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AN AUTOMATED DYNAMIC WATER VAPOR PERMEATION TEST METHOD

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13. ABSTRACT (Maximum 200 words) This report describes an automated apparatus developed to measure the transport of water vapor through materials under a variety of conditions. The apparatus is more convenient to use than the traditional test methods for textiles and clothing materials, and allows one to use a wider variety of test conditions to investigate the concentration-dependent and nonlinear transport behavior of many of the semipermeable membrane laminates which are now available. The dynamic moisture permeation cell (DMPC) has been automated to permit multiple setpoint testing under computer control, and to facilitate investigation of transient phenomena. Results generated with the DMPC are in agreement with and of comparable accuracy to those from the ISO 11092 (sweating guarded hot plate) method of measuring water vapor permeability.				
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PREFACE

This report describes an automated dynamic water vapor permeation test method, called the Dynamic Moisture Permeation Cell (DMPC), which is useful for characterizing the transport behavior of fabrics, fabric systems, and semipermeable membrane laminates. The original concept of the test originated with Cy Kendrick and Don Rivin of the Special Protection Division, Survivability Directorate, U.S. Army Soldier Systems Command (SSCOM) (PROV), Natick Research and Development and Engineering Center (NRDEC). Linda Sicuranza, a Dedham High School teacher (Dedham, Massachusetts) working under a summer Army Research Office program, performed tests with the original version of the apparatus and demonstrated its usefulness for membrane laminates. Phil Gibson, of the Textile Research and Engineering Division at NRDEC, recognized that the test setup was similar to a test developed by John Wehner, Ludwig Rebenfeld, and Bernard Miller of Princeton University/Textile Research Institute. Using their test method as a guide, he redesigned the flow cell, and added instrumentation to allow automatic operation of the test. The DMPC and sweating guarded hot plate testing and analysis described in the report were performed by Phil Gibson.

This work is associated with Gibson's doctoral dissertation research at the University of Massachusetts Lowell, although in that work the test apparatus is used for transient testing rather than for steady-state measurements. Dr. Majid Charmchi, Phil Gibson's advisor in the Department of Mechanical Engineering, has provided useful suggestions and ideas on the design of the system and the analysis of the results. Quoc Truong of the Special Protection Division at NRDEC provided several of the semipermeable membrane laminates used to demonstrate the use of the DMPC. Elizabeth Klemperer of the Special Protection Division reviewed the draft report and pointed out areas needing further clarification, which were included in the final version.

An Automated Dynamic Water Vapor Permeation Test Method

1. Introduction

This report describes an automated apparatus developed to measure the transport of water vapor through materials under a variety of conditions. This apparatus is more convenient to use than the traditional test steady-state methods used for textile and clothing materials. It also allows one to use a wide variety of test conditions to investigate concentration-dependent and nonlinear transport in air permeable and impermeable fabrics.

Two test methods are used routinely at the U.S. Army Soldier Systems Command (SSCOM) Natick Research, Development, and Engineering Center (NRDEC) to determine the water vapor permeation properties of clothing materials. These two methods are ASTM Method E96 [1], and ISO Method 11092 [2]. Both methods are time-consuming, require large amounts of material, and are not capable of a very wide range of test conditions. These are also pseudoequilibrium test methods, and do not give any information on any of the dynamic properties of the test materials which may be important under transient conditions. There was a need at NRDEC for a convenient water vapor diffusion test which allows one to test small quantities of membranes or membrane laminates, and also to conduct research into the behavior of these materials under nonstandard conditions.

There are a wide variety of test methods developed over the years to characterize the diffusion and permeation properties of polymer films, laminates, textiles, and paper products [3-10]. The method we developed, which we call the Dynamic Moisture Permeation Cell (DMPC), is similar to a method used by Wehner, et.al. [11], to measure the dynamics of water vapor transmission through hygroscopic and nonhygroscopic fabrics.

In this report we document the details of the test apparatus and describe the instrumentation and automation of the test procedure. We also present typical test results for representative types of fabrics and semipermeable membrane laminates and give a correlation between the DMPC and the sweating guarded hot plate test method (ISO 11092). In this report we focus on the use of the DMPC to obtain steady-state results. In a later report, the use of the DMPC to obtain dynamic data for transient situations will be described. The dynamic data is used in conjunction with the numerical solution of equations developed to describe the coupled transport of energy and mass through hygroscopic and nonhygroscopic porous materials [12].

2. Test Methods

The DMPC test arrangement is shown below in Figure 1.

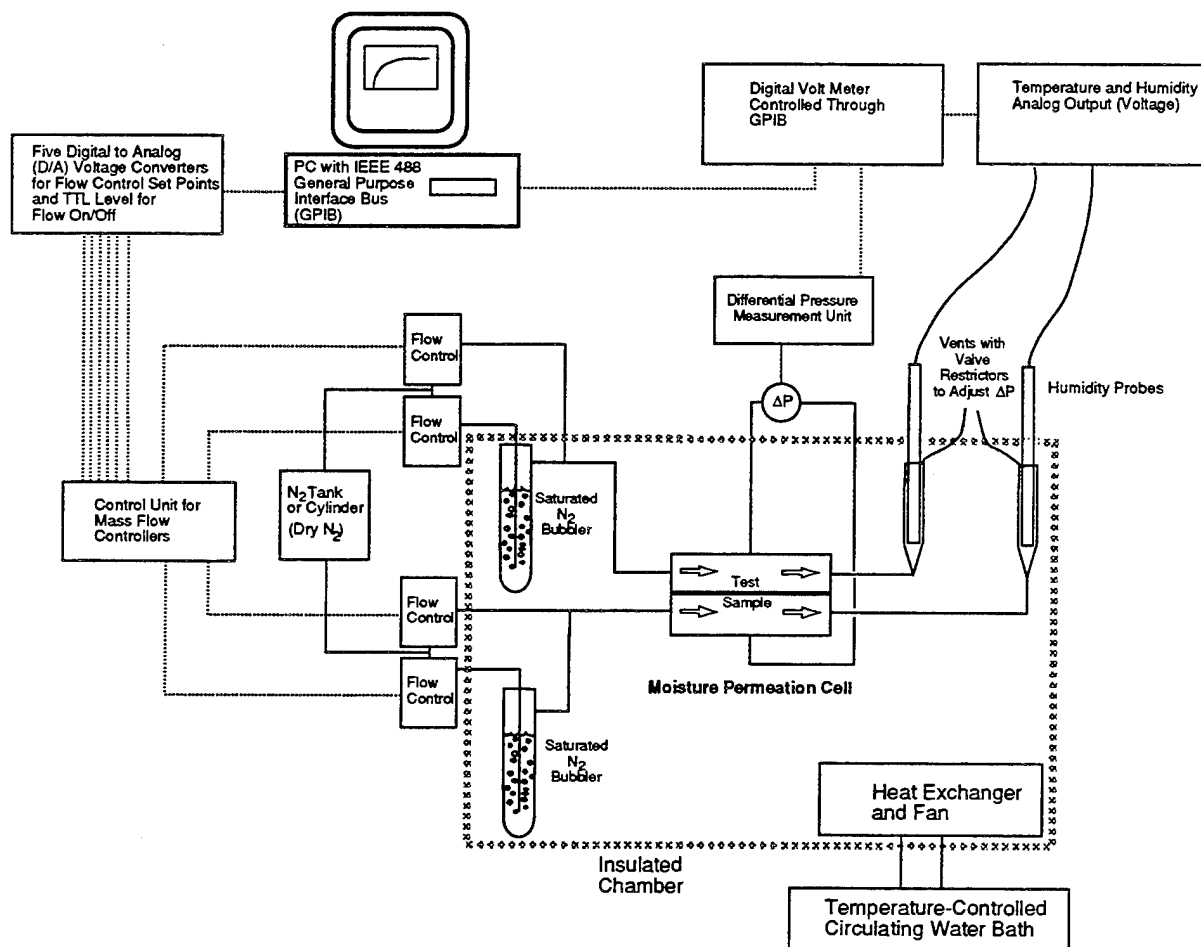


Figure 1. Dynamic Moisture Permeation Cell (DMPC) Arrangement.

Nitrogen streams consisting of a mixture of dry nitrogen and water-saturated nitrogen are passed over the top and bottom surfaces of the sample. The relative humidity of these streams is varied by controlling the proportion of the saturated and the dry components. By knowing the temperature and water vapor concentration of the entering nitrogen flows, and by measuring the temperature and water vapor concentration of the nitrogen flows leaving the cell, one may measure the flux of water vapor diffusing through the test sample.

The following equations for calculating water vapor flux apply to either the top or bottom flows in the cell. Strictly speaking, only one measurement on one side of the cell is necessary; the use of two separate humidity transducer for the top and bottom flows allows two measurements of water vapor flux to be made at the same time, using the equations given below for either the top or bottom flow, as appropriate.

For this type of test, the mass flow rate of water vapor diffusing through the test sample from one side of the cell to the other is given by:

$$\frac{\dot{m}}{A} = \frac{Q(\delta C)}{A} = \frac{Q(C_2 - C_1)}{A} \quad (1)$$

where:

$\dot{m}$ = mass flux of water vapor across the sample (kg/s)

A = area of test sample (m²)

Q = volumetric flow rate through top or bottom portion of the cell (m³/s)

δC = water vapor concentration difference between incoming stream (C_1) and outgoing stream (C_2) in top or bottom portion of the moisture permeation cell (kg/m³)

The incoming water vapor concentration is determined by the relative mass flow of the saturated and the dry nitrogen streams, which can be monitored with a flow meter both at the outlets from the controllers, and from the cell.

The mass flow rates are controlled by MKS model 1259C mass flow controllers, with a Model 247C 4-Channel Readout (MKS Instruments, Inc.). At constant mass flow, the true volumetric flow rate will vary with temperature; the flow rate set by the MKS controllers is indicated in terms of volumetric flow rates at standard conditions of 0°C and atmospheric pressure (1.01325 x 10⁵ Pa). The actual volumetric flow rate at different temperatures may be found from the mass flow rate, the temperature, and the pressure of the actual flow.

The mass flow rate may be found from the volumetric flow rate given by the mass flow controllers:

$$\dot{m} = \rho_{N_2} Q_s \quad (2)$$

Q_s = volumetric flow rate at standard conditions of 0°C

ρ_{N_2} = density of dry nitrogen at 0°C = 1.24 kg / m³

The actual volumetric flow rate at a given temperature and pressure may be found from the indicated volumetric flow rates at standard conditions by:

$$Q = Q_s \left(\frac{T}{T_s} \right) \left(\frac{p_s}{p} \right) \quad (3)$$

Q = true volumetric flow rate (m³ / s) at test temperature T

$\left(\frac{T}{T_s} \right)$ = ratio of actual temperature (T) to temperature at standard conditions (T_s)

$\left(\frac{p_s}{p} \right)$ = ratio of reference atmospheric pressure (p_s) to actual pressure (p)

The critical measurement is the outgoing flow water vapor concentration C_2 , which we may measure in a variety of ways. At present, capacitance-type relative humidity probes (Vaisala HMI 32) with Type HMP 35 sensors are used (Vaisala Inc.). To obtain the water vapor concentration in the outgoing air stream, one must be able to convert from the known values of relative humidity and temperature to water vapor concentration. We may use an empirical formula (or tables) for the vapor pressure of saturated water vapor in air as a function of temperature, and then convert vapor pressure to concentration.

Saturation vapor pressure as a function of temperature is correlated with table values [13] by the equation:

$$p_{sat} = 614.3 \exp \left[17.06 \left(\frac{T - 273.15}{T - 40.25} \right) \right] \quad (4)$$

p_{sat} = saturation vapor pressure (Pa) at temperature T
 T = temperature (K)

To obtain the actual concentration of water vapor in the water vapor / air mixture, we may use the perfect gas law:

$$C_{sat} = \frac{p_{sat} M_w}{RT} ; \quad (5)$$

C_{sat} = saturated concentration of water vapor in air (kg/m³)

p_{sat} = saturation vapor pressure of water vapor in air (Pa)

M_w = molecular weight of water (18.015 kg/kmole)

R = universal gas constant (8314.5 N-m/kmole-°K)

Since relative humidity ϕ is defined as: $\phi = \frac{p}{p_{sat}} = \frac{C}{C_{sat}}$,

the equation for the measured water vapor transmission rate may be written as:

$$\frac{\dot{m}}{A} = \frac{\delta\phi Q}{A} \left[\frac{p_{sat} M_w}{RT} \right] \quad (6)$$

$\delta\phi = \phi_2 - \phi_1$

ϕ_1 = relative humidity of incoming flow

ϕ_2 = relative humidity of outgoing flow

If we substitute equation (3) into the expression for the water vapor transmission rate (and if we neglect pressure differences), we note that the actual test temperature divides out, and we may express the water vapor transmission rate in terms of the volumetric flow rate, the humidity difference, and the temperature:

$$\frac{\dot{m}}{A} = \frac{\delta\phi Q_s p_{sat} M_w}{A R T_s} ; \text{ here } T_s \text{ is the reference temperature in degrees K (273.15 K).} \quad (7)$$

For the present test apparatus, various sample holders are available, which have different test sample measurement areas, and which have different downstream locations from the flow inlet. All test results given in this report used a sample measurement area of $1.0 \times 10^{-3} \text{ m}^2$, and the sample was located equidistant from the inlet and outlet ports of the cell. The typical volumetric flow rate used was $3.333 \times 10^{-5} \text{ m}^3/\text{s}$ ($2000 \text{ cm}^3/\text{min}$). Most of the tests were conducted with the flow parallel on the two sides of the test sample; for comparison, some countercurrent flow testing was also conducted and is described later. Details of the moisture permeation cell are shown in Figure 2.

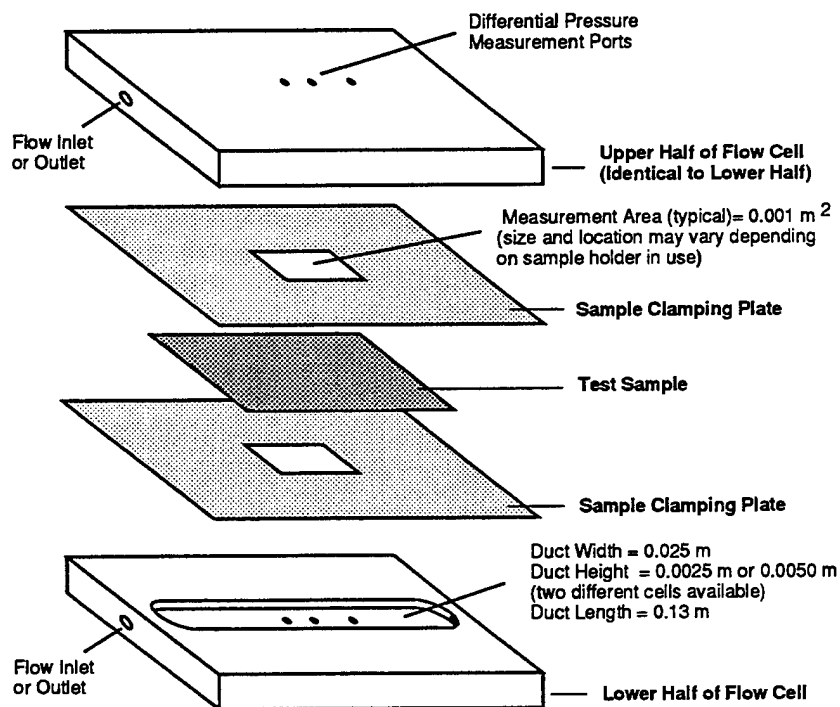


Figure 2. Schematic and Dimensions of the Dynamic Moisture Permeation Cell.

The dimensions of the DMPC were optimized allow for flow velocities of at least 0.5 m/sec over the sample to minimize the contribution of boundary air layer resistances to the test measurements. A simple flow analysis of the velocity profile in the cell is given in the Appendix.

The sample size was kept quite small to make it possible to evaluate novel membranes and laminates, which are often produced in quantities too small for testing by some of the standard water vapor diffusion test methods. Sample mounting methods vary according to the material being tested. Most thin materials, such a laminated materials and woven cloth, were originally tested with rubber sealing gaskets to prevent leakage, but the sealing proved to be unnecessary for most materials; the clamping force provided by the mounting bolts (not shown in Figure 2) has proven to be sufficient to prevent any leakage. Thick materials which are highly permeable require special sealing methods such as edge sealing by molten wax, or the use of a curable sealant.

Test Procedure

The actual test is conducted under the control of a personal computer (PC) connected to the flow controllers and the relative humidity instruments through a General Purpose Interface Bus (GPIB) controller (see Figure 1). The operator inputs up to 20 desired humidity setpoints for the upper and lower nitrogen streams. The computer applies the proper setpoint voltage to each controller to produce the desired relative humidity in the upper and lower streams entering the moisture permeation cell. The analog voltage output of the relative humidity measurement instrument is read by the digital voltmeter and sent to the PC through the GPIB, and displayed on the screen. The computer plots the relative humidity, records the data to disk, and applies operator-determined equilibration criteria to determine when equilibration has been reached for that setpoint. Once equilibration is reached, the results (humidity, calculated flux, etc.) are output on a printer and to a data file on disk. The computer then proceeds to the next setpoint and repeats the process.

Calibration and Setup

Three calibration procedures must be observed before a series of tests begins. The flow meters must be calibrated either by an independent flow meter, or they may be calibrated directly by a special procedure of balancing using the humidity meters and switching of gas inputs to check for equality of flows. The zero reading and a full scale range check have proved sufficient so far. The particular flow controllers in use are quite stable from day to day if left on and warmed up.

After the calibration of the flow meter, the offset of the digital-to-analog (D/A) converters used to apply a setpoint voltage to the flow controllers must be checked. This is done by applying the nominal full scale voltage for each D/A converter to the controller, and checking the setpoint. The actual setpoint is then used by the software in the data acquisition and control program on the PC to determine a scale factor for each D/A converter.

The third calibration procedure is for the relative humidity instrument. A calibration curve for the relative humidity instrument may be determined *in situ* by placing an impermeable aluminum foil sample in the cell and varying the relative humidity of the gas flow in the top and bottom of the cell by means of the flow controllers. The resulting curves of measured relative humidity versus true relative humidity (set by the flow controllers) are used as calibration factors to correct the measured relative humidity for subsequent tests.

The pressure drop across the sample is monitored by means of an MKS Baratron Type 398 differential pressure transducer, with a Type 270B signal conditioner (MKS Instruments, Inc.). For measurement of pure diffusion, especially for materials such as fabrics, which may be quite permeable to convective flows, it is important to make sure that the pressure drop across the sample is zero, so that transport takes place only by pure diffusion. The pressure drop is continuously monitored and displayed, and is controlled by means of a valve restrictor on the outlet of one of the gas streams. For the permeable fabrics, this system also allows one to do testing under controlled conditions of a defined pressure drop across the sample, so that transport takes place by both diffusion and convection. This makes it possible to determine an air permeability value from the apparatus, in addition to the water vapor diffusion properties of the test sample.

It is useful to have a calibration or reference material to check the operation of the system. A microporous polytetrafluorethylene (PTFE) membrane has proven to be very useful for this purpose. A single layer of this membrane has a very small resistance to water vapor diffusion, but has very high resistance to convective flow, because of the very small pore sizes. The membrane may be layered to produce a material with a lower effective diffusivity. Since diffusion takes place only through the pore spaces of the membrane, the material has a very linear and reproducible plot of flux versus concentration difference. The plot of flux versus concentration difference may be used as a calibration curve for the apparatus, and may also be used to determine the boundary air layer resistance present in the test cell.

A typical automated test series consists of a series of set points. For most of the materials discussed later, 9 setpoints were used. The humidity test sequence was: Top @ 100%, Bottom @ 0, 20, 40, 60%; Top @ 80%, Bottom @ 0, 20, 40%; Top @ 60%, Bottom @ 0, 20%. The test temperature is usually 20°C.

Materials which have a constant mass transfer coefficient show a linear constant slope on plots of flux versus concentration difference across the sample. These kinds of materials do not change their transport properties as a function of water content or test conditions.

For materials which do not have a constant linear slope, the data points for a test series will not superimpose, but will form a set of curves for each test condition. We may still calculate a diffusion resistance for these materials, but now we have to evaluate the flux versus concentration difference curve at various points to derive our values for the material diffusion resistance, which will now be a function of the concentration of water in the material.

Our definitions for the various mass transfer coefficients and diffusion resistances are:

$$\frac{\dot{m}}{A} = h_m(\Delta\bar{C}) = \frac{\Delta\bar{C}}{(R_f + R_{bl})} \quad (8)$$

$$R_f = \left[\frac{\Delta\bar{C}}{\left(\frac{\dot{m}}{A} \right)} \right] - R_{bl} \quad (9)$$

$\dot{m}$ = mass flux of water vapor across the sample (kg/s)

A = area of test sample (m²)

$h_m = [1 / (R_f + R_{bl})]$ = mass transfer coefficient (m/s)

$\Delta\bar{C}$ = log mean concentration difference between top and bottom nitrogen streams (kg/m³)

R_f = intrinsic diffusion resistance of sample (s/m)

R_{bl} = diffusion resistance of boundary air layers (s/m)

The log mean concentration difference across the sample is appropriate since there is a significant change in the concentration of the gas stream both below and above the sample. In addition, the gas streams may not necessarily be in parallel flow, but may be run in counter flow to maintain a more constant concentration gradient across the sample. The log mean concentration difference [14] is defined as:

$$\Delta \bar{C} = \frac{\Delta C_a - \Delta C_b}{\ln(\Delta C_a / \Delta C_b)} \quad (10)$$

ΔC_a = concentration difference between the two gas streams at one end of the flow cell

ΔC_b = concentration difference between the two gas streams at the other end of the flow cell

For parallel flow, the concentration differences are between the top and bottom incoming flow at one end of the cell, and the difference between the top and bottom outgoing flows at the other end of the cell. For countercurrent flow, the concentration differences are between the incoming and outgoing flows at one end of the cell, and the incoming and outgoing flows at the other end of the cell.

It is easiest to first look at the results for the PTFE membranes, shown in Figure 3.

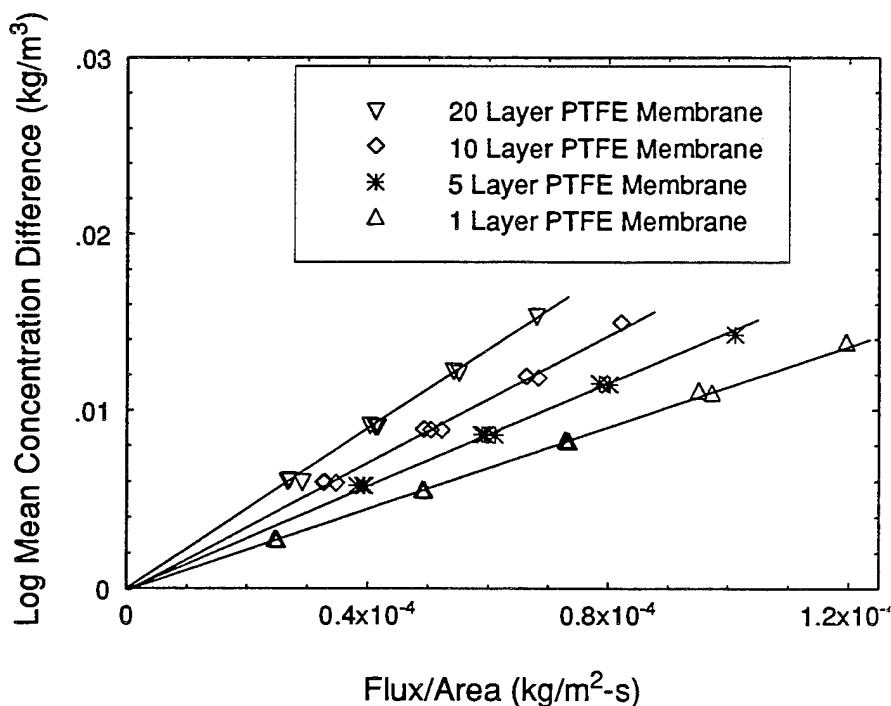


Figure 3. Results for Samples Made of Combined Layers of Microporous PTFE Membranes.

The results for the series of PTFE membranes show that the moisture permeation cell gives the expected type of plots. Since these materials are microporous, and transport takes place only through the interconnected air spaces of the membrane, we expect that the plot of the mass flux versus the concentration difference across the sample will be linear, which is readily apparent from Figure 3. We also note that these linear plots have all the test results from the nine different test conditions superimposed on the same constant slope line, which means that the diffusion resistance of each sample is constant.

We may use this test series of microporous PTFE membranes to derive an estimate of the boundary layer resistance on both the top and bottom of the sample. From our definition of resistance, we know that the resistance of the sample and the boundary air layers is equal to the slope of the line for each sample in Figure 3. We also know that for these types of materials, we can assume that the mass transfer resistance is additive; the resistance of 20 layers is twice the resistance of 10 layers. We may derive a value for the boundary air layer resistance from the relation:

$$R_{bl} = R_{total} - nR_{1-layer} \quad (11)$$

R_{bl} = boundary air layer resistance (s / m)

R_{total} = measured mass transfer resistance of sample (s / m)

n = number of teflon layers

$R_{1-layer}$ = calculated resistance of 1 PTFE layer (s / m)

From the relations given above, we find the boundary air layer resistance (R_{bl}) is approximately equal to 105 s/m, and the resistance of a single layer of the PTFE membrane is in the range of 6-8 s/m.

The boundary air layer resistance is fairly constant at a given set of flow conditions. We note that a single layer of the PTFE membrane will give practically the same value as the boundary air layer resistance, and thus serves as a convenient way to directly measure the boundary layer resistance present within the cell at other flow conditions (if we correct for the resistance of the single PTFE layer), and as a standard reference material to check the results generated by the cell.

3. DMPC Results for Various Clothing Materials

The test results for three different classes of clothing materials will illustrate various factors which must be taken into account when analyzing the results generated by this type of test. In another section of this report, the results obtained by the DMPC are compared with the results obtained by another method (sweating guarded hot plate) to show the correlation between the two types of tests.

The three classes of materials are 1) permeable fabrics, 2) microporous fabric-laminated membranes, 3) hydrophilic nonporous fabric laminated membranes. The sample designations, and information about the materials, are given below in Table 1.

Table 1. Test Fabrics for DMPC

Material Type	Sample Identification	Materials and Reference
Air-Permeable Fabrics	HWBDU Fabric	Hot Weather Battle Dress Uniform (HWBDU) 100% cotton fabric [15]
	BDO Shell Fabric	Battle Dress Overgarment (BDO) Shell Fabric 50% cotton/ 50% nylon fabric [16]
	USMC System (Shell Fabric + Liner)	U.S. Marine Corps chemical protective garment system 100% cotton fabric over nonwoven laminated carbon-loaded polyester knit liner [17],[18],[19]
	BDO System (Shell Fabric + Liner)	Battle Dress Overgarment (BDO) chemical protective garment system 50% cotton/50% nylon fabric over nylon tricot laminated carbon-loaded polyurethane foam liner [16], [20], [21]
Microporous Laminated Membranes	PTFE Membrane (1 layer)	
	Gore Tex III Membrane Laminate	Gore Tex III membrane laminated between Taslan nylon shell fabric and nylon tricot knit liner [22]
	Repel Membrane Laminate	Repel membrane laminated between Nomex shell fabric and knit liner
Hydrophilic Laminated Membranes	Azekura Membrane Laminate	
	Gore Tex II Membrane Laminate	Gore Tex II membrane laminated between Taslan nylon shell fabric and nylon tricot knit liner [22]
	Sympatex Membrane Laminate	Sympatex membrane laminated between Taslan nylon shell fabric and nylon tricot knit liner [22]

We would expect in general that under conditions of pure diffusion (i.e. no pressure drop or convective flow across the sample) the permeable fabrics and the microporous fabric-laminated membranes will behave much like the PTFE membranes in that they will have a linear slope on a plot of flux versus concentration difference across the sample. We would expect that the hydrophilic nonporous fabric laminated membranes would not be linear but would show lower resistances at test conditions which produce a high water content in the membrane, and higher resistances at test conditions where less water is present in the membrane.

Figure 4 shows the flux versus concentration difference plots for the fabrics and the microporous fabric-laminated membranes. We see that these materials all look quite linear and that it is quite easy to derive a single mass transfer resistance value by taking the slope of the plot and subtracting off the boundary air layer resistance.

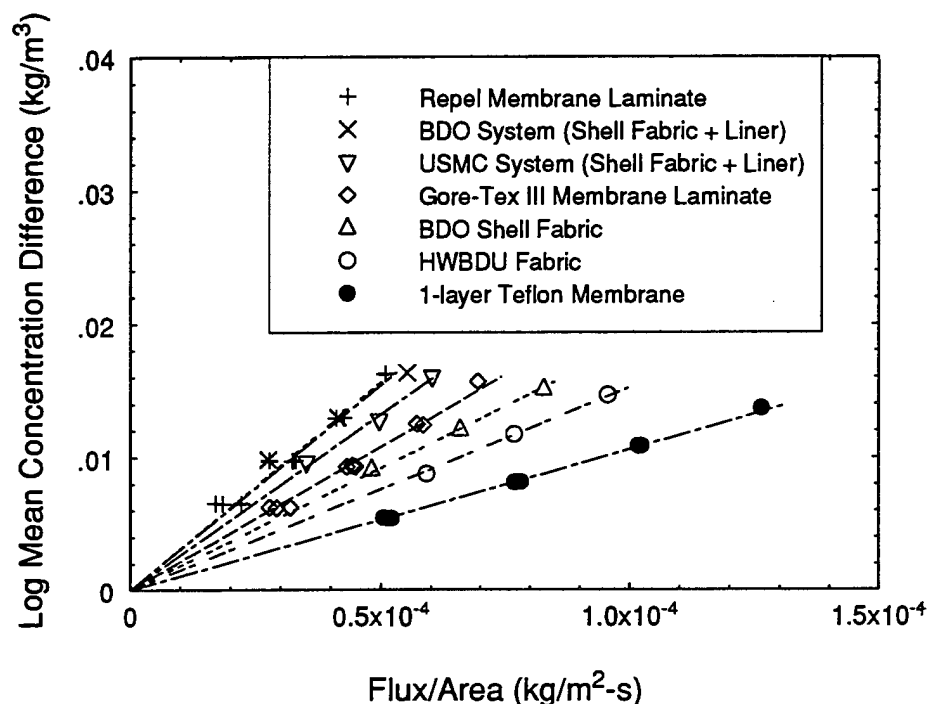


Figure 4. Flux Versus Concentration Difference for Fabrics and Microporous Laminated Membranes.

From the slope of the plots given above, and using equation 8 we may calculate the diffusion resistance of each of these materials. If we subtract the boundary layer resistance (105 s/m) from the total resistance measured in this test, we obtain the intrinsic mass transfer resistance of each sample. The intrinsic resistance for each material is given in Table 2.

Table 2. Intrinsic resistance (s/m) for Fabrics and Microporous Laminated Membranes

Material	Intrinsic Resistance (s/m)
PTFE Membrane (1 layer)	6
HWBDU Fabric	48
BDO Shell Fabric	79
Gore Tex III Membrane Laminate	111
USMC System (Shell Fabric + Liner)	160
BDO System (Shell Fabric + Liner)	205
Repel Membrane Laminate	211

In contrast to the relatively simple behavior of the fabrics and the microporous membrane laminates, the three hydrophilic laminated membranes have a great variation in their measured mass transfer resistance depending on the test conditions used, as shown in Figure 5.

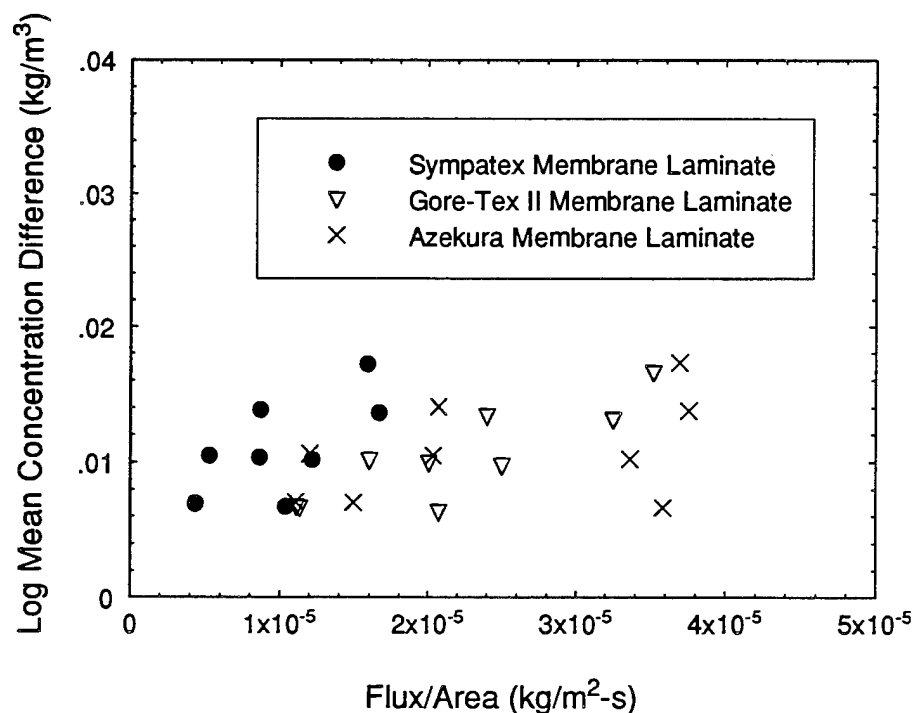


Figure 5. Flux Versus Concentration Difference for Hydrophilic Laminated Membranes.

We can see that the properties of the hydrophilic membranes are much more complicated than the other materials tested. The primary barrier to water vapor diffusion in these materials is the solid polymer layer through which the water must diffuse. The permeability of water in these materials is highly concentration-dependent, and makes the analysis of the test results generated for these materials much more complicated.

These materials also illustrate a practical limitation of the DMPC. The accuracy of the test method depends on the ability of the humidity meters to detect the relative humidity difference between the incoming and outgoing gas flow in each half of the cell. If a material has a very low water vapor flux under a given test condition, then the measurement becomes compromised by the error inherent in the relative humidity meter. By special calibration procedures we have been able to increase the measurement accuracy of the meters to around half a percent of relative humidity, but this accuracy is not sufficient for materials with a very high water vapor resistance. Based on the accuracy of the humidity meters presently in use, we believe that materials which have a resistance higher than approximately 600 to 1000 s/m have exceeded the accuracy of the system. This is not a serious problem for our work, since we are mainly interested in materials which have higher water vapor transport. Higher resistance materials could be tested by increasing the sample measurement area, or by using more accurate instrumentation to measure water vapor concentration in the gas streams.

The results for the hydrophilic membrane laminates shown in Figure 5 are quite confusing, and are more readily interpreted using a different frame of reference.

We will present the results of the testing in terms of an average relative humidity at the membrane. By doing this, we are assuming a very simple definition for the average water concentration in the membrane, defined only as the average of the relative humidities of the two incoming gas streams. This definition neglects the influence of the resistance of the boundary air layer, which will further decrease the concentrations at the surfaces of the hydrophilic materials, and it neglects the variation in vapor concentration along the sample. A log mean average concentration in the sample would be a more appropriate factor to use, but the average relative humidity method will be sufficient to illustrate the general trend of material behavior. A plot of measured intrinsic resistance as a function of average relative humidity is shown below in Figure 6. Here we have omitted most of the data points where the resistance was above 1000 s/m, since we do not believe our present measurement system is accurate at such low concentrations.

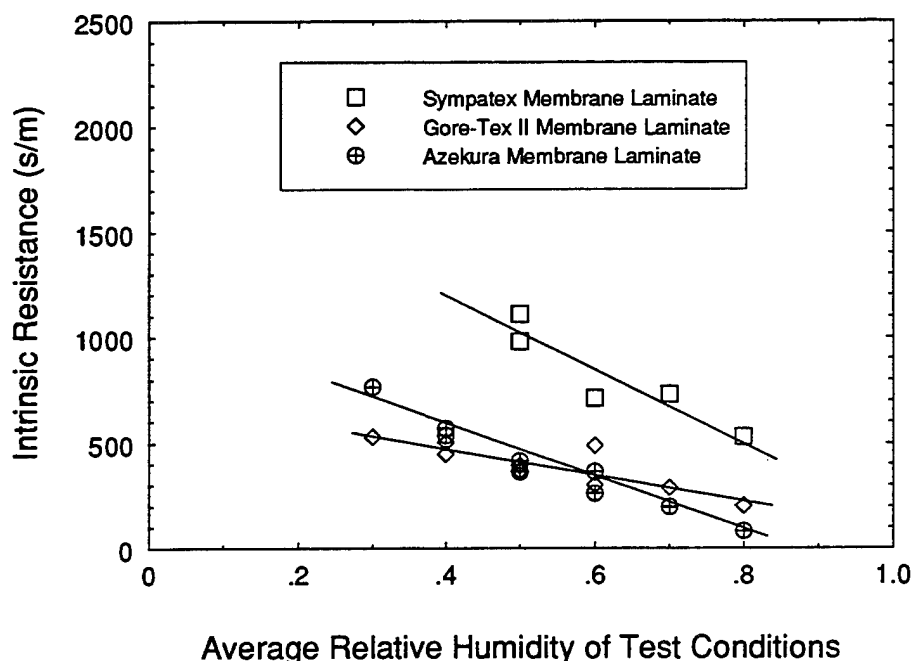


Figure 6. Variation in Diffusion Resistance as a Function of the Mean Relative Humidity on the Two Sides of the Test Sample.

We see that there is a clear trend of decreasing resistance as the average relative humidity (equivalent to average water concentration) in the material increases. As the hydrophilic materials approach saturation, they become comparable to some of the low resistance materials such as the fabrics. There also seem to be clear differences in the degree of the dependence of material properties on the average relative humidity. We believe that presenting data in this way, as a function of average relative humidity, gives a clearer picture of the differences in materials which show up under actual use conditions. From the trend of the data shown in Figure 6, we would also expect all three of these materials to show much lower resistance values when tested under conditions which produce a high water content in the membrane, e.g. ASTM E-96, Procedure BW [1].

4. Comparison of Results Generated by the DMPC with the Sweating Guarded Hot Plate

Results generated by the moisture permeation cell may be compared with results generated by other methods used for measuring the water vapor diffusion properties of materials. One well-accepted standard method is the International Standards Organization Test Method ISO 11092, Measurement of Thermal and Water-Vapour Resistance Under Steady-State Conditions (Sweating Guarded Hot Plate Test) [2]. The test method is briefly described below, further details of the test method and some typical results may be found in Reference 23. A schematic of the sweating guarded hot plate test is given below in Figure 7.

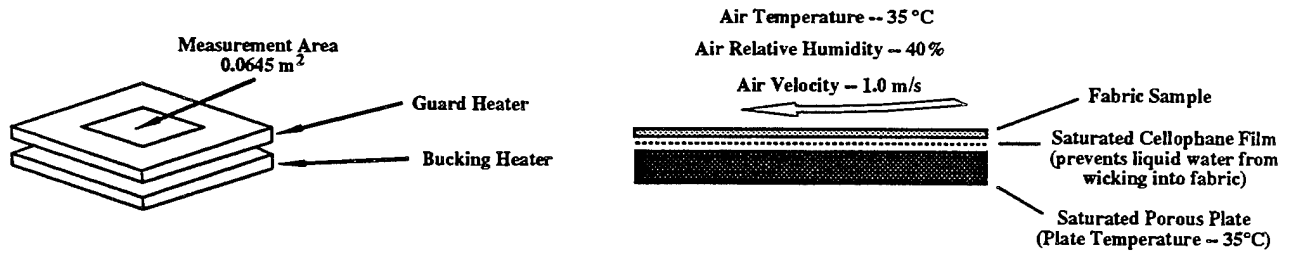


Figure 7. Sweating Guarded Hot Plate Test Method.

The porous guarded hot plate is saturated with water so that its surface is completely wet. A thin saturated cellophane film placed over the plate prevents liquid water from wicking into the fabric, yet allows water to freely permeate through the film and evaporate from the surface. The resistance of the film is so small that it is equivalent to the surface of liquid water, and the vapor pressure is equal to saturation vapor pressure. Since the ISO 11092 test conditions call for no temperature difference between the plate surface and the ambient air, the power required to maintain the plate surface at a given temperature is directly related to the rate at which water evaporates from the surface of the plate and diffuses through the test material.

The equation used for calculating the water vapor resistance is:

$$R_{etotal} = \frac{A(p_s - \phi p_a)}{E} \quad (12)$$

R_{etotal} = water vapor resistance of the material plus the boundary air layer resistance ($\text{m}^2 \cdot \text{Pa} / \text{Watt}$)

A = guarded hot plate measurement area (m^2)

p_s = saturated water vapor pressure at the plate surface (Pa) at the plate temperature

p_a = saturated water vapor pressure of ambient air (Pa) at ambient air temperature

E = power required to maintain a constant plate surface temperature (Watt)

ϕ = relative humidity of the ambient air (fractional)

The intrinsic water vapor resistance R_{et} of the fabric may be determined by subtracting out the value of the water vapor resistance measured for the bare plate, R_{eto} :

$$R_{et} = R_{etotal} - R_{eto} \quad (13)$$

The same group of materials which were tested in the DMPC were also tested according to ISO 11092, and a correlation was determined. In Figure 8 we show the correlation between the two tests for the permeable fabrics and the microporous laminated membranes. The values for the hydrophilic membrane materials are not shown on Figure 8, since they are much more dependent on test conditions than are the values for the fabrics and microporous membrane laminates.

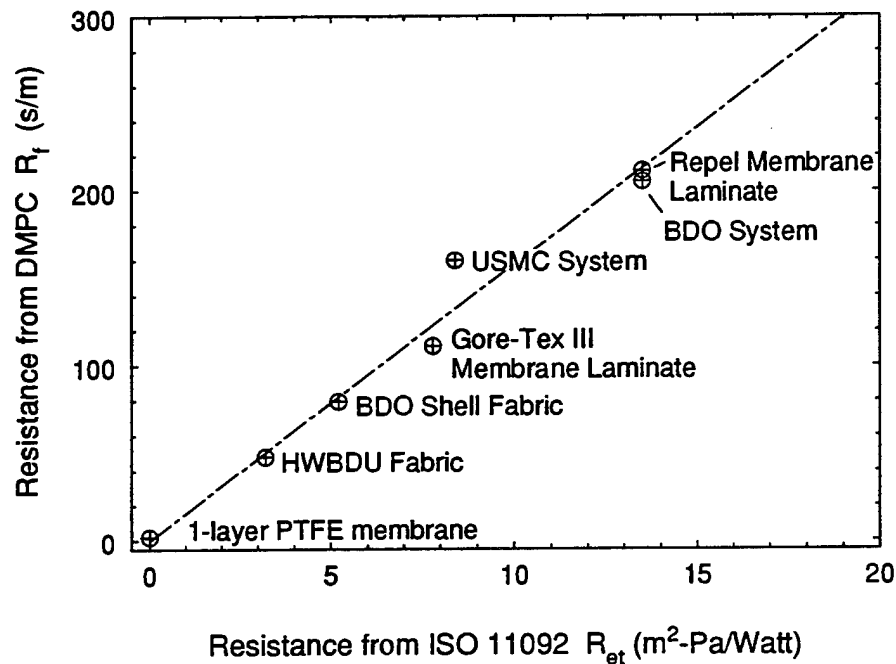


Figure 8. Correlation Between DMPC Test and ISO 11092 Sweating Guarded Hot Plate Test.

We see that we have good correlation between the two types of tests for the materials shown. We feel that this is an excellent correlation between the tests, especially when one considers that the test methods are very different, in that the sweating hot plate is a calorimetric method, and the DMPC is a direct measurement of concentration differences.

The intrinsic water vapor resistance is equivalent to the intrinsic mass transfer resistance measured in the DMPC apparatus. We may also convert the R_{et} value obtained from the sweating guarded hot plate to the intrinsic mass transfer units of (s/m) used in the DMPC test.

$$R_f = R_{et} \left(\frac{M_w \Delta H_{vap}}{RT} \right) \quad (14)$$

R_f = intrinsic mass transfer resistance (s / m)

R_{et} = intrinsic water vapor resistance (m^2 - Pa / Watt)

ΔH_{vap} = enthalpy of vaporization for water (2.42×10^6 J / kg @ 35°C)

R = universal gas constant (8314.5 N - m / Kgmole - K)

T = temperature (K)

We can make another small correction for the difference in test temperatures for the fabrics and microporous laminates by using the ratio of the diffusion coefficients of water vapor in air at the two test temperatures. This particular temperature correction is only valid for the materials in which water vapor transport occurs through the air spaces of the material. A similar temperature correction for the hydrophilic solid polymer membranes would be possible if information is available on the variation of their diffusion coefficients with temperature.

$$R_f(@T_1) = R_f(@T_2) \left(\frac{D_a(@T_1)}{D_a(@T_2)} \right) \quad (15)$$

$$D_a = 2.23 \times 10^{-5} \left[\frac{(T + 273.15)}{273.15} \right]^{1.75} = \text{diffusion coefficient of water vapor in air (m}^2/\text{s)}$$

We can show the same correlation after we have converted the ISO 11092 resistance units to mass transfer resistance units of (s/m). We also show the resistance of the hydrophilic materials on this plot.

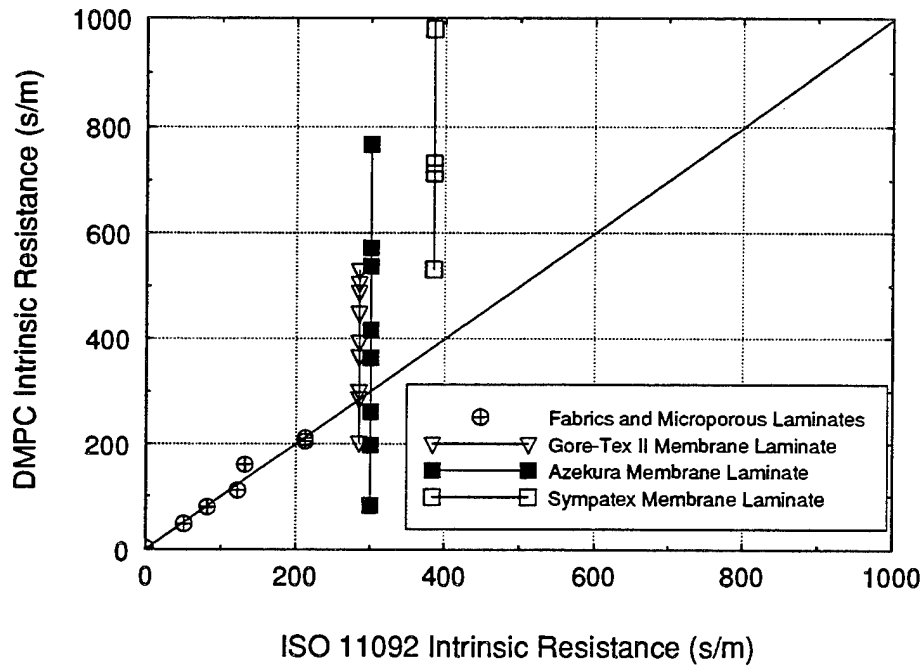


Figure 9. Correlation Between DMPC and ISO 11092 Sweating Guarded Hot Plate in Units of s/m, and Including Values for Hydrophilic Membrane Laminates.

Since Figure 9 shows the mass transfer resistance in identical units of s/m, the agreement between the two tests is even more clear. Even though the tests are conducted at two different temperatures, differing by 15 °C, the correlation between the two tests is almost perfect. Again, we also see from Figure 9 that the evaluation and ranking of hydrophilic membrane laminates is much more difficult, and requires more thought than when evaluating the simpler types of materials. We would also expect that the temperature of the test would affect the values obtained by the two tests, since the transport behavior of the solid polymer layer is more affected by temperature. The values for the Sympatex membrane laminate do not fall within the correlation, but as discussed previously, the resistance of this material is so high that the results are of doubtful accuracy. Even the sweating guarded hot plate results are not felt to be very accurate for this material, since very little power was required to maintain the plate temperature at 35°C, because not much water was able to diffuse through the material.

5. Effect of Parallel versus Countercurrent Flows in the DMPC

The fact that we use a log-mean concentration difference in analyzing the results for the DMPC means that we recognize the direction of flow has an influence on the concentration profile down the length of the test sample. For parallel flow, we would expect that the concentration difference between the two sides of the sample becomes smaller at the downstream end of the sample. To maintain a more constant concentration gradient down the length of the sample, we would expect that a countercurrent arrangement would promote higher fluxes (and possibly lower boundary layer resistances). The two flow situations are illustrated in Figure 10.

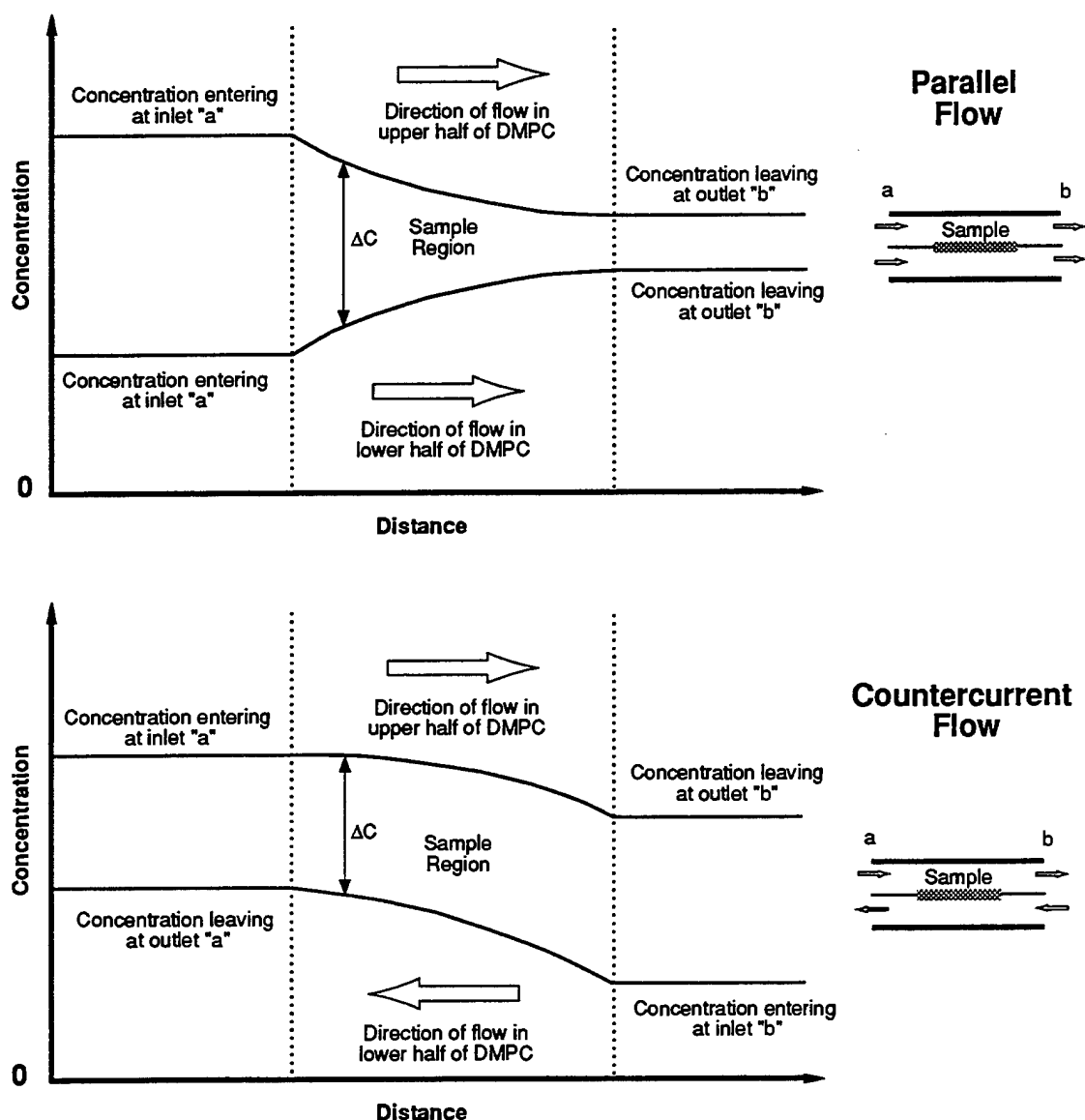


Figure 10. Effect of Parallel or Countercurrent Flow Direction on Concentration Gradient Across Test Sample.

We were curious to see if the direction of flow has a large effect on the results obtained with DMPC. We used two materials to look at this effect. The 1-layer PTFE membrane was tested in both parallel and counterflow to determine the relative effect on the boundary layers. We also tested the Gore Tex III sample in both the parallel and counterflow situations. The results are shown in Figure 11.

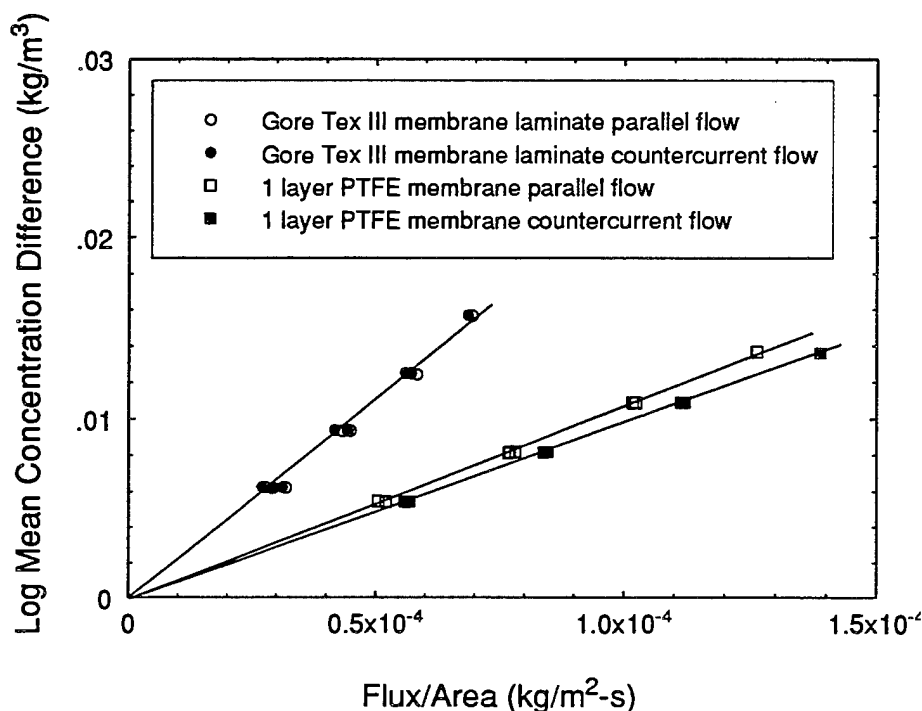


Figure 11. Testing of Two Materials in Both Parallel and Counterflow Arrangements in the DMPC.

We do not observe any difference in the measured properties of the Gore Tex III membrane laminate for the two flow situations. We do see some differences in the measurement for the PTFE membrane, which since it has a resistance of 6 to 8 s/m, is essentially a measurement of the boundary layer resistance. From the slope of the lines in Figure 11, the parallel flow resistance is about 105 s/m and the counterflow resistance is about 95 s/m. The countercurrent flow resistance is lower, which we would expect, but the difference between the two flow situations is not large enough to be discernible for materials with lower water vapor transmission rates. Although all the testing results given in this report are for parallel flow, it may be better practice to conduct testing using countercurrent flow, to maximize water vapor flux, and to maintain a more nearly constant concentration difference across the test sample. Both methods would be useful when studying the hydrophilic materials, since for the same test conditions of relative humidity in the upper and lower halves of the DMPC, the mean relative humidity (average of both sides) would be quite different down the length of the sample for the two different flow situations.

6. Conclusions

The dynamic moisture permeation cell permits rapid testing of small quantities of permeable fabrics and semipermeable laminates in a short amount of time and under a wide variety of test conditions. The DMPC allows one to examine a material's transport behavior under test conditions which are difficult or impossible to reach using existing standard laboratory test methods.

The moisture permeation cell has been automated to permit multiple setpoint testing under computer control. This provides the ability to evaluate more materials while at the same time decreasing the time necessary to conduct a test, which is a major drawback to the two tests presently in use (ASTM E-96 and ISO 11092).

The moisture permeation cell has an excellent correlation and agreement with the ISO 11092 sweating guarded hot plate test method. We feel confident that the results generated with the DMPC are of comparable accuracy to the ISO 11092 method of measuring water vapor permeability.

The moisture permeation cell will be particularly useful in evaluating the concentration-dependent permeation behavior of hydrophilic semipermeable membrane laminates. The ASTM E-96 cup method, which has been the primary evaluation method for these types of materials, gives very misleading results about the properties and ranking of material candidates for new U.S. Army clothing and uniform systems. The moisture permeation cell will be particularly useful in the chemical protective clothing area, where new semipermeable membrane laminates are constantly being developed and evaluated, and where good water vapor transmission properties are critically important for providing clothing systems which do not produce a heat stress burden on the wearer.

7. References

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20. Military Specification, MIL-C-43858B(GL), Cloth, Laminated, Nylon Tricot Knit, Polyurethane Foam Laminate, Chemical Protective and Flame Resistant, Type III.
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APPENDIX

Flow Analysis of the DMPC

A rough schematic of the DMPC is shown below in Figure A-1, with relevant flow dimensions indicated. The flow path both above and below the sample is a long, wide duct, with a small clearance between the sample and the upper or lower duct surface. The flow in such a duct is approximated by the flow between parallel plates.

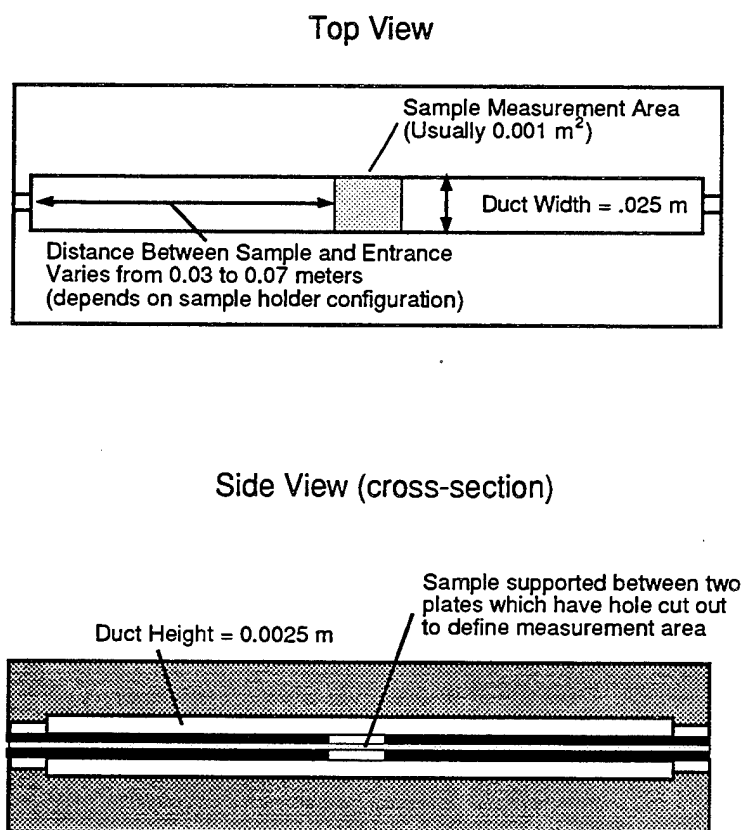


Figure A-1. Schematic of the Dynamic Moisture Permeation Cell

Since our flow velocities are so low, and our flow geometry is quite narrow, we know that the flow is laminar. We just want to make sure that the entrance length is long enough for the flow to become fully developed by the time the flow reaches the sample.

For laminar flow in a duct, the entrance length for the velocity profile to become fully-developed is given by:

$$X_{Lv} = 0.05 \text{Re}_{Duct} H = 0.022 \text{ m}$$

X_{Lv} = entrance length for fully developed momentum boundary layer

$$\text{Re}_{Duct} = \frac{\rho V D_H}{\mu} = 175$$

$$\rho = 1.2 \text{ kg/m}^3 \text{ (Dry Nitrogen)}$$

$$V = 0.57 \text{ m/s (based on flow rate of } 2000 \text{ cm}^3/\text{min} \text{ and given flow area)}$$

$$D_H = \text{hydraulic diameter} = 4 \times (\text{flow area} / \text{wetted perimeter}) = 0.00454 \text{ m}$$

$$\mu = \text{absolute viscosity} = 1.78 \times 10^{-5} \text{ N-s/m}^2$$

$$H = \text{duct height (0.0025 m)}$$

The fluid properties and geometry of the duct give an entrance length for the fully-developed velocity profile of 0.02 meters, or 2 centimeters, which is a good distance away from the beginning of the sample location in the moisture permeation cell.

We can check this result by also assuming that we have flow between parallel plates, and base our Reynolds number solely on the plate separation distance. In the paper by Chen [24], we have a solution for the entrance length as a function of Reynolds number.

The Reynolds number is defined based on the half-height of the channel:

$$Re = \frac{2a\rho V}{\mu} = \frac{H\rho V}{\mu} = 96$$

$$a = \text{duct half - height} = H/2$$

For parallel plate flow, the dimensionless entry length X_e is given as a function of Reynolds number as:

$$X_e = \frac{x_e}{2a} = \frac{x_e}{H} = \frac{0.79}{0.04 Re + 1} + 0.053 Re = 6.09 \Rightarrow x_e = 0.015 \text{ m}$$

As we will see later from the flow analysis using a two-dimensional numerical fluid flow program, this shorter entrance length of 1.5 cm agrees with the calculated flow velocity profiles.

These calculations, which we did before we designed the cell, give us confidence that we have a well-characterized and stable flow over the sample. We can use the known laminar velocity profile to compare the measured boundary layer mass transfer coefficients with what we can calculate. For example, for flow between parallel plates, the fully-developed laminar velocity profile is parabolic, with the horizontal velocity distribution given in terms of the nominal bulk flow velocity (V_{nom}) as:

$$u = 6V_{nom} \left(\frac{y}{H} - \frac{y^2}{H^2} \right); \text{ the maximum centerline velocity is given by } u_{max} = 1.5 V_{nom}.$$

We can use the analytical results to check our numerical calculations of the boundary growth within the flow geometry of the moisture permeation cell (DMPC). We used the SIMPLEC program [25] to examine two situations. The first situation is when the flow enters uniformly at a velocity determined by the given mass flow rate and the flow area. The second situation is when the flow enters as a jet. The second situation is more realistic for the DMPC since the flow actually enters through a hole drilled into the entrance of the flow duct.

We only show the horizontal (u) components of velocity on the graphs. For the situation where the flow entered as a jet, there was swirl and backflow near the entrance, as there should have been, and the velocity components reflected this.

Figure A-2 shows the centerline velocity down the duct for the analytical case, and for the two numerically calculated cases.

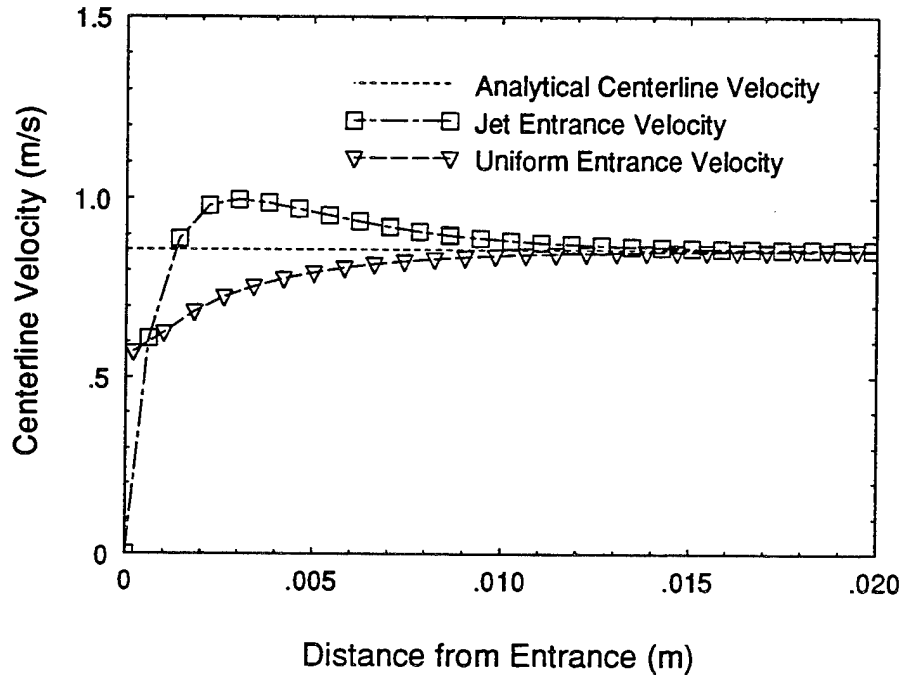


Figure A-2. Centerline Flow Velocity Near Duct Entrance.

We see that even for the extreme case of the jet, we get a stable fully-developed flow within the first 2 cm of the duct.

We can also show the actual velocity profiles (horizontal component) at different stations along the duct for the two cases of the flow entering uniformly, and for the flow entering as a jet.

First, for comparison, we will show in Figure A-3 the fully developed velocity profile from the analytical solution .

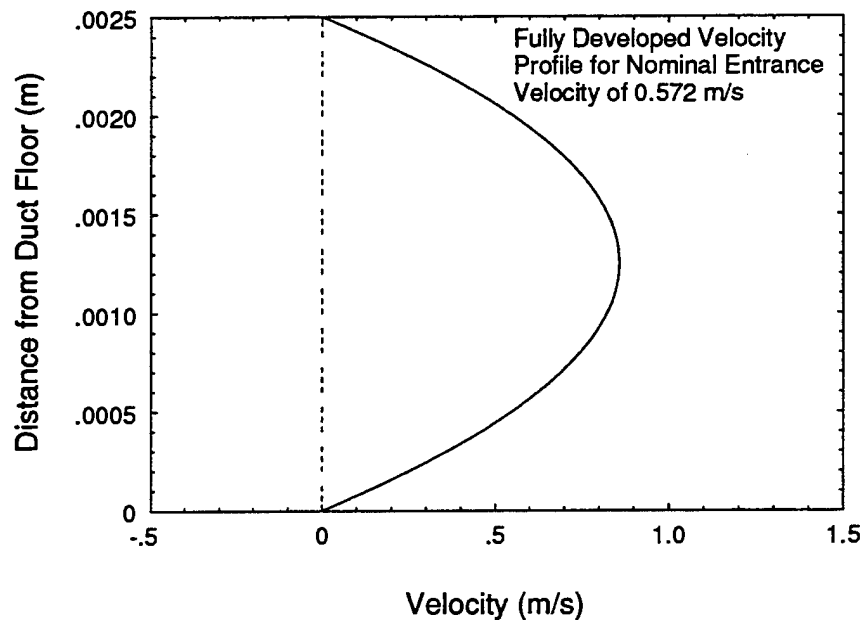


Figure A-3. Analytical Fully-Developed Flow Profile.

Figure A-4 shows the results of the flow simulation using the SIMPLEC code for the uniformly entering flow. To avoid clutter, the location of each flow profile is not shown, but the flow, which is initially a square, uniform velocity profile, assumes the fully-developed velocity profile within 2 centimeters.

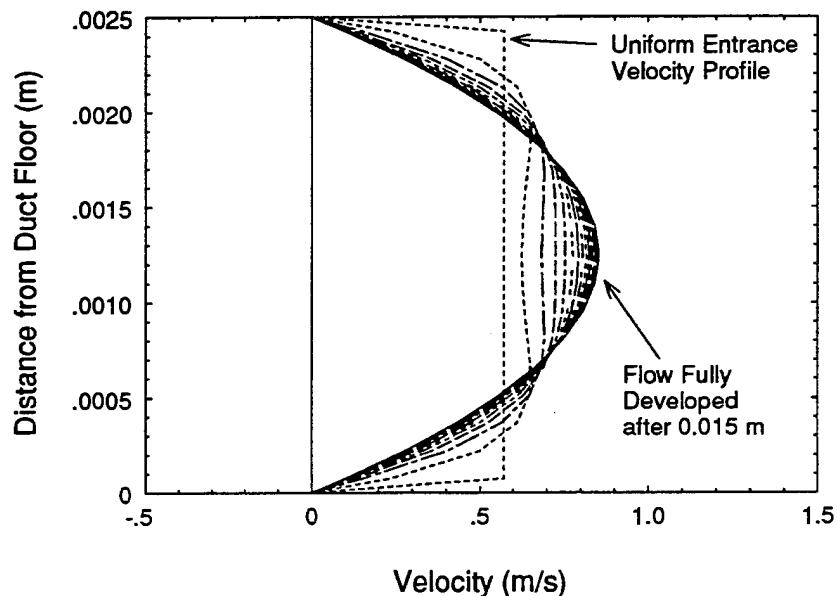


Figure A-4. Flow Development Near Duct Entrance for Uniform Entrance Velocity Profile.

The case of the flow entering as a jet is shown in Figure A-5. We see that there is some backflow and swirl near the entrance (also indicated by vertical velocity components not shown), but the flow quickly assumes a fully-developed profile within 2 centimeters after entering the cell.

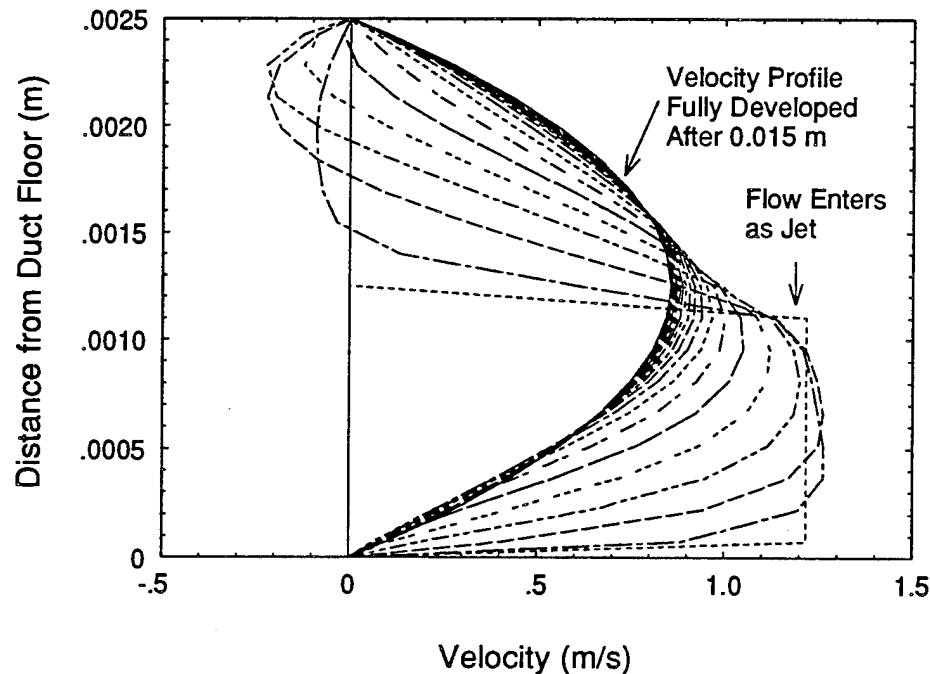


Figure A-5. Flow Development Near Duct Entrance for Flow Entering as Jet.

The moisture permeation cell seems to be well-designed to give an stable, fully-developed flow. For an actual test, we will have diffusion or diffusion/convection of water vapor from one side of the cell to the other through the sample, so we will have non-symmetric concentration contours within the flow areas of the cell, and we will have transient concentrations and temperatures (if the sample is hygroscopic) which are not uniform either through the thickness of the sample or across the length of the sample.

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