl iodide	3.6	142	127, 141
cis-1,3-dichloropropene	14.2	75	39, 110
Dimethyl disulfide	14.3	94	79, 45
Methyl isothiocyanate	16.0	73	45, 72
Chloropicrin	16.6	117	119, 82
trans-1,3-dichloropropene	16.7	75	39, 110

permeability. For high-permeability films, a sampling schedule of 5 min, 0.25, 0.5, 1, 2, 3, 4, 6, and 8 h may be used. For lower permeability films, longer sampling intervals may be used, for example, 5 min, 1, 4, 8, 24, 48, 72 h, and so forth. Very low-permeability films may require ten or more days to allow measurable amounts of compounds to permeate through the films and generate enough non-zero data points for calculation of the MTC. The purpose of frequent sampling is to obtain sufficient data points to calculate MTC reliably, particularly at the beginning of the experiment when changes in concentration are the largest.

13.3.6 *Testing Completion*—Move the apparatus to the fume hood and open the ports to allow test chemicals to escape. Disassemble the apparatus and remove the epoxy glue from the edges of the cylinders. Replace septa if apparatus used septum ports.

**NOTE 7**—For an apparatus that is difficult to disassemble due to epoxy bond strength, using a rubber mallet, or equivalent, and tapping one half of the apparatus may help to loosen the bond. Heating epoxy also weakens bond strength. A razor blade maybe used to remove the epoxy glue from the metal surfaces.

### 13.4 Sample Analysis:

13.4.1 *Analysis Equipment*—For fumigants and similar organic chemicals, gas samples are analyzed using a gas chromatograph/mass spectrometer (GC/MSD) with a headspace (if using 13.3.1.1) or liquid autosampler (for 13.3.1.2). A gas chromatograph/electron capture detector (GC/ECD) can also be used in place of GC/MSD for halogenated compounds.

13.4.2 *Suggested Headspace Autosampler Initial Conditions for Fumigants*—Oven temperature 80°C, loop temperature 90°C, transfer line temperature 100°C, equilibration time 3 min, carrier gas pressure 69 kPa, and vial pressure 97 kPa.

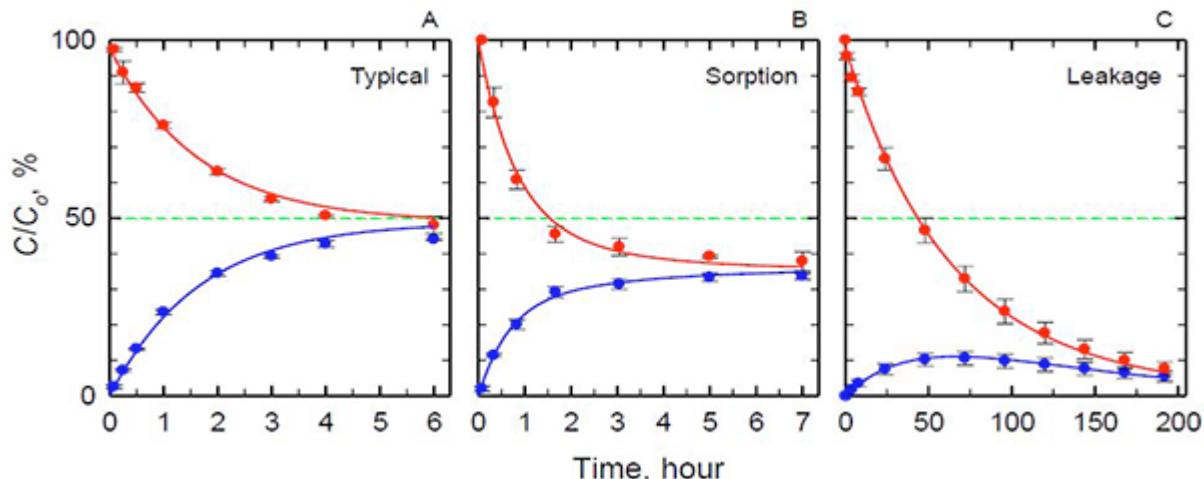
13.4.3 *Suggested GC Conditions for Fumigants*—DB-624 column (30-m by 0.25-mm inside diameter [ID], 1.4-µm film thickness); helium carrier gas, 1.2 mL/min; GC oven temperature: 40°C (hold for 3 min); increase at 10°C/min to 50°C (hold for 10 min.); and increase at 20°C/min to 110°C.

13.4.4 The mass spectrometer is operated in select ion mode (SIM). Instrument response of the primary ion is used for quantitation, while secondary ions are monitored for analyte confirmation or if there is interference with the primary ion. For fumigants, the ions monitored are listed in Table 2.

## 14. Calculation or Interpretation of Results

14.1 *Data Requirements*—The sampling times and concentrations of each gas in each chamber of each apparatus are used to calculate the MTCs.

<sup>7</sup> Qian, Y., Kamel, A., Stafford, C., Nguyen, T., Chism, W., Dawson, J., Smith, C. Evaluation of the permeability of agricultural films to various fumigants. Environmental Science & Technology 45:9711-9718. 2011



**FIG. 4 Concentrations (Three Replications) in Source (Upper Curve) and Collection (Lower Curve) Chambers Several Times after Injection—A and B are Tests with Relatively Permeable Film and C is a Film with Low Permeability—Symbols are Measurements, Error Bars are Standard Deviations, and Lines are Model Fits to the Measurements—Values are in Percent**

**14.2 Normalized Data**—The chemical concentration (or peak area) at each sampling interval may be normalized relative to the initial sampling, and the normalized values may be used for the MTC calculation. Values should be normalized to the sum of the concentrations in the source and collection chambers at the first sampling time (for example, 5 min).

NOTE 8—Normalizing the concentrations will lead to gas response values in the range zero to approximately one (or 0 to 100 %). This results in graphic output that has a more standardized format across a wide range of values.

**14.3 Monitoring the Diffusion Process**—The percent recovery of each compound at each sampling time (sum of source and collection chambers) is used to monitor the integrity of the apparatus (for example, leaks), processes occurring inside the apparatus (for example, sorption), and as a check for possible loss of the compounds during sampling and analysis. Values should be relative to the total amount applied,  $C_o$ , or the total amount present at the initial sampling (for example,  $t_o = 5$  min). It is typically reported as a fraction or with units of percent.

**14.3.1** When low recovery is encountered because of a leakage, chemical sorption to the film, or loss during sampling and analysis, the equilibrium concentrations in the source ( $C_s/C_o$ ) and collection chambers ( $C_r/C_o$ ), fall below 50 %.

**14.3.2 Calculation**—Recovery, as a fraction, can be estimated using:

$$\text{Recovery}(t) = \frac{C_s(t) + C_r(t)}{C_o} \approx \frac{C_s(t) + C_r(t)}{C_s(t_o) + C_r(t_o)} \quad (1)$$

NOTE 9—Low recovery as a result of leakage compromises a test result. In general, measurements collected from apparatus that exhibit extensive leakage should be discarded. Low recovery as a result of adsorption/absorption can be addressed by using a sorption model when determining the MTC (see Appendix XI). For fumigant chemicals, sorption has not been found to be a major complicating factor for many tested films, which include metalized polyethylene film, films comprised of polyethylene and barrier polymers (for example, nylon, ethyl vinyl alcohol), and polyethylene films with UV stabilizers and other common additives. However, a

>40-day test conducted using silver-mirrored Mylar®<sup>8</sup> and chloropicrin had recoveries of approximately 25 % but concentrations in the source chamber clearly reached equilibrium after several hours. This test was strongly affected by chloropicrin sorption to Mylar, so use of the sorption model was required to obtain the MTC.

**14.3.3** For long tests (for example, ten or more days), it is particularly important to monitor recovery as an indicator of leakage. In general, when the percent recovery (Eq 1) remains above 60 % for all sample times and all replicates, the concentration measurements can be considered acceptable. Otherwise, it will be necessary to determine if low recovery is due to leakage or sorption. If literature or experimental information is available that rules out sorption as a likely cause of low recovery, the concentrations measurements should be considered questionable. Conducting a test that includes an inert and nonreactive tracer gas could assist in identifying leakage, since losses of the tracer gas would presumably be due to leakage.

**14.3.3.1** There are many indicators of leakage, some are:

(1) Concentration in the source chamber that continually decreases and never approaches equilibrium (for example, see Fig. 4C);

(2) Nonmeasureable concentrations in the collection chamber and decreasing concentrations in the source chamber without reaching equilibrium;

(3) Concentrations in the collection chamber increasing early in the test and then continually decreasing (for example, see Fig. 4C);

(4) High variability in recovery of a gas between replicated apparatus. An outlier might be a result of leakage and a statistical test might help with identifying outliers; and

(5) The presence of the test gas inside a secondary containment vessel (for example, a sealed apparatus placed inside the containment vessel).

<sup>8</sup> Mylar® is a registered trademark of DuPont Teijin Films for its brand of polyester film. Only DuPont Teijin Films makes Mylar® brand films.

**14.4 Degrading Gases**—Compounds that degrade under test conditions may not be suitable for this test method unless an appropriate diffusion-degradation model is available. A degrading gas will also complicate isolation of sorption, leakage, sampling and analysis, and degradation effects.

**14.5 Computing the MTC**—The MTC is obtained using a model of the mass diffusion through the film. Several models have been developed, which vary in complexity depending on the processes that occur in the apparatus (see [Appendix X1](#)). The simplest models require the source and collection chamber lengths to be the same and are appropriate for gases that do not adsorb to the test film (see [X1.2.2.1](#)).

**14.5.1 No Sorption**—If gas sorption to the film can be ignored, an appropriate model for determining the MTC is shown in [X1.2.2](#). [Eq X1.4](#) and [X1.5](#) are used when the source and collection chambers have different lengths and [Eq X1.6](#) and [X1.7](#) when the chamber lengths are the same.

**14.5.1.1 Example of concentration data when chamber lengths are the same**—Examples of the changes in concentration with time in the source and collection chambers are shown in [Fig. 4](#). [Fig. 4A](#) shows data for methyl bromide and a high-density polyethylene film membrane. The shapes of these curves are indicative of minimal leakage or sorption, since the equilibrium concentrations are near 50 %. [Fig. 4B](#) shows data for *cis*-1,3-D and a high-density polyethylene film membrane. These data are indicative of chemical sorption to the film, since the equilibrium concentrations are below 50 % but have stabilized at about 35 %. [Fig. 4C](#) shows data for *trans*-1,3-D and a film with low permeability. These data suggest that leakage has occurred since the concentrations in both chambers are approaching zero.

**14.5.1.2 Example computing the MTC value using linear regression**—When  $C_s(t)/C_o$  and  $C_r(t)/C_o$  are fractions, the MTC can be obtained using the linear regression model,  $y = ht$ , as follows:

$$ht = -\frac{L}{2} \ln \left[ \frac{2C_s(t)}{C_o} - 1 \right] \quad (2)$$

$$ht = -\frac{L}{2} \ln \left[ 1 - \frac{2C_r(t)}{C_o} \right] \quad (3)$$

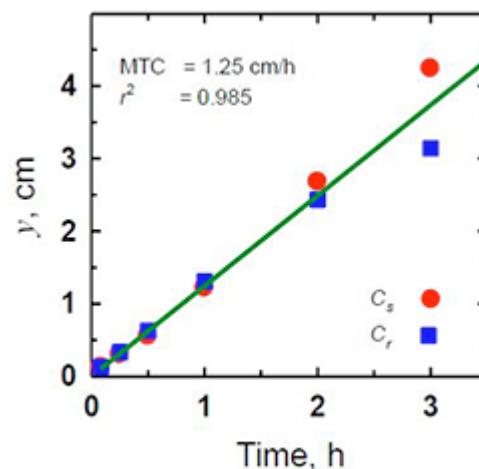
where  $h$  is the MTC.

(1) This example uses the data shown in [Fig. 4A](#). Since the equilibrium concentration at 6 hours is approximately 45 %, [Eq 2](#) cannot be evaluated for this sample value, since  $2 C_s(t)/C_o - 1$  is  $\leq 0$ . Nevertheless, the measurements up to 3 hours provide useful information.

**14.5.2 Sorption**—If there is significant sorption of the gas to the film membrane, the appropriate model for determining the MTC is [X1.2.3](#) (for example, [Eq X1.11-X1.13](#)). To use these equations, the length of the source and collection chambers shall be the same (for example,  $L_s = L_r = 4$  cm).

**14.5.2.1 Parameter Sensitivity**—Using the sorption model to obtain a value for the MTC is relatively insensitive to the sorption effect. The MTC depends primarily on the time to reach equilibrium. The sorption parameter,  $k_p$ , depends primarily on the equilibrium concentration values at the end of the test, and the sorption kinetic parameter,  $\alpha$ , depends on early-time behavior involving the time rate of change in the

**TABLE 3 Computation of the MTC Using Linear Regression Using Linear Regression, Using Data from [Fig. 4A](#). In this example,  $L = 4$**



Time, $t$ (h)	$C_s/C_o$ (fraction)	$C_r/C_o$ (fraction)	Y, cm	Total Amount
0.083	0.969		0.128	
0.083		0.031	0.128	1.000
0.250	0.930		0.302	
0.250		0.077	0.334	
0.500	0.879		0.554	
0.500		0.135	0.629	1.014
1.000	0.772		1.22	
1.000		0.240	1.31	1.012
2.000	0.631		2.68	
2.000		0.352	2.43	0.983
3.000	0.560 <sup>a</sup>		4.24	
3.000		0.396	3.14	0.956

<sup>a</sup> Only values greater than 0.5 are valid for [Eq 2](#). Only values less than 0.5 are valid for [Eq 3](#).

concentrations. The two sorption effects are relatively independent of the MTC, which leads to a reliable MTC estimation process.

**14.6 Leakage**—In general, leakage indicates experimental problems that should be corrected. However, it is possible to model leakage as a first-order loss process and the appropriate equations for determining the MTC are given in [X1.2.4.2](#) (for example, [Eq X1.18](#) and [X1.19](#)). The leakage solutions should be used with caution, since leakage from the apparatus may not follow the simple first-order process described in [X1.2.4](#), which could lead to significant errors in the MTC value.

**14.7 Film Permeability Calculator**—To facilitate calculation of MTC, a Windows-based software program (FilmPC v. 3.0.4, 2011) and FilmPC Excel add-in have been developed. These programs use a nonlinear least squares algorithm described by Marquardt<sup>9</sup> to obtain the model parameters and parameter statistics. The program also includes graphical, statistical, and reporting information. The FilmPC program and FilmPC Excel add-in can be obtained from the URL (search

<sup>9</sup> Marquardt, D. W., "An algorithm for least squares estimation of nonlinear parameters," *SIAM J. Appl. Math.*, Vol 11, 1963, pp. 431-441.

**TABLE 4 Precision Statistics for MTC (MTC, cm/h), Film 1, and Five Fumigants**

Material	$MTC_{ILS}$	$s_{MTC,ILS}$	$s_r$	$s_R$	$r$	$R$
Methyl bromide	1.0944	0.0476	0.1101	0.1101	0.31	0.31
Methyl iodide	1.9112	0.1474	0.2134	0.2282	0.60	0.64
cis-1,3-Dichloropropene	5.5414	1.3077	1.1793	1.6239	3.30	4.55
trans-1,3-Dichloropropene	7.6305	2.9079	1.8934	3.2933	5.30	9.22
Chloropicrin	2.1112	0.3294	0.2599	0.3918	0.73	1.10

FilmPC): <http://www.ars.usda.gov/Services/docs.htm?docid=15992>.

## 15. Report

15.1 The report shall include the following:

- 15.1.1 Date of testing and analyst conducting test;
- 15.1.2 Identification of the film material tested, including its thickness;
- 15.1.3 Identification of the gas(es) used during testing;
- 15.1.4 Test temperature and range;
- 15.1.5 Relative humidity on each side of film within the test chamber;

15.1.6 A description of each side of the film specimen—Side A is attached to the source chamber; distinguish as “Side A” and “Side B” when there is no obvious difference, otherwise state the differences (for example, Side A—metallized and Side B—unmetallized);

15.1.7 A table that includes the time values, source chamber gas values, and the collection chamber gas values for each replicate apparatus and include a description of the gas values (for example, concentration, peak area, normalized ratio, and so forth) and information about changes in system mass with time (see 14.3);

15.1.8 The MTC for each gas in each apparatus (that is, each replicate) and the MTC of each specimen should be reported in cm/hour;

15.1.9 The average and standard deviation of the MTC for each gas tested (usually three or more replications) and the average and standard deviation are taken as representative for the chemical, film, relative humidity, and temperature combination;

15.1.10 Include a graphical representation of the concentration measurements and the calculated (that is, modeled) concentrations using the estimated MTC and other model parameters (that is, MTC,  $C_o$ ,  $\alpha$ ,  $k_p$ ); and

15.1.11 If a parameter-fitting routine was used to estimate model parameters, report the parameter values and statistics (that is, confidence limits, standard error, t-stat, p-value, and so forth) and provide any additional information related to obtaining and using model parameters.

## 16. Precision and Bias

16.1 The precision of this test method was determined from an interlaboratory study #661, “Test Method for Film Permeability Determination Using Static Permeability Cells,” conducted in 2011.<sup>10</sup>

16.2 The precision and bias values for the interlaboratory study (ILS) were obtained using NIST DATAPLOT software, routine E691 (<http://www.itl.nist.gov/div898/software/dataplot/>).

16.3 Each of seven laboratories tested the permeability of four different plastic films to four different chemicals (five including isomers). Each “test result” was calculated using three individual replicates. For Films 1 and 3, the precision statement was determined through statistical examination of 140 ( $7 \times 4 \times 5$ ) results from seven laboratories on four materials and five chemicals. For Films 2 and 4, the precision statement was determined through statistical examination of 120 ( $6 \times 4 \times 5$ ) results from six laboratories on four materials and five chemicals. For these films, one laboratory only reported two MTCs and the DATAPLOT software used to calculate the precision statistics required a balanced dataset with three replicates, so the results from the two-replicate laboratory was not included for these film materials.

16.4 A summary of the results from the interlaboratory test are presented in the following Tables 4-7.

16.4.1 Definitions used in Tables 4-7 are:

16.4.1.1  $r$ —Repeatability within each laboratory.

16.4.1.2  $R$ —Reproducibility among different laboratories.

16.4.1.3  $s_r$ —Repeatability standard deviation and is a measure of the variability that can be expected within a laboratory under repeatability conditions.

16.4.1.4  $s_R$ —Reproducibility standard deviation and is a measure of between-laboratory variability.

16.4.1.5  $MTC_{ILS}$ —Average of the MTC for a film and fumigant combination for all replicates and laboratories as described in 16.3.

16.4.1.6  $s_{MTC,ILS}$ —Standard deviation of the MTC for a film and fumigant combination for all replicates and laboratories as described in 16.3.

16.4.1.7  $h$ -statistic—Consistency statistic computed for repeatability conditions. The  $h$ -statistic provides a measure of each laboratory’s within-laboratory variability compared with the within-laboratory variability of all the other laboratories combined. By comparing the  $h$ -statistics with a critical value, the percentage of measurements judged equivalent was obtained for each film and chemical combination.

16.4.1.8  $k$ -statistic—Consistency statistic computed for reproducibility conditions. The  $k$ -statistic provides a measure of each laboratory’s testing average compared with the average of the other laboratories combined. By comparing the  $k$ -statistics to a critical value, the percentage of measurements judged equivalent was obtained for each film and chemical combination.

<sup>10</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E35-1009. Contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org).

**TABLE 5 Precision Statistics for MTC (MTC, cm/h), Film 2, and Five Fumigants**

Material	$MTC_{ILS}$	$s_{MTC,ILS}$	$s_r$	$s_R$	$r$	$R$
Methyl bromide	0.0323	0.0125	0.0073	0.0139	0.02	0.04
Methyl iodide	0.0478	0.0225	0.0139	0.0252	0.04	0.07
<i>cis</i> -1,3-Dichloropropene	0.1828	0.0987	0.0650	0.1120	0.18	0.31
<i>trans</i> -1,3-Dichloropropene	0.2835	0.1807	0.1052	0.2001	0.29	0.56
Chloropicrin	0.0489	0.0238	0.0139	0.0264	0.04	0.07

**TABLE 6 Precision Statistics for MTC (MTC, cm/h), Film 3, and Five Fumigants**

Material	$MTC_{ILS}$	$s_{MTC,ILS}$	$s_r$	$s_R$	$r$	$R$
Methyl bromide	0.0103	0.0065	0.0069	0.0086	0.019	0.024
Methyl iodide	0.0059	0.0045	0.0027	0.0050	0.008	0.014
<i>cis</i> -1,3-Dichloropropene	0.0115	0.0129	0.0059	0.0138	0.017	0.039
<i>trans</i> -1,3-Dichloropropene	0.0211	0.0201	0.0125	0.0225	0.035	0.063
Chloropicrin	0.0029	0.0050	0.0011	0.0051	0.003	0.014

**TABLE 7 Precision Statistics for MTC (MTC, cm/h), Film 4, and Five Fumigants**

Material	$MTC_{ILS}$	$s_{MTC,ILS}$	$s_r$	$s_R$	$r$	$R$
Methyl bromide	0.0062	0.0035	0.0017	0.0037	0.005	0.010
Methyl iodide	0.0045	0.0047	0.0012	0.0048	0.003	0.014
<i>cis</i> -1,3-Dichloropropene	0.0089	0.0113	0.0015	0.0113	0.004	0.032
<i>trans</i> -1,3-Dichloropropene	0.0146	0.0145	0.0028	0.0147	0.008	0.041
Chloropicrin	0.0036	0.0062	0.0018	0.0063	0.005	0.018

16.5 **Table 8** shows the percentage of MTC values that were judged to be the same (that is, below the critical test statistic) for repeatability (*h*-statistic) and reproducibility (*k*-statistic) conditions. The results indicate that repeatability was >94 % and reproducibility was >90 %; therefore, a high fraction of the MTC values were in the acceptable range even though the tested films had MTCs that varied over four orders of magnitude and several participants had no experience conducting this test method.

16.6 **Bias**—This test method has no statement of bias since the MTC is defined in terms of this test method.

## 17. Keywords

17.1 fumigants; mass transfer coefficient; MTC; plastic film permeability

**TABLE 8 Percentage of MTC Values (cm/h) That Were Judged to be the Same (that is, below the Critical Test Statistic) for Repeatability (*h*-statistic) and Reproducibility (*k*-statistic)**

	Repeatability ( <i>h</i> -statistic)	Reproducibility ( <i>k</i> -statistic)
Film 1	94 %	94 %
Film 2	97 %	90 %
Film 3	94 %	89 %
Film 4	97 %	93 %

## APPENDIX

### (Nonmandatory Information)

#### X1. EQUATIONS USED TO OBTAIN MTC

##### X1.1 Mathematical Symbols Used

X1.1.1  $C_s$  = Concentration in source chamber air space, g cm<sup>-3</sup>;

X1.1.2  $C_o$  = Concentration in source chamber air space initially, g cm<sup>-3</sup>;

X1.1.3  $C_r$  = Concentration in collection chamber air space, g cm<sup>-3</sup>;

X1.1.4  $S_s$  = Concentration in source chamber adsorbed to film membrane, g cm<sup>-2</sup>;

X1.1.5  $S_r$  = Concentration in collection chamber adsorbed to film membrane, g cm<sup>-2</sup>;

X1.1.6  $h$  = MTC, cm h<sup>-1</sup>;

X1.1.7  $J$  = Flux density of gas crossing film membrane, g cm<sup>-2</sup> h<sup>-1</sup>;

X1.1.8  $L_s$  = Length of the source chamber, cm;

X1.1.9  $L_r$  = Length of the collection chamber, cm;

X1.1.10  $Q$  = Source or sink term, g cm<sup>-3</sup> h<sup>-1</sup>;

X1.1.11  $k_p$  = Equilibrium sorption parameter, cm; and

X1.1.12  $\alpha$  = Kinetic sorption parameter, t<sup>-1</sup>.

##### X1.2 Diffusion Model

X1.2.1 *System Equations*—Vapor diffusion between two chambers separated by a permeable membrane can be described using two coupled differential equations and an equation describing transfer across the film interface:

$$\frac{\partial C_s}{\partial t} + \frac{1}{L_s} \frac{\partial S_s}{\partial t} = Q_s(x, t) \quad (\text{X1.1})$$

$$\frac{\partial C_r}{\partial t} + \frac{1}{L_r} \frac{dS_r}{dt} = Q_r(x, t) \quad (\text{X1.2})$$

$$J = h(C_s - C_r) = Q_s L_s = Q_r L_r \quad (\text{X1.3})$$

X1.2.1.1 The first terms of [Eq X1.1](#) and [X1.2](#) describe the change in concentration within the chamber in time, the second terms describe adsorption of the chemical to the film membrane, and the third terms describe chemical leaving the source chamber or entering the collection chamber. [Eq X1.3](#) describes the transfer of chemical between chambers and allows estimation of the MTC,  $h$ .

X1.2.2 *No Gas Sorption to Film Membrane*—The equations describing the gas concentration in the source,  $C_s$ , and collection,  $C_r$ , chambers as a function of time are:

$$\frac{C_s(t)}{C_o} = \frac{\left( e^{\frac{h(L_r + L_s)t}{L_r L_s}} L_r + L_s \right)}{L_r + L_s} \quad (\text{X1.4})$$

$$\frac{C_r(t)}{C_o} = \frac{\left( 1 - e^{\frac{h(L_r + L_s)t}{L_r L_s}} \right) L_s}{L_r + L_s} \quad (\text{X1.5})$$

X1.2.2.1 *No gas sorption to film membrane and equal chamber lengths*—This provides the simplest equations for describing the fumigant concentration in each chamber but requires that the source length,  $L_s$ , and the collection chamber length,  $L_r$ , are the same (that is,  $L$ ).

$$\frac{C_s(t)}{C_o} = \frac{1}{2} \left( 1 + e^{-\frac{2ht}{L}} \right) \quad (\text{X1.6})$$

$$\frac{C_r(t)}{C_o} = \frac{1}{2} \left( 1 - e^{-\frac{2ht}{L}} \right) \quad (\text{X1.7})$$

X1.2.2.2 Explicit equations for MTC,  $h$ , where  $C_s/C_o$  is a fraction.

$$ht = -\frac{L}{2} \ln \left[ \frac{2C_s(t)}{C_o} - 1 \right] \quad (\text{X1.8})$$

$$ht = -\frac{L}{2} \ln \left[ 1 - \frac{2C_r(t)}{C_o} \right] \quad (\text{X1.9})$$

X1.2.3 *Chamber Concentration with Gas Sorption to Film Membrane*—Gas sorption is described mathematically as:

$$\frac{dS_x}{dt} = \alpha(k_p C_x - S_x) \quad (\text{X1.10})$$

Where subscript  $x$  = either the source (for example, “ $s$ ”) or collection (for example, “ $r$ ”) chambers.

X1.2.3.1 Using [Eq X1.4](#), the equations describing the fumigant concentration in each chamber as a function of time are:

$$\frac{C_s}{C_o} = \frac{1}{2} \left( \frac{k_d e^{\frac{\alpha(k_d + L)t}{L}} + L}{k_d + L} - \frac{e^{\frac{(\beta - \gamma)t}{2L}} (\gamma - 2\alpha L - \beta)}{2\beta} \right. \\ \left. + \frac{e^{\frac{-(\beta + \gamma)t}{2L}} (\gamma - 2\alpha L + \beta)}{2\beta} \right) \quad (\text{X1.11})$$

$$\frac{C_r}{C_o} = \frac{1}{2} \left( \frac{k_d e^{\frac{\alpha(k_d + L)t}{L}} + L}{k_d + L} + \frac{e^{\frac{(\beta - \gamma)t}{2L}} (\gamma - 2\alpha L - \beta)}{2\beta} \right. \\ \left. - \frac{e^{\frac{-(\beta + \gamma)t}{2L}} (\gamma - 2\alpha L + \beta)}{2\beta} \right) \quad (\text{X1.12})$$

Where:

$$\gamma = 2h + \alpha(k_d + L) \\ \beta = \sqrt{\gamma^2 - 8\alpha h L} \quad (\text{X1.13})$$

NOTE X1.1—To use these equations to determine the concentration in the source and collection chambers, the chamber lengths (for example,  $L = L_s = L_r$ ) shall be the same. Also, the solution can be naturally written in terms of a ratio,  $C/C_o$ .

X1.2.4 *Modeling Leakage of Gas from Apparatus*—When sorption of fumigant vapor to plastic is negligible, but the apparatus slowly leaks gas, the leakage process can be modeled as a first-order process, that is:

$$\frac{\partial C_x}{\partial t} = -\mu_x C_x \quad (\text{X1.14})$$

Where subscript  $x$  = the source or collection chamber.

X1.2.4.1 *Leakage rate different in chambers*—Equations describing the fumigant concentration inside the apparatus as a function of time in the absence of gas sorption to the film membrane can be written as:

$$\frac{C_s(t)}{C_o} = \frac{\left(e^{-\frac{(\alpha - \beta)t}{2L_r L_s}} (\alpha + \beta - \gamma) - e^{-\frac{(\alpha + \beta)t}{2L_r L_s}} (\alpha - \beta - \gamma)\right)}{2\beta} \quad (\text{X1.15})$$

$$\frac{C_r(t)}{C_o} = \frac{e^{-\frac{(\alpha + \beta)t}{2L_r L_s}} \left(e^{\frac{\beta t}{L_r L_s}} - 1\right) h L_s}{\beta} \quad (\text{X1.16})$$

Where  $\alpha$ ,  $\gamma$ , and  $\beta$  are defined as:

$$\alpha = h(L_r + L_s) + L_r L_s (\mu_r + \mu_s)$$

$$\gamma = 2L_r(h + L_s \mu_s)$$

$$\beta = \sqrt{h^2(L_r + L_s)^2 - 2hL_r L_s(L_r - L_s)(\mu_r - \mu_s) + L_r^2 L_s^2(\mu_r - \mu_s)^2} \quad (\text{X1.17})$$

Where  $\mu_s$  and  $\mu_r$ , respectively = first-order leakage rates for the source and collection chambers.

NOTE X1.2—Using Eq X1.12–X1.14 to obtain a value for the MTC,  $h$ , requires an iterative or nonlinear least squares methodology, since  $h$

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occurs in the exponent and the parameters  $\alpha$ ,  $\gamma$ , and  $\beta$ . A generalized program, FilmPC, has been developed and enables evaluation of the MTC,  $h$ , for a variety of conditions. FilmPC uses a nonlinear least-squares algorithm to fit the selected model parameters simultaneously (for example,  $h$ ,  $C_o$ ,  $\alpha$ ,  $k_d$ , and  $\mu$ ).

X1.2.4.2 *Leakage rates same in both chambers*—Equations describing the fumigant concentration inside the apparatus as a function of time in the absence of gas sorption to the film membrane can be written as:

$$\frac{C_s(t)}{C_o} = \frac{e^{-\mu t} \left(e^{\frac{h(L_r + L_s)t}{L_r L_s}} L_r + L_s\right)}{L_r + L_s} \quad (\text{X1.18})$$

$$\frac{C_r(t)}{C_o} = \frac{e^{-\mu t} \left(1 - e^{\frac{h(L_r + L_s)t}{L_r L_s}}\right) L_s}{L_r + L_s} \quad (\text{X1.19})$$

NOTE X1.3—Since  $h$  occurs only in the exponent in Eq X1.15 and X1.16, explicit equations for  $h$  can be written. Assuming that  $L = L_s = L_r$  for simplicity and  $C_x/C_o$  are fractions, a value for the MTC,  $h$ , can be obtained using a linear regression model,  $y = m t$ , with  $m = h + L\mu/2$ . See 14.5.1.2 for an example (that is, when  $\mu = 0$ ).

$$\left(h + \frac{L\mu}{2}\right)t = -\frac{L}{2} \ln \left[ \frac{2C_s(t)}{C_o} - e^{-\mu t} \right] \quad (\text{X1.20})$$

$$\left(h + \frac{L\mu}{2}\right)t = -\frac{L}{2} \ln \left[ e^{-\mu t} - \frac{2C_r(t)}{C_o} \right] \quad (\text{X1.21})$$