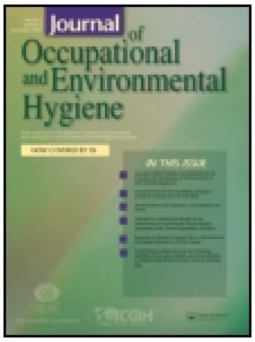

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Development of a Computer Program for Permeation Testing Data Analysis

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A Microsoft Windows-compatible computer program, referred to as "Permeation Calculator," was developed at the National Institute for Occupational Safety and Health (NIOSH) to automate and standardize permeation testing data analysis. The program imports the data file collected during a permeation test and calculates relevant permeation parameters within a few seconds, based on a series of algorithms, strategies, and automated decision-making processes. It allows calculations of all the permeation parameters related to American Society for Testing and Materials (ASTM) F 739, International Organization for Standardization (ISO) 6529, and ASTM D 6978 standards, including standardized breakthrough time, normalized breakthrough time, breakthrough detection time, steady-state permeation rate, cumulative permeation mass at a given elapsed time, and elapsed time at a given cumulative permeation mass for either a closed-loop or an open-loop permeation test. For open-loop permeation testing, the software also allows changing sampling flowrate and allows calculations of average permeation rate and maximum permeation rate to see if the rates ever reach the threshold maximum for decision making. On completion, the software displays all the permeation parameters together with relevant information and the permeation curve as a report file in Microsoft Excel and text file formats. This software helps industrial hygienists and researchers to avoid labor-intensive hand calculations of the permeation parameters. The software also prevents experimenter bias, thus ensuring identical permeation parameters will be obtained from a given permeation testing data file. The Permeation Calculator is available either on the NIOSH website or on CD by request.

Keywords breakthrough time, chemical protective clothing, computer program, permeation testing, cumulative permeation, permeation rate

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INTRODUCTION

Permeation testing has been widely used for the determination of chemical resistance of protective clothing, including gloves, garments, footwear, and full ensembles.(1) A permeation test can be performed in either a closed-loop or an open-loop mode. A closed-loop testing system has no outlet, and the permeated chemical flows through a nondestructive analytical device, often a MIRAN infrared gas analyzer. Because the permeated chemical is not exhausted, it accumulates inside the testing system. The closed-loop test can be conducted using either continuous sampling or discrete sampling. The discrete sampling includes tests when sample volume is replaced and also when sample volume is not replaced.(2) For an open-loop test, fresh collection medium flows continuously through the collection chamber of the test cell and is not reused or recycled. A gas chromatograph (GC) is often used as the analytical device for sampling and analyzing a portion of the permeant stream and the remainder is vented.

Figure 1 illustrates the configurations of the two testing modes. Typical permeation curves for each of the two testing modes are shown in Figure 2, assuming that the permeation behaviors obey Fick's Laws, i.e., the permeation rate stabilizes at a steady-state value. While Figure 2 shows the most typical permeation behavior, the other four different permeation behaviors that are not frequently observed were identified by Nelson et al.(3)

After a permeation test is completed, the permeation parameters are calculated as indicators of the chemical resistance. The parameters include standardized breakthrough time (SBT),(2) normalized breakthrough time (NBT),(4) breakthrough detection time (BDT), steady-state permeation rate (SSPR), cumulative permeation mass at a given elapsed time (CP_a), elapsed time at a given cumulative permeation mass (T_a), average permeation rate($\bar{P}$), and maximum permeation rate.

In addition to using these permeation parameters to select suitable chemical protective clothing, recent studies also

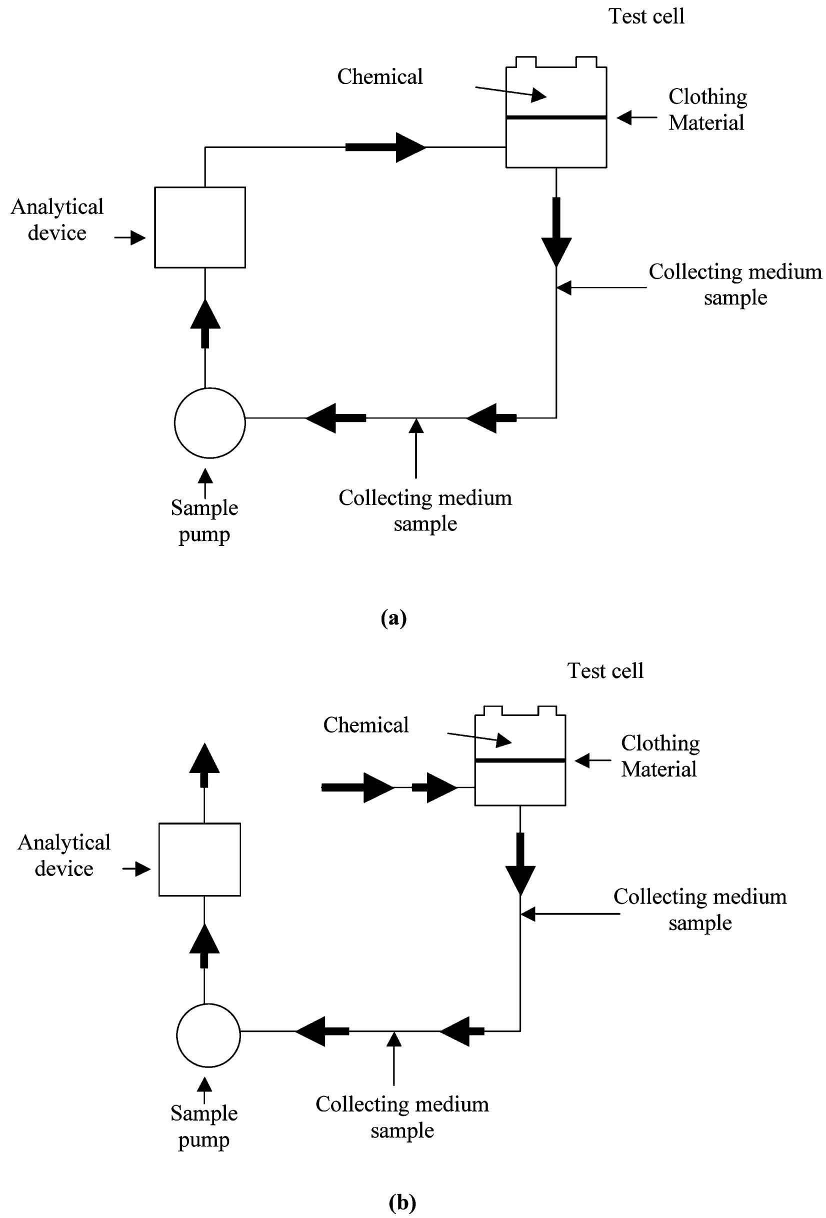

FIGURE 1. Configurations of permeation testing systems. (a) closed-loop testing system, and (b) open-loop testing system

used change in permeation parameters[5] combined with tensile properties[6] as indexes for evaluating decontamination efficacy of chemical protective clothing. For these studies, new clothing materials were exposed to test chemicals, followed by decontamination procedures. Changes in their permeation parameters were measured.

Because data analysis for permeation testing involves a number of equations[2,4] and experimental factors,[5–7] experimenter bias and possible calculation errors are critical concerns when determining permeation parameters. For instance, hand calculations of breakthrough detection times using seven open-loop permeation data files performed by two NIOSH personnel showed relative differences ranging from 5.1% to 29.9% ($p = 0.029$, one-tail paired t-test), with an average of 19.2% (Table I). Furthermore, because the calculations of some of the permeation parameters are mathematically complex and time-consuming, they are best handled by a computer. Therefore, a Microsoft Windows-compatible computer program, referred to as Permeation Calculator, was developed for this purpose. The latest version, together with detailed operating instructions and screen shots, is available either on the NIOSH website[8] or on CD by request.

DESCRIPTION OF THE COMPUTER PROGRAM

Permeation Calculator was created using Microsoft Visual C++ Version 2003 (Microsoft Corporation, Redmond, Wash.) and compiled to an executable file "PermCalc.exe."

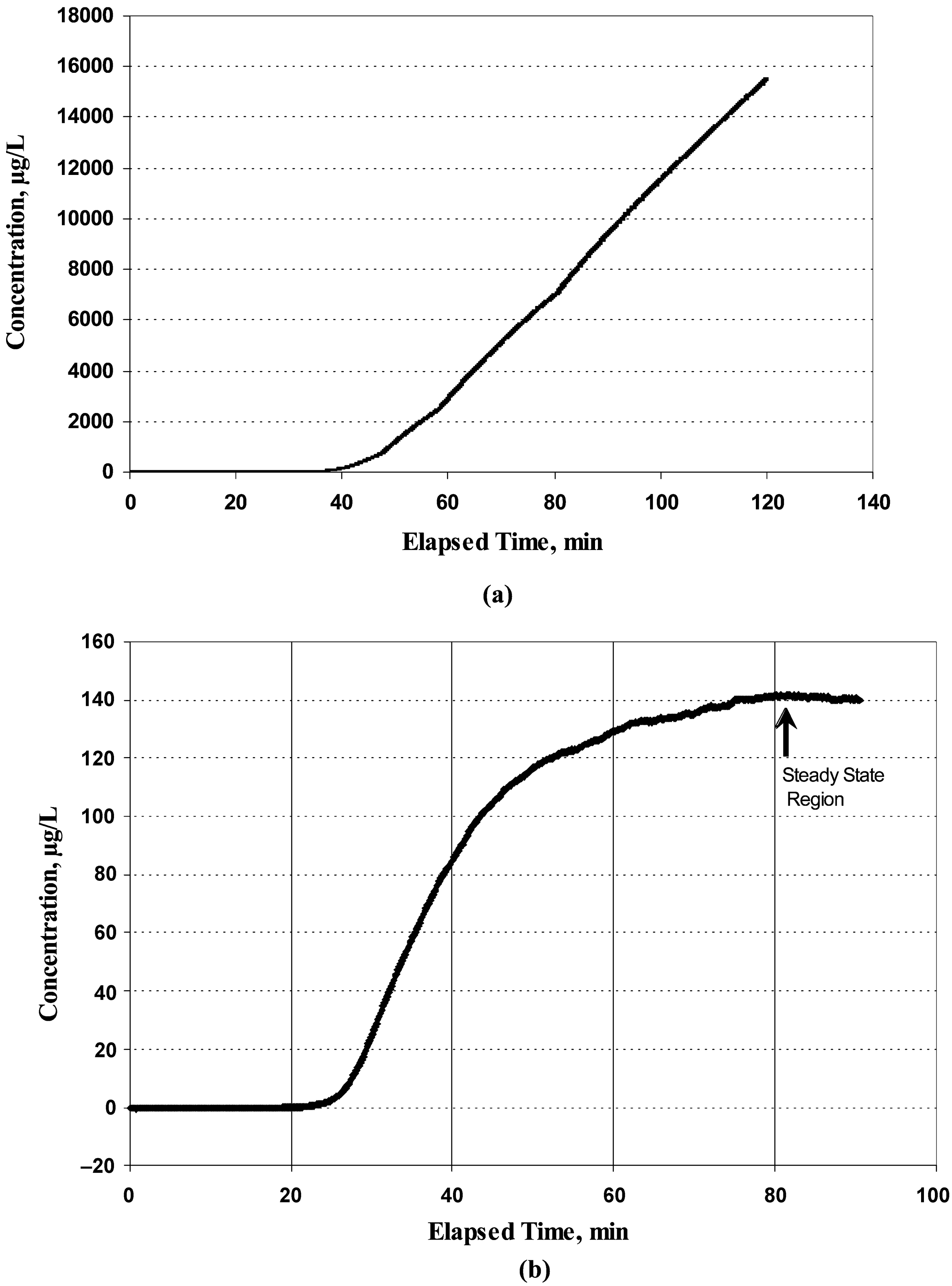

FIGURE 2. Typical permeation curves. (a) permeation curve for a closed-loop testing system, and (b) permeation curve for an open-loop testing system

TABLE I. Comparison of Hand Calculations Conducted by Two NIOSH Personnel

Sample ID	BDT, min		Relative Difference (%)	SSPR, μg/cm^2/min		Relative Difference (%)
	Subject A	Subject B		Subject A	Subject B	
1	47	56.3	18.0	100.92	96.59	−4.4
2	44	58.0	27.5	83.31	81.57	−2.1
3	52	54.7	5.1	412.25	405.00	−1.8
4	6.5	7.5	14.3	1676.10	1721.4	2.7
5	7.4	8.8	17.3	1365.85	1375.9	0.7
6	5.2	6.5	22.2	4289.26	4273.2	−0.4
7	5.4	7.3	29.9	4175.19	4165.9	−0.2
Paired t-test (one-tail)		p = 0.029			p = 0.39	

It will run on the following 32-bit or 64-bit operating systems: Microsoft Windows 95, Windows 98SE, Windows NT, Windows 2000, and Windows XP. The program will be modified to be compatible with future versions of Microsoft Windows if needed. The program starts by importing permeation testing data located in a Microsoft Excel-formatted file, which contains data points in time vs. concentration or other analyzer output readings.

The program then allows the user to enter variables under the "Choice of Variable" window. The user has different options to specify the analyzer response format: Option 1: Use concentration (in μg/L), Option 2: Use concentration (in ppm), or Option 3: Use other analyzer output reading. If Option 2 is selected, the molecular weight of the test chemical needs to be entered. If Option 3 is selected, the user needs to enter an equation for converting the output reading to a concentration reading in μg/L to generate a standard curve. This equation can be linear or polynomial (2nd to 9th order). The output reading could be the signal or response for the analyzer that was used to measure the amount of test chemical that permeated through the test material, such as concentration, voltage, GC peak height, or GC peak area, etc. In the next step, the program allows the user to select the Time Format either in minutes, YYYY/MM/DD HH:MM:SS, or MM/DD/YYYY HH:MM:SS ##. After that, the program asks the user to select either Open Loop System or Closed Loop System for the Choose System Type section.

Next, the program calls for more variables under the Data Input window. These include the diameter of the swatch contacted, specimen weight, the time value to be used to calculate the cumulative permeation, the value of "mass/area" for the cumulative permeation mass target section, and the time values to be used to calculate the average permeation rate.

Finally, the program allows the user to enter additional information under the Additional Data Input window. Although the information is not required for the calculations, it will be incorporated into the final report file. These include report title, project number, operator, date, material type, average material thickness, name of the test chemical, physical state of the test chemical, collection medium, the analytical instrument that was used, data sampling interval and test temperature when conducting the permeation testing. The user can enter part or all of the information in the fields, or leave all the fields blank.

After all the required quantities for the calculations are entered, the program calculates the permeation parameters and then displays a report file immediately after the user clicks Finish on the screen. On completion of the calculation, the software displays all the permeation parameters together with relevant information and the permeation curve as a report file that can be saved in Microsoft Excel and text file formats.

This computer program allows calculation of all the permeation parameters related to American Society for Testing and Materials (ASTM) F 739 and International Organization for Standardization (ISO) 6529 standards, as stated earlier. The closed-loop test includes continuous sampling and discrete sampling. The discrete sampling includes tests when sample volume is replaced and also when sample volume is not replaced. For open-loop permeation testing, the software also allows changing sampling flow rate and allows calculations of average permeation rate and maximum permeation rate to see if the rates ever reach the threshold maximum for decision making; although some of these options are not included in current versions of the ASTM F 739 and ISO 6529 standards.

This program is also applicable to ASTM D 6978 standard[9] titled *Standard Practice for Assessment of Resistance of Medical Gloves to Permeation by Chemotherapy Drugs*. The ASTM D 6978 standard specifies a closed-loop permeation testing with discrete sampling and when volume is replaced to determine minimum breakthrough detection time, which is defined as the time at which the permeation rate reaches 0.01 μg/cm^2/min. However, it is not applicable to ASTM F 1383 standard titled *Standard Test Method for Resistance of Protective Clothing Materials to Permeation by Liquids and Gases Under Conditions of Intermittent Contact*,[10] because the permeation behavior under conditions of intermittent contact is different from that for continuous contact.

METHOD OF CALCULATIONS

The following symbols are used in the calculations, where:

a = a coefficient for a polynomial equation, Eqs. 1–3 and 15; an arbitrary data point before data point b, Eqs. 6–10
A = area of the material specimen contacted, cm^2, Eqs. 4, 5, 11, 13, and 14
b = a constant for a polynomial equation, Eqs. 1–3 and 15; an arbitrary data point after data point a, Eq. 10
C = concentration of test chemical in collection medium, μg/L, Eqs. 11, 13, and 14
$\overline{C}$ = average concentration of test chemical in collection medium, μg/L, Eq. 5
CP = cumulative permeation beginning with initial chemical contact, $\mu g/cm^2$, Eqs. 6–11, 13, and 14
F = flow rate of collection medium through the permeation cell, L/min, Eq. 5
i = data point, Eqs. 11–14; data point immediately before data point a or b, Eqs. 6–9
m = a collection or a series of data points i, Eqs. 7 and 9
n = total number of data points i, Eqs. 11, 13, and 14
P = permeation rate, $\mu g/cm^2$/min, Eqs. 7 and 9
$\bar{P}$ = average permeation rate for the time interval T_a to T_b, $\mu g/cm^2$/min, Eq. 10
R = correlation coefficient of a regression analysis
$SSPR$ = Steady-state permeation rate, $\mu g/cm^2$/min, Eqs. 4 and 5
T = elapsed time, min, Eqs. 6–10
V_t = total volume of the collection medium, L, Eqs. 4, 12–14
V_s = volume of discrete sample removed from the collection medium, L, Eqs. 11–14
x = value of x axis in a permeation curve, min, and
y = value of y axis in a permeation curve, μg/L, $\mu g/cm^2$, or $\mu g/cm^2$/min.

Breakthrough Detection Time for Closed- and Open-Loop Permeation Tests

Breakthrough detection time is the elapsed time measured from the start of the test to the sampling time that immediately precedes the sampling time at which the challenge chemical is first detected.[2,4] The program first calculates the slope and regression correlation coefficient centered around each data point n starting at n = 8 by performing a linear regression for points n-7 to n+7. It then calculates the slope between the data point closest to 50% and the data point closest to 90% of the maximum concentration, i.e., $(y_{90}-y_{50})/(x_{90}-x_{50})$. This is referred to as the largest slope. The calculations stop when the following conditions are met: (1) the slopes calculated increase consecutively for 7 times, (2) each of these 7 slopes is greater than 2% of the largest slope calculated, and (3) the square of the correlation coefficient (R^2) for the last slope is greater than 0.9.

When the last slope is determined, the program selects the first data point used in that slope's calculation as the breakthrough point (BP). It uses the data points from BP to the point closest to 15% of the maximum concentration to perform a regression analysis for a polynomial equation ($y_{BP} = ax^2+bx+c$), as illustrated in Figure 3a. The program then calculates the breakthrough detection time by solving the polynomial equation for x; taking either root x1 or x2, whichever is closest to x_{BP}.

Based on our evaluation, adequately predicting the real tendency of the data could not be assured when using fewer than 7 data points before the breakthrough point for the linear regression analysis. This may result in an inability to accurately determine the breakthrough detection time for a data file that contains fewer than 7 data points before the breakthrough point. As shown in Table II, there is no statistical difference between the computed BDTs and those obtained by hand calculation on the same data ($p = 0.18$). Subject A's hand calculation results were used for the comparison because the individual had more experience in permeation date analysis than Subject B.

Standardized Breakthrough Time

According to ASTM,[2] standardized breakthrough time is the time at which the permeation rate reaches 0.1 $\mu g/cm^2$/min for either a closed-loop or an open-loop permeation test.

Closed-Loop Permeation Test with Continuous Sampling

As shown in Figure 3b, for a permeation curve of y ($\mu g/cm^2$) against x (min), the program performs a regression analysis using a range of data points to obtain a polynomial equation, i.e., Eq. 1. The first data point is the one with an elapsed time closest to 75% of the time value for the BP, as determined previously for calculation of the BDT, and the last data point is the one with a CP closest to 15% of the maximum CP.

$$y = ax^2 + bx + c \quad (1)$$

Take the derivative to obtain the permeation rate in $\mu g/cm^2$/min:

$$\frac{dy}{dx} = 2ax + b \quad (2)$$

Based on the ASTM definition stated above,[2] let

$$2ax + b = 0.1 \quad (3)$$

Solve Eq. 3 for the standardized breakthrough time (x). If the calculated x value is outside the time range for the data points used for the regression, repeat the above procedures. The data for the next regression analysis use the same number of data points but the starting point is incremented by one. Once the conditions are satisfied, the value of x as standardized breakthrough time for a closed-loop permeation test is then reported.

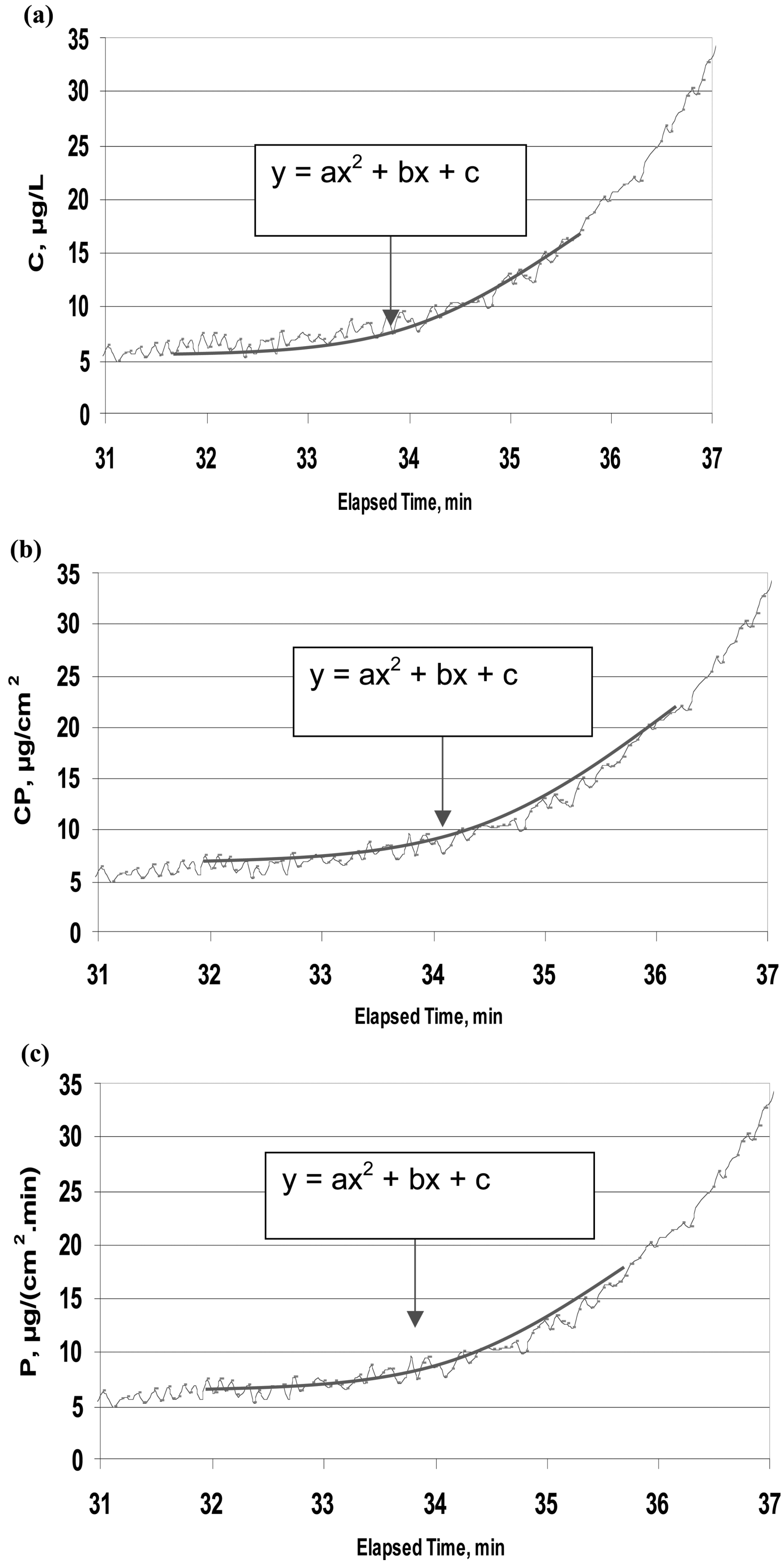

FIGURE 3. Regression analyses for determination of various breakthrough times. (a) concentration against elapsed time for either a closed-loop or an open-loop testing system, (b) cumulative permeation against elapsed time for closed-loop testing systems, and (c) permeation rate against elapsed time for open-loop testing system

TABLE II. Comparison of Hand Calculations vs. the Permeation Calculator Software

Sample ID	BDT, min		Relative Difference (%)	SSPR, μg/cm^2/min		Relative Difference (%)
	Subject A	Software		Subject A	Software	
1	47	47.1	0.2	100.92	100.91	0.0
2	44	43.7	−0.7	83.31	83.29	0.0
3	52	58.5	12.5	412.25	411.53	−0.2
4	6.5	6.61	1.7	1676.10	1682.33	0.4
5	7.4	7.48	1.1	1365.85	1367.98	0.2
6	5.2	5.18	−0.4	4289.26	4287.07	−0.1
7	5.4	5.38	−0.4	4175.19	4172.82	−0.1
Paired t-test (one-tail)		p = 0.18			p = 0.36	

Open-Loop Permeation Test

The program looks for two consecutive points where the permeation rate at point i < 0.1 μg/cm^2/min and point i+1 $\geq$ 0.1 μg/cm^2/min and selects the point with a permeation rate closest to the ASTM specified value of 0.1 μg/cm^2/min. It then performs a regression analysis[11] using 11 data points centered around the selected point to obtain a polynomial equation ($0.1 = ax^2 + bx + c$), as shown in Figure 3c. The standardized breakthrough time is calculated by solving the polynomial equation, which results in two possible root values. The program selects the root value that is closest to the time of the two consecutive points i and $i + 1$.

Calculating standardized breakthrough time by taking the regression analysis and then solving the polynomial equation allows the program to determine a more accurate value within a data collection time interval, since it is not limited to the times (T_i) that are shown in the data file. The regression analysis serves the same purpose for the calculations of normalized breakthrough time and breakthrough detection time.

Normalized Breakthrough Time

Based on ISO Method 6529:2001,[4] normalized breakthrough time is the elapsed time at which the permeation rate reaches 1.0 μg/ cm^2/min for an open-loop test. In a closed-loop test, it is the time at which the mass of chemical permeation reaches 2.5 μg/cm^2. The processes as described in this section are similar to finding the Standardized Breakthrough Time for an open-loop permeation test. However, the equations are solved for different constants due to the differences between ISO 6529 and ASTM F 739 standards.

Closed-Loop Permeation Test with Continuous Sampling

In this case, the program first looks for two consecutive points where the cumulative permeation at point i < 2.5 μg/cm^2 and point i + 1 ≥ 2.5 μg/cm.^{2} Normalized breakthrough time is calculated by solving the polynomial equation ($2.5 = ax^2 + bx + c$) (Figure 3b). Again, the program selects the root value that is closest to the time of the two consecutive points i and i + 1.

Open-Loop Permeation Test

The program looks for two consecutive points where the permeation rate at point i < 1.0 μg/cm^2/min and point i+1 ≥ 1.0 μg/cm^2/min and selects the point with a permeation rate closest to 1.0 μg/cm^2/min. It then performs a regression analysis[11] using 11 data points centered around the selected point to obtain a polynomial equation ($1.0 = ax^2+bx+c$) as shown in Figure 3c. Normalized breakthrough time is calculated by solving the polynomial equation. The program takes the root x1 or x2, whichever is closest to the time of the two consecutive points that were found at the beginning.

Steady-State Permeation Rate (SSPR)

Steady-state permeation rate is the constant rate of permeation that occurs after breakthrough when the chemical contact is continuous and all forces affecting permeation have reached equilibrium.[4]

Closed-Loop Permeation Test with Continuous Sampling

For a permeation curve of y (μg/L) against x (min), the program determines the slope of the steady-state region by taking a linear regression of the data points between 65% and 85% of the maximum concentration point to obtain the slope. This region was selected so as to avoid the transition region and possible nonlinear region which sometimes happens with higher concentrations. The transition region appears after breakthrough and before a steady state is achieved.[13] The SSPR is then calculated based on Eq. 4:

$$SSPR = \frac{Slope * V_t}{A} \quad (4)$$

where slope is in μg/(L*min), V_t is total volume of the collection medium in L, and A is area of the material specimen contacted in cm^2.

Figure 4 shows a calculation of the SSPR for a closed-loop test as an example. A slope of 205.11 μg/(L*min) is determined for this data file. Note that Eq. 4 assumes that the concentration of permeant in the collection medium never rises to a level where it exerts a reverse driving force that

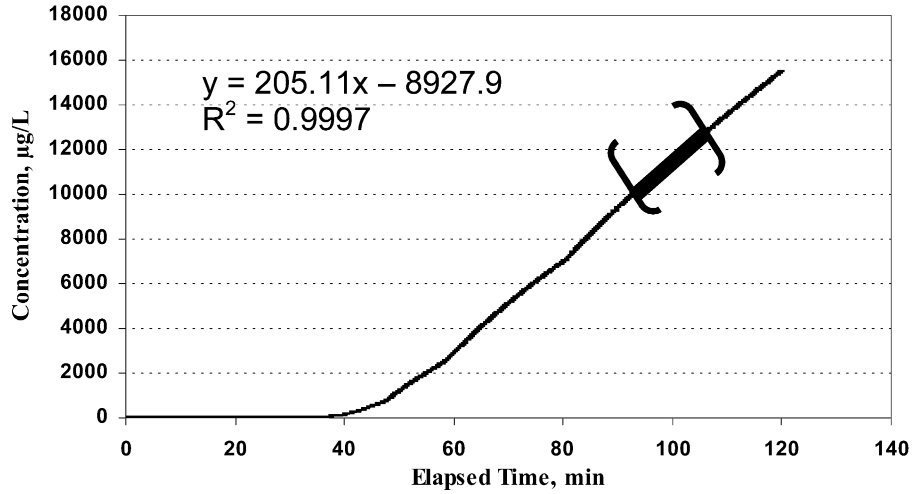

FIGURE 4. Determination of slope for a closed-loop permeation test

would move the permeant back to the challenge side of the permeation cell.

Open-Loop Permeation Test

The program finds three data points with the three highest concentrations located in the steady-state region as shown in Figure 2b and then takes the average of the three concentrations. The SSPR is calculated based on the following equation:[2,4]

$$SSPR = \frac{\overline{C} * F}{A} \tag{5}$$

where $\overline{C}$ is the average concentration of test chemical in collection medium in μg/L, F is flow rate of fresh collection medium through the permeation cell in L/min, and A is area of the material specimen contacted in cm^2.

Table II shows that there is no statistical difference between the computed SSPRs and those obtained by hand calculation on the same data ($p = 0.36$). For the user to determine the SSPR using more data points or remove any outliers, the program not only reports value calculated based on Eq. 5 as the SSPR but also reports the seven highest individual permeation rates.

Cumulative Permeation (CP_a) at a Given Elapsed Time

Cumulative permeation is the total amount of chemical that permeates during a specified time from the time the clothing material specimen is first contacted with the test chemical.[4]

Closed-Loop Permeation Test with Continuous Sampling

For the permeation curve of y (μg/cm^2) against x (min), the program identifies the data point (i) immediately before the given elapsed time (a), and the next data point (i+1). It then adds the cumulative permeation (CP) of the first point (i) to the resultant of the difference in cumulative permeation for these two points multiplied by the ratio of the difference between the given elapsed time(a) and the time (i+1), and divided by the difference in time (i) and time (i+1).

$$CP_a = CP_i + \frac{(CP_{i+1} - CP_i)(T_a - T_i)}{T_{i+1} - T_i} \tag{6}$$

Open-Loop Permeation Test

As shown in Figure 5, the shaded area under the permeation curve of y (μg/(cm^2*min)) against x (min) from t_0 to t_a is the cumulative permeation, which is the product of y (μg/(cm^2*min)) and x (min) with an unit of μg/cm^2 for cumulative permeation. First, the area from t_0 to the point immediately before the given elapsed time (T_i) is estimated by adding the area of trapezoids under the curve between consecutive points, using the mean permeation rate between points ([P(m) + P(m+1)]/2) multiplied by the difference in time between points (T(m+1) – T(m)). For the final portion between elapsed times (T_i) and (T_a), the proportion of the area

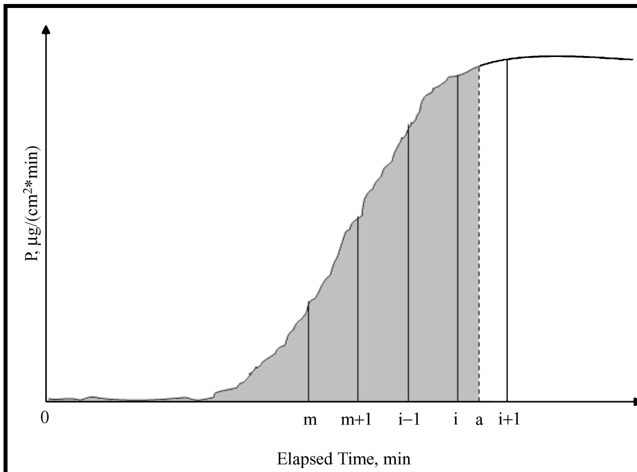

FIGURE 5. A diagram for calculating the cumulative permeation for an open-loop test

of the trapezoid between points i and i+1 is added: (T(a)-T(i))/(T(i+1)-T(i)). Note that for any intervals with a constant permeation rate, the trapezoid becomes a rectangle or square where P(n) = P(n+1), but the formula for calculating the areas remains the same.

$$CP_a = \sum_{m=0}^{i-1} \left[\frac{1}{2}(P_m + P_{m+1})(T_{m+1} - T_m) \right] + \frac{1}{2}(P_i + P_{i+1})(T_{i+1} - T_i) * \frac{(T_a - T_i)}{(T_{i+1} - T_i)},$$

which can be reduced to:

$$CP_a = \sum_{m=0}^{i-1} \left[\frac{1}{2}(P_m + P_{m+1})(T_{m+1} - T_m) \right] + \frac{1}{2}(P_i + P_{i+1}) * (T_a - T_i) \quad \textbf{(7)}$$

Elapsed time (T_a) at a Given Cumulative Permeation

The elapsed time at a given cumulative permeation is the time at which the cumulative permeation reaches an arbitrary amount in μg/cm^2 specified by the user. The calculation uses the same equation, i.e., Eq. 6, for cumulative permeation (CP) at a given elapsed time but solves for T_a rather than CP_a.

Closed-Loop Permeation Test with Continuous Sampling

For a given cumulative permeation CP_a, rearranging Eq. 6, T_a can be expressed as:

$$T_a = T_i + \frac{[T_{i+1} - T_i] * [CP_a - CP_i]}{CP_{i+1} - CP_i} \quad \textbf{(8)}$$

Open-Loop Permeation Test

For a given cumulative permeation CP_a, rearranging Eq. 7, T_a can be expressed as:

$$T_a = T_i + \frac{2\left\{CP_a - \sum_{m=0}^{i-1} \left[\frac{1}{2}(P_m + P_{m+1})(T_{m+1} - T_m) \right] \right\}}{P_i + P_{i+1}} \quad \textbf{(9)}$$

Average Permeation Rate ($\overline{P}$)

Average permeation rate is the ratio of the cumulative permeation to the specified time interval.

Closed-Loop Permeation Test with Continuous Sampling

The program first calculates cumulative permeations CP_a and CP_b using Eq. 6 for time a (T_a) and time b (T_b), respectively. The average permeation rate ($\overline{P}$) between time a (T_a) and time b (T_b) is then obtained:

$$\overline{P} = \frac{CP_b - CP_a}{T_b - T_a} \quad \textbf{(10)}$$

Open-Loop Permeation Test

The program calculates cumulative permeations CP_a and CP_b using Eq. 7 for time a (T_a) and time b (T_b), respectively. The average permeation rate ($\overline{P}$) between time a (T_a) and time b (T_b) is calculated using Eq. 10.

Maximum Permeation Rate for Open-Loop Permeation Testing

The program reports the maximum permeation rate, which is the highest permeation rate observed during the test.

Perform All the Permeation Calculations for Variable Sampling Flow Rate for Open-Loop Permeation Testing

As stated earlier, for an open-loop test, fresh collection medium flows continuously through the collection chamber of the test cell, and a gas chromatograph is often used as the analytical device. The range for the amount of chemical permeating through the material has the potential to be large because none or very little of the chemical permeates before breakthrough, and usually, a relatively large amount will permeate when steady-state permeation is reached. Therefore, it is sometimes necessary to increase the sampling flow rate to dilute the permeant stream during the test so that a good linearity for the calibration curve can be obtained.

The program first takes the product of the sampling flow rate (F) and the concentration (C) to calculate the permeation rate, i.e., P = C*F/A. Then, all of the permeation parameters can be calculated in the same manner as those for a constant sampling flow. Because the value of (C*F) is independent of changes in the sampling flow rate, concentration is inversely proportional to flow rate. For variable sampling flow rate, an additional column in the Excel data file is needed. Although variable sampling flow rate is not included in current ASTM F 739 and the ISO 6529:2001 standards, this option provides users with a very useful tool for data analysis.

Perform All the Permeation Calculations for Closed-Loop Permeation Testing with Discrete Sampling

First, the program converts the cumulative permeation from discrete sampling to that of a continuous sampling mode and then calculates all the permeation parameters in the same manner as for continuous sampling. To obtain the corresponding continuous sampling mode y values for the permeation curve of y (μg/cm^2) against x (min), the following equations are used based on whether the sample volume is or is not replaced.

$$CP_i = \frac{C_i V_i}{A} + \frac{\sum_{n=0}^{i-1} C_n V_s}{A} \quad \textbf{(11)}$$

where V_s is volume of discrete sample removed from the collection medium.

Closed-Loop Testing with Discrete Sampling and the Sample Volume Is Not Replaced

Assuming that V_i is the remaining medium volume at t_i, i.e.,

$$V_i = V_t - (i-1)V_s \quad \textbf{(12)}$$

Note that V_t is the total medium volume at the beginning of the permeation testing, i.e., t_0. Therefore, Eq. 11 can be unambiguously expressed as the following:

$$CP_i = \frac{C_i[V_t - (i-1)V_s]}{A} + \frac{\sum_{n=0}^{i-1} C_n V_s}{A} \quad \textbf{(13)}$$

Closed-Loop Testing with Discrete Sampling and the Sample Volume Is Replaced

Since sample volume is replaced, V_i is equal to V_t so Eq. 11 becomes:

$$CP_i = \frac{C_i V_t}{A} + \frac{\sum_{n=0}^{i-1} C_n V_s}{A} \quad \textbf{(14)}$$

Minimum Breakthrough Detection Time for ASTM D 6978 Standard

The ASTM D 6978-05 standard[9] defines the minimum breakthrough detection time as the time in minutes measured from the start of the test to the sampling time that immediately precedes the sampling time at which the permeation rate reaches 0.01 μg/cm^2/min.

Cumulative permeation from discrete sampling is first converted into a continuous sampling mode using Eqs. 11 and 14. Minimum breakthrough detection time is then calculated based on the procedure described earlier in the Standardized Breakthrough Time section but replacing Eq. 3 with Eq. 15:

$$2ax + b = 0.01 \quad \textbf{(15)}$$

DISCUSSION AND CONCLUSIONS

This computer program is an attempt to standardize and automate permeation testing data analysis. Previously, the data analysis was subject to the experimenter's interpretation of the relevant ISO and ASTM standards. For this program, specific algorithms and criteria were established for calculating the permeation parameters.

The precision of the computer program is a function of the program's code used to calculate the results. The computer program was written to calculate significant figures based on a combination of addition/subtraction and multiplication/division operations. The number of significant figures for each calculated value was determined based on the input values used in the calculations and was limited in precision to the least precise of these input values. As shown in Table II, there are no statistical difference between the computed results with those obtained by hand calculation on the same data for BDTs and SSPRs. Comparisons for other permeation parameters are not shown because hand calculations for some of them are either impossible (such as elapsed time at a given cumulative permeation) or impossible to obtain relatively accurate results (such as average permeation rate and cumulative permeation at a given elapsed time) for the open-loop permeation data files.

The accuracy of the calculated permeation parameters is dependent on the data quality of the permeation testing data file. The smaller the data sampling interval (i.e., higher frequency of sampling) in a data file, the more accurate the results will be. Fewer than 7 data points before the breakthrough may result in the inability to accurately determine the various breakthrough times. While ASTM F 739 method determines breakthrough times by visual estimation, the given approach to solving the polynomial regression equation is proposed for more accurate determinations.

In addition to the data sampling interval, several factors also influence accurately determining the various breakthrough times. These factors include the accuracy of analytical method used to generate the data points, the background noise level of the analyzer, and the use of the second-order polynomial equation ($y = ax^2 + bx + c$). A higher order of polynomial equation may be better, but it will result in a more complicated computer program and a longer computing time. Furthermore, using larger data sampling intervals for the permeation test may result in inaccurate determinations of cumulative permeation at a given elapsed time, elapsed time at a given cumulative permeation, and average permeation rate.

From a standardization point of view, this computer program uses data points ranging from 65% to 85% of the maximum concentration point for the linear regression to obtain the slope for a closed-loop system (Figure 4). Switching the range up or down (or enlarging the range) may improve the accuracy of the calculation for the slope. However, starting at a lower percentage may include data points from the transition region, resulting in a smaller slope, thus a smaller steady-state permeation rate, whereas extending the range to higher than 85% may also result in a smaller slope because the calibration curve usually bends down at high concentrations.

Alternatively, selecting multiple regions using data points ranging from 55% to 95% of the maximum concentration point and then using an algorithm for selection of the optimal steady-state region is under consideration in a revised version. The permeation testing should be continued until a steady state is fully established and there are enough data points for performing the linear regression calculations for the slope of the steady-state region.

Similarly, the values specified for determination of the breakthrough detection time, such as the slopes >2% of the largest calculated slope and $R^2 > 0.9$, may have an effect on the accuracy as well. Breakthrough detection time is very dependent on the detection limit of the analyzer used.[12] It is even dependent on the sampling flowrate for an-open loop test system.[14] For a higher sampling flowrate, a longer breakthrough detection time will be determined because the permeated chemical is diluted. Therefore, breakthrough detection time is less useful than standardized breakthrough time and normalized breakthrough time.

The program calculates the steady-state permeation rate for an open-loop test by averaging the concentration for the three highest data points. The accuracy depends on the variance of the three data points. However, the report file also includes seven SSPRs calculated with the seven highest concentrations so the user can remove any outliers.

This computer program will help researchers and industrial hygienists avoid labor-intensive hand calculations of the permeation parameters. From a standardization point of view, the computer program will prevent experimenter bias, thus ensuring identical permeation parameters will be obtained from a given permeation test data file. Protective clothing manufacturers worldwide will benefit since they must inform customers about the permeation parameters of their products in a consistent manner. The computer program will also help diagnostic laboratories and research centers involved in chemical protective clothing testing.

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