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Chemical Protective Clothing: A Comparison of Chemical Permeation Test Cells and Direct-Reading Instruments

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Chemical permeation of acetone through unsupported Neoprene using the ASTM cell and another commercially-available, but smaller, test cell was compared.* Also, different portable direct-reading instruments were used to determine breakthrough time and steady-state permeation. The breakthrough times between the two permeation cells and among different portable direct-reading instruments were not statistically different. However, steady-state permeation rates between the two cells using the same direct-reading instrument were statistically different. Chemical permeation test methods suitable for field evaluation of chemical protective clothing are discussed.

Introduction

The major routes of entry into the human body are inhalation, ingestion, and percutaneous absorption. In order to evaluate dermal exposure of potentially hazardous liquids, chemical permeation measurements are made. Two important parameters are obtained: (1) breakthrough time and (2) steady-state permeation rate. Breakthrough time serves as an estimation of the protection provided by chemical protective clothing. The steady-state permeation rate permits quantitation of the amount of hazardous liquid to which the skin is exposed through clothing. For more details, the reader is referred to a recent article in this journal.[1]

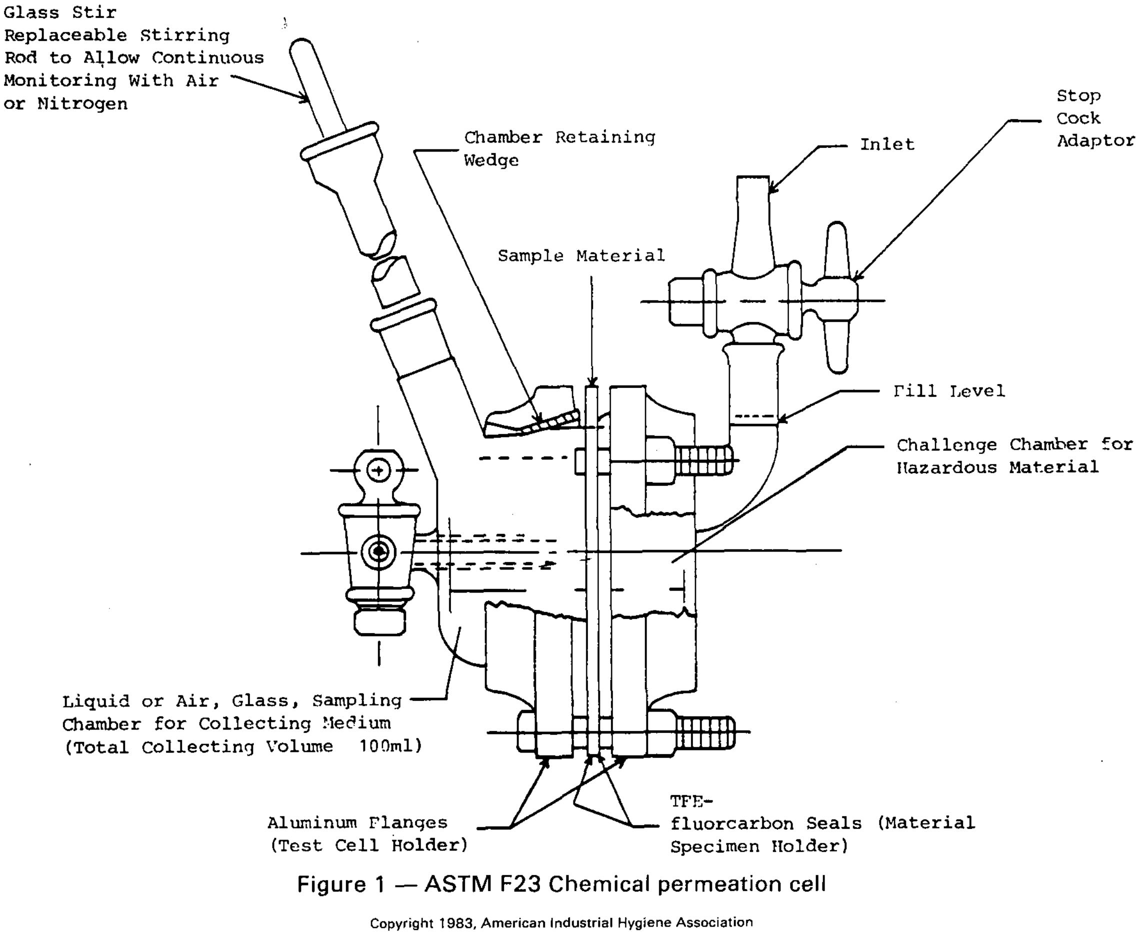

Figure 1 — ASTM F23 Chemical permeation cell

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*Mention of a company name or product does not constitute endorsement by the National Institute for Occupational Safety and Health (NIOSH).

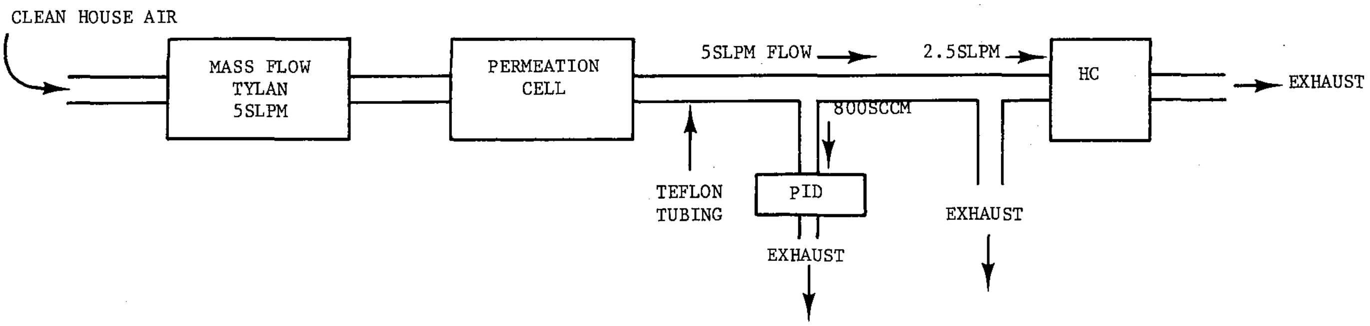

Figure 2 — Block diagram of experimental apparatus

The American Society for Testing and Materials (ASTM) has recently adopted a chemical permeation standard test method.(2) This standard method uses a commercially-available test cell.(3) Round robin validation testing has been successfully completed using acetone as the permeant against unsupported neoprene as the protective clothing material. Acetone is miscible with water and has a significant vapor pressure at room temperature; therefore, it can be collected either in water or in air. Acetone against Neoprene has a breakthrough time of less than 30 minutes and reaches steady-state permeation within the first hour of testing. Such times are optimum for this test procedure; hence, acetone against Neoprene was selected for our study. The scope of the present study was to compare the AMK cell,(4) a commercially-available chemical-permeation test cell, to the ASTM cell. The AMK cell is easy to assemble, compact, and requires less liquid permeant than the ASTM cell. However, the ASTM cell is more versatile since it permits liquid or gas collection. Also, a comparison of direct-reading instruments, most often found in an industrial hygiene laboratory, was conducted to determine breakthrough time and steady-state permeation.

Experimental

The ASTM cell (see Figure 1) was modified to expedite gas collection. The Teflon stopcocks were bored out to 1/8″. A circle of fabric approximately 7.8 cm in diameter was required for a test; with approximately 20 cm^2 of fabric exposed to the liquid permeant. About 45 mL of liquid permeant was required to fill the permeant side of the cell. Both aqueous and gaseous modes of collection were investigated. The aqueous collection medium was deionized-distilled water. Stirring was accomplished with a small peristaltic pump. Analysis was performed by a gas chromatograph using a Poropak Q column and a flame ionization detector (GC/FID). The gas collection medium was clean house air, and analysis by a Beckman Model 400 total Hydrocarbon Analyzer (HC) (a flame ionization detector) and an H-NU PI-101 portable Photoionization Detector (PID), side by side as shown in Figure 2.

An inexpensive, smaller, commercially-available chemical-permeation test cell suitable for gas collection only was purchased from AMK Glass Company (see Figure 3).(4) A circle of fabric approximately 2.5 cm in diameter was required; with about 5.3 cm^2 of fabric exposed to the liquid permeant. About 10 mL of liquid permeant was required to fill the permeant side of the cell. Analysis was by total hydrocarbon analyzer and a PID as described previously.

Materials

Fisher 99+% mole pure acetone; Edmont unsupported Neoprene gloves, #29-870 from one batch.

Procedure

The procedure is essentially that of the ASTM F739-81 chemical permeation method.(2) Samples were conditioned 24 hours at 22±2° C, 50±20% relative humidity. After condi-

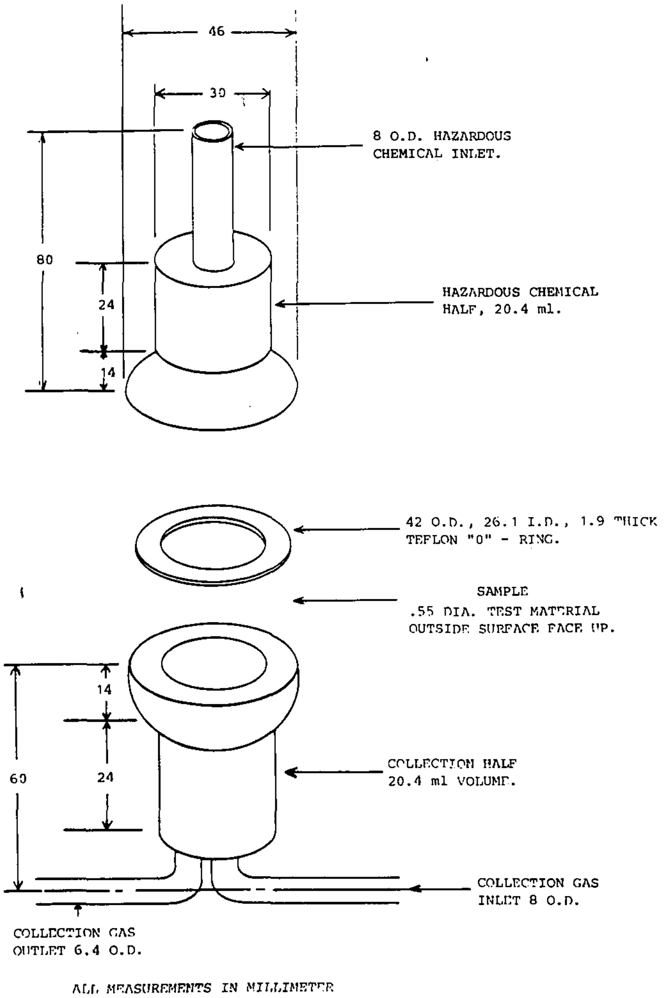

Figure 3 — AMK chemical permeation cell

tioning, 10 thickness measurements per specimen were recorded and the mean thickness computed. The test specimen was mounted in the cell, then gas or water was initiated, with stirring, into the collection side of the test cell. The flow rate for gas collection was 5 standard liters per minute (sLpm) unless noted otherwise. Also, for aqueous collection a peristaltic pump was started. Entrapped air bubbles in the line were monitored to ensure good mixing. Finally, the liquid permeant was added to the permeant side of the test cell and the stop watch turned on. The breakthrough time was recorded as the time when the first amount of acetone was detected by the direct-reading instrument. The lower limit of detection for the GC was 1 μg acetone/mL. The lower detection limit for the PID and HC was 1 ppm of acetone in air. The test continued until steady state was attained. For our system, steady-state permeation was reached within the first hour of the experiment. Four specimens in each category were run. The categories were:

Category	Cell	Collection	Detection	Flow Rate
1	ASTM	Water	GC / FID	-
2	ASTM	Air	HC / PID	5 sLpm
3	AMK	Air	HC / PID	5 sLpm
4	AMK	Air	PID	1 sLpm

For aqueous collection a 1.0-mL sample was withdrawn as a function of time (*e.g.*, one-minute intervals), immediately inserted into an auto sampler, and analyzed on the GC/FID. After each 1.0-mL sample was removed, 1.0 mL of distilled water was added to the collection side of the cell to maintain a constant volume. Therefore, the acetone concentrations, μg/mL, had to be concentration corrected. The equation for concentration correction is explained by ASTM.(2) The corrected concentration versus time was plotted on a Tektronix 4051 microcomputer. The slope of the linear portion of the graph or steady-state permeation was determined using linear regression analysis. The steady-state permeation rate(2) was calculated using the formula $J = SV/A$, where:

J is the steady-state permeation rate (mg/m^2/min),
S is the slope (mg/mL/min),
V is the volume of water in the collection side of the cell in mL, and
A is the exposed area of the specimen in m^2.

Where an open loop system is used for gas collection, analysis of the data has been described by Spence.(5,6) The advantage of such an approach is that samples are not withdrawn. The effluent stream, all or part, runs directly to the analyzer. No concentration correction is required. Steady-state permeation is easily determined since the recorder on the analyzer monitors the permeation events as they occur. Steady-state permeation is reached when the permeant concentration is constant versus time. Using a vapor pressure saturator method, each recorder-detector was calibrated daily as a unit, and the steady-state concentration interpolated from the graph. The formula $J = FC/A$ was used to obtain the steady-state permeation rate, where:

F is the flow rate through the permeation cell, mL/min,
C is the concentration at steady state, mg/mL, and
A is the exposed area of the specimen in m^2.

Results and Discussion

The data for each run and the mean with standard deviation for breakthrough times and steady-state permeation rates appear in Table I. Glove thickness differences were found to be statistically insignificant at the 95% confidence level. The data of categories 1 through 3 were analyzed statistically using a two-way unbalanced analysis of variance at the 95% confidence level via a Tektronix software package. The statistical analyses demonstrated that variation of breakthrough times between the two cells and among different direct-reading instruments was insignificant. Further, steady-state permeation rate differences between direct-reading instruments for the same cell were statistically insignificant. However, steady-state permeation rate differences between the two cells using the same analyzer were significant. Using Duncan's Multiple F-range tests,(7) the steady-state permeation rates for the AMK and ASTM cells using the total hydrocarbon analyzer were statistically different.

The steady-state permeation rate standard deviation of the mean for the hydrocarbon analyzer is twice that of the photoionization detector in each category, with the largest deviation in category three. Further, the mean steady-state permeation rate increases from category two to three. The largest mean steady-state permeation rate with the largest standard deviation is the hydrocarbon analyzer data of category three. This could be an artifact or a slight malfunction of the HC itself.

The paucity of data of category 4 did not permit complete statistical analysis. The important variable is the flow rate at 1 sLpm versus 5 sLpm of categories one to three. The lower flow rate causes a higher acetone-in-air concentration. Thus, breakthrough time occurs sooner. Also, the acetone concentration at steady state is so high that the photoionization detector reads off-scale.

The AMK cell in conjunction with a reliable, direct-reading instrument can be used as a field technique, but is limited due to the need for: (1) clean compressed air, (2) flow controllers, and (3) a vapor pressure saturator technique to calibrate. Further, the liquid permeant must be known, must have sufficient vapor pressure to volatilize, and must be analyzable by a direct-reading instrument. To overcome some limitations, a portable battery-powered pump with a rotameter might be used in lieu of compressed air and mass flow controllers.

Other direct-reading instruments can be used.(8) An alternate method to calibrate the direct-reading instrument by use of gas bags is discussed by Nelson.(9)

Conclusion

A relatively inexpensive and rapid field technique to determine preliminary breakthrough times and approximate steady-state permeation rates has been described. This tech-

TABLE I
Chemical Permeation Data

Cell, Collection Phase, Analyzer	Category	Run				Mean ± Standard Deviation
		1	2	3	4	
	1					
ASTM, WATER, GC	Mean Thickness (mm)	0.439	0.412	0.421	0.409	
	Breakthrough (minutes)	16.0	14.0	15.0	15.0	15.0 ± 0.8
	Steady-state permeation rate (mg/min/m^2)	2830	2550	2710	2850	2735 ± 138
	2					
ASTM, AIR, HC + PID	Mean Thickness (mm)	0.436	0.417	0.399	0.454	
	Breakthrough (minutes)^A	14.6	13.1	13.3	15.5	14.1 ± 1.1
	Steady-state permeation rate (HC) (mg/min/m^2)	2574	2628	2764	2466	2608 ± 124
	Steady-state permeation rate (PID) (mg/min/m^2)	2740	2764	2666	2666	2709 ± 50
	3					
AMK, AIR, HC + PID	Mean Thickness (mm)	0.439	0.447	0.431	0.429	
	Breakthrough (minutes)^A	14.4	13.9	13.2	16.0	14.4 ± 1.2
	Steady-state permeation rate (HC) (mg/min/m^2)	2907	3228	3412	2826	3093 ± 274
	Steady-state permeation rate (PID) (mg/min/m^2)	2684	2983	2798	2761	2806 ± 127
	4					
AMK, AIR, PID	Mean Thickness (mm)	0.460	0.440	0.440	0.440	
	Breakthrough (minutes)	10.6	10.3	12.3	12.3	11.4 ± 1.1

^{A}Breakthrough times for both analyzers were identical; therefore, only one breakthrough time is reported.

nique furnishes quantitative preliminary data and is intended to complement the quantitative laboratory permeation method adopted by the ASTM.

The AMK cell compares favorably to the ASTM cell for determination of breakthrough time. The AMK and ASTM cells do yield comparable steady-state permeation rates using a photoionization detector. The hydrocarbon analyzer must be evaluated further before recommendations can be made.

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