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# PROTECTIVE GLOVE MATERIAL PERMEATION BY ORGANIC SOLIDS*

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*A method has been developed for the determination of permeation characteristics of glove materials by organic solids. The system employs a stainless steel exposure cell and allows rapid and uniform contact of either solid disks or powders with minimal membrane bowing. A gas chromatograph equipped with a flame ionization detector was used for monitoring the permeation process, which provided detection limits of 0.9–1.2 ng for the organic solids evaluated. By using an automated system for instrument control and data collection, breakthrough times, steady-state times, and steady-state permeation rates have been determined for five common glove materials when exposed to nine organic solids.*

The barrier properties of gloves to chemical permeation are of growing interest to those responsible for protecting workers from contact with potentially hazardous chemicals.(1) In an industrial application, the need for protection against carcinogens is critical. Exposure to hazardous chemicals has been directly related to skin diseases and disorders that accounted for more than 40% of all reported occupational illnesses every year from 1972–1982.(2) To date, most experimental work with permeation of chemicals through glove materials has focused on organic solvents as the permeant.

In general, chemical permeation testing requires that a challenge material be placed in contact with a membrane. The substance permeates through the membrane and is then collected in an appropriate medium. The method currently used for the evaluation of the permeation of liquids is American Society for Testing and Materials (ASTM) F 739-85.(3) Method F 739-85 utilizes a glass sampling apparatus that is applicable to either liquids or gases and permits either open- or closed-loop testing. Henry and Schlatter(4) reported data for toluene and dimethyl formamide (DMF) versus three membranes by using this approach. Others have also applied this approach to study a wide variety of solvent/glove combinations. Forsberg and Faniadis(5) measured permeation characteristics of eight common glove materials against 20 single- or multicomponent organic solvents. Menke and Chelton(6) evaluated seven glove materials versus ethylene glycol dimethyl ether. Investigations in the authors' laboratory(7) reported data for eight commercially available glove materials versus six organic solvents by using a modification of ASTM Method F 739-85.

Although it is reasonable to assume that organic solids are capable of permeating through a glove material, to date there has been no reported method for the direct determination of their permeation characteristics beyond solution studies.(5,8) Foresberg and Faniadis(5) tested two glove materials, neoprene and polyvinyl alcohol (PVA), against a mixture of methylene chloride and phenol, with breakthrough times ranging from 18 min to over 4 hr. Vo-Dinh and White(8) focused on polynuclear aromatic compounds, such as H-Coal in fuel oil, which permeated through latex in 1 hr. These studies could not be used for direct evaluation of permeation characteristics of solids because of the effect the solvent would have on the membrane.

With a considerable amount of occupational illnesses attributed to organic solids used in industry,(2) there is a need for direct (solid) exposure data of protective clothing. The purpose of this study was twofold: (1) to show that organic solids can permeate through protective clothing materials when the solids are in direct contact with the materials and (2) to evaluate the experimental apparatus for conducting permeation tests with organic solids.

A method to evaluate organic solvent permeation characteristics(7) designed in the authors' laboratory has been modified for use with solids. The cell design was modified to accept solid samples without distortion of the membrane and utilizes a continuous, open-loop sampling technique. The solids and glove materials used in the permeation tests were chosen to be representative of those commonly used in the chemical industry.

## EXPERIMENTAL MATERIALS AND METHODS

Solids evaluated were *p*-dichlorobenzene, 4,6-dinitro-*o*-cresol, 2,4-dinitrotoluene, quinone, camphor, and naphthalene

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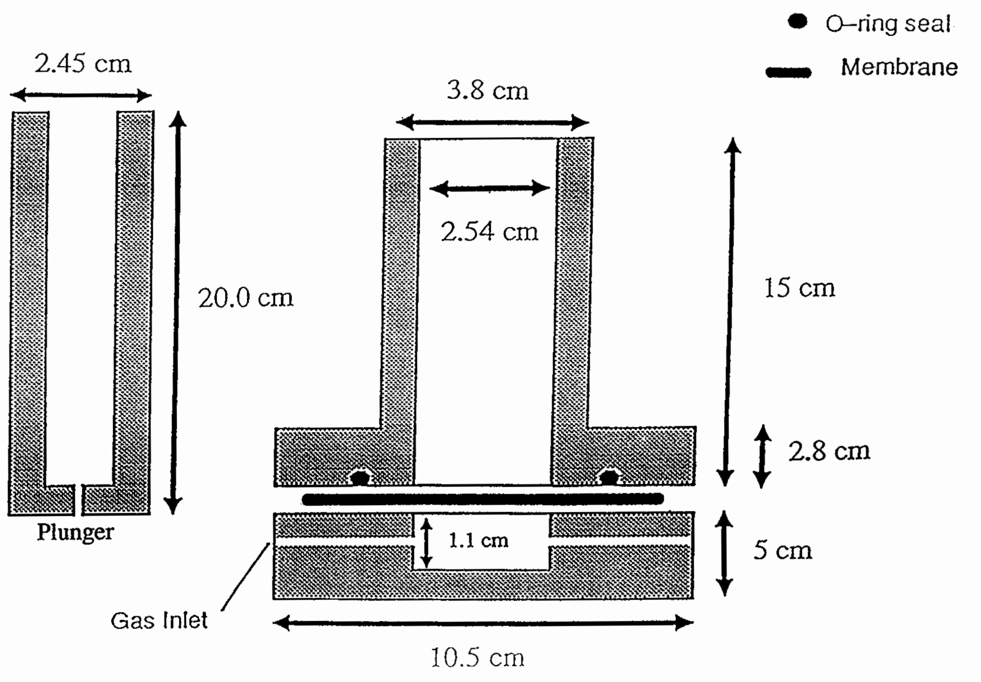

*FIGURE 1. Permeation cell*

(Aldrich Chemical Co., Milwaukee, Wis.); hydroquinone and phenol (Fisher Scientific, Pittsburgh, Pa.); and *p*-nitrotoluene (Kodak, Rochester, N.Y.). Each solid was certified American Chemical Society grade. These chemicals were chosen to represent a range of vapor pressures and polarities and because of their common use in industry. Disks of each chemical were prepared by compressing approximately 4 g of the material with a stainless steel press having an internal diameter of 2.5 cm. All solids were evaluated in disk form except for 2,4-dinitrotoluene and camphor, which were both evaluated in powder form because of poor disk quality. Gas chromatographic standards for each of the nine solids were prepared in methylene chloride at a concentration of 0.100–0.150 mg/mL for quantification.

Natural rubber (latex), PVC, urethane, nitrile, and neoprene membranes with a nominal thickness of 5 mil were evaluated as glove samples. Membranes of the glove materials were prepared by the Oak Rubber Co. (Ravenna, Ohio) by using a dip process. A Mylar® polyester film (film-92A, Dupont, Wilmington, Del.) with a thickness of 0.92 mil was also included in the test set. The thickness of each sample was determined as the arithmetic mean of four measurements taken at different locations on each test sample by using a micrometer.

A Hewlett Packard 5890A Series II gas chromatograph (GC) equipped with a flame ionization detector (FID) was utilized for all permeation experiments. A 10-port, high-temperature valve was used for flow control of the permeant effluent. A Hewlett Packard 5895A Pascal GC Chemstation provided automated valve control, data acquisition, and data editing. During data acquisition, the FID signal and the flow rate were monitored and stored. For quantification, a SPB-5 fused silica capillary column (30-m, 0.53-mm ID, 1.5-µm film thickness, Supelco, Bellefonte, Pa.) was used. All quantifications were performed isothermally at 100°C with a 250-µL gas sampling loop and a helium flow rate of 7.0 mL/min.

### Permeation Cell

The permeation cell, transfer line, and all fittings were constructed of 316 stainless steel to resist chemical degradation. The cell measurements are listed in Figure 1. A fluorocarbon O-ring (Parker Seals, Lexington, Ky.) was used to seal the membrane and cell. The long neck allowed for easier sample addition and for a weighted plunger to be placed over the sample to promote sample contact with the membrane. An external gage was used to monitor the back pressure of 0.2 to 0.5 psi from the system flow rate of 7.0 mL/min. The plunger also minimized membrane distortion by acting as a counterweight to hold the sample in position. Any reduction in pressure during the analysis was construed as a hole or distortion of the membrane. The cell's internal flow inlet was at a 90° angle to the outlet. The cell can also be inverted, allowing for experimentation in which the solid is not in direct contact with the membrane.

The experimental setup is shown in Figure 2. Helium flowed through the cell (7.0 mL/min), came into contact with the back of the membrane, and then exited through a heated transfer line (200 ± 10°C) to the sampling valve (250 ± 0.1°C).

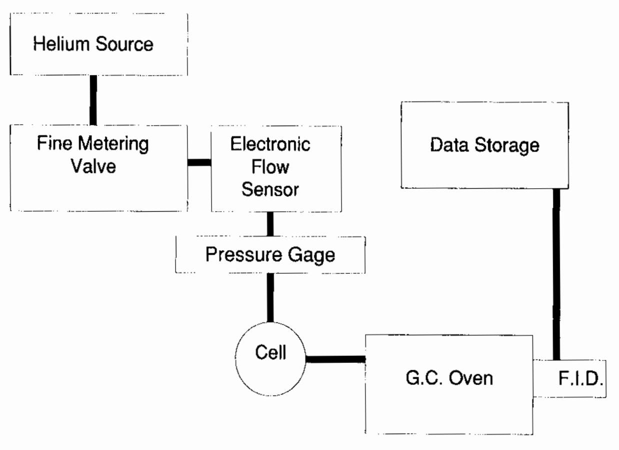

*FIGURE 2. Experimental setup*

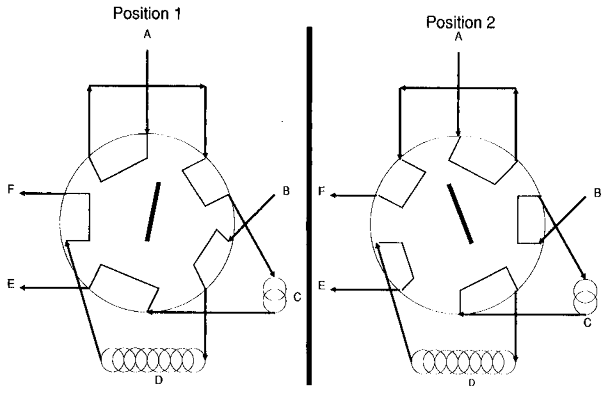

*FIGURE 3. GC sampling valve flow path: (A) permeation cell, (B) injection port, (C) sample loop, (D) analytical column, (E) FID, (F) vent*

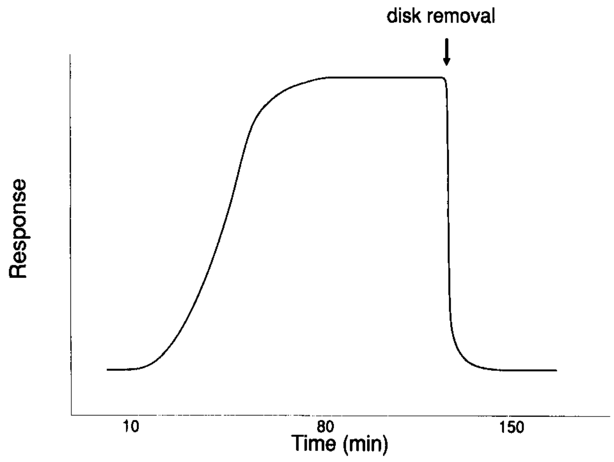

*FIGURE 4. Phenol versus latex*

On the basis of the valve's position, one of two flow paths could be selected (Figure 3). In Position 1, after leaving the cell (A), the flow travels initially through a 0.25-mL gas sampling loop (C) and then to the detector (E), allowing for continuous sampling. In this position, a second helium flow is maintained through the analytical column (D), which is vented (F). In Position 2, cell flow is vented (F) and the contents of the sample loop (C) are injected (B) onto the analytical column (D) for quantification.

### *Testing Procedure*

Each experiment was conducted in three stages at a relative humidity of 50 ± 10% and a temperature of 25 ± 1°C. Initially, the instrument was calibrated by making three standard injections with the valve in Position 2. The valve was then switched to Position 1 and the solid disk was placed in contact with the membrane. Response was monitored continuously until steady-state permeation rate was reached or 650 min had elapsed, which is the maximum run time for the software. At this point, the valve was switched to Position 2, which passed the contents of the sampling loop to the GC column for quantification. This final step was repeated, producing three replicate assays for determination of the steady-state permeation rate (SSPR).

## RESULTS AND DISCUSSION

### *System Evaluation*

Because of the low volatility of the chemicals investigated, the possibility of permeant condensation on the backside of the glove material or exposed cell surfaces exists. Observed permeation rates could also be artificially low because of saturation of the helium flow directed through the cell. Although the sampling valve and transfer lines were heated to at least 200°C to reduce any condensation, investigations were conducted to determine and then minimize these effects for each permeant versus the latex membrane. Initially, helium flow rates from 1–30 mL/min were evaluated for each latex/permeant pair after having reached steady-state permeation at a flow rate of 10 mL/min. At flow rates above 20 mL/min, distortion of the membrane was observed by a displacement of the plunger along with an increase in the response caused by increased membrane area and reduced thickness. At flows below 2 mL/min, a decrease in the response was observed for phenol and *p*-dichlorobenzene, indicating saturation of the helium or condensation within the cell. A flow rate of 7.0 mL/min was selected for subsequent studies to prevent saturation of the helium flow and provide maximum response for the quantitation portion of the analysis.

Additional testing for condensation was also conducted, in which the system was allowed to reach steady state at 7.0 mL/min helium flow for each latex/permeant pair, followed by removal of the disk. An immediate decrease in response, ultimately returning to baseline, was observed, as shown in Figure 4 for the phenol/latex pair. If condensation were occurring, the observed response would be maintained while the permeant evaporated from the system.

The system dead volume of 6.0 mL was found by replicate injections of methane (1.0 µL) through the membrane at known system flow. A peak width of 0.3 min indicated that there was adequate mixing and flushing of the effluent from the cell. All breakthrough time (BT) and steady-state permeation time (SST) values were corrected for the system dead volume by

$$BT = t - \frac{V_O}{FR} \quad (1)$$

where t represents the time of initial detection (min), $V_o$ is the system dead volume (mL), and FR is the flow rate (mL/min). The flow rate was continuously monitored during each experiment.

### *Glove/Solid Permeation Characteristics*

Permeation by organic solids was found to exhibit the same general permeation curve observed for liquids.(4) Figure 5 is a representative permeation curve, indicating the three deter-

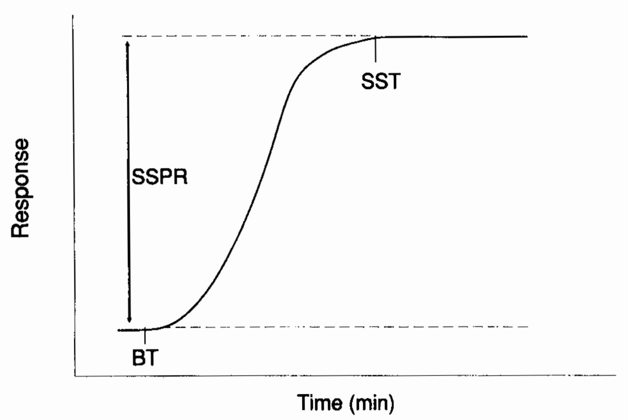

*FIGURE 5. Representative permeation curve (BT and SST labeled)*

mined values: breakthrough time, steady-state permeation time, and steady-state permeation rate. In practice, after correcting for system dead volume, BT is commonly determined as the time required for a permeant to produce a detectable response. The time for initial detection was determined as the point where detector response was at least three times the signal-to-noise ratio. Detection limits from 0.9 to 1.2 ng were observed for the materials investigated. A system was considered to have reached steady state if the change in response was less than 1%/min. For glove/permeant systems, SST was determined as the time required to reach 99% response. The steady-state permeation rate was calculated based on the average of three effluent assays for three membrane exposures$^{(9)}$ as:

$$\mathrm{SSPR} = \frac{\mathrm{MF}}{\mathrm{VA}} \qquad \textbf{(2)}$$

where SSPR is determined in μg/min/cm$^2$. M is the mass of permeant at steady state (μg), V is the volume of the sample loop (mL), F is the flow rate of the collecting medium (mL/min), and A is the area of exposed membrane in cm$^2$.

The BT, SST, and SSPR for each solid/membrane pair evaluated are presented in Tables I through III, respectively. The Mylar membrane exhibited no detectable permeation in 650 min by any of the target solids under continuous exposure and is not listed in the tables. For all compounds that exhibited breakthrough but did not reach steady state within 650 min, the permeation rate was calculated at the maximum run time.

Breakthrough was observed for the majority of the solid-membrane pairs tested with times ranging from 2 min to 8 hr for the five glove materials, although most breakthrough times were less than 10 min.

Breakthrough time variability ranged from 0.43% for *p*-nitrotoluene versus latex to 8.5% for dichlorobenzene versus nitrile with the precision ranging from 1% to 4.5% for the majority of solid/membrane pairs. Overall, nitrile and neoprene exhibited the longest BT values. Some of the solids exhibited a wide range of breakthrough times through the five glove materials tested; other solids exhibited narrower ranges of breakthrough times. For example, the breakthrough times for camphor ranged from 3.5 min through latex to 475 min through neoprene, whereas the breakthrough times for dichlorobenzene ranged only from 3.6 min through polyurethane to 9.5 min through nitrile. This allows the user to select a particular glove material according to the time of glove use.

Steady-state permeation rates of up to 8.1 μg/min/cm$^2$ were observed with nitrile and neoprene exhibiting lower SSPRs for the materials tested (Table III). The phenol versus

**TABLE I. Breakthrough Times for Solids (min)**

| | *Glove Membrane*$^A$ | | | | |
|---|---|---|---|---|---|
| *Compound (Solids)* | *Latex*$^B$ | *PVC*$^B$ | *Polyurethane*$^B$ | *Neoprene*$^B$ | *Nitrile*$^B$ |
| Benzoquinone | 8.5 ± 0.3 | 9.6 ± 0.4 | 18.5 ± 0.2 | 45.0 ± 1.7 | ND$^C$ |
| Naphthalene | 4.3 ± 0.2 | 4.7 ± 0.2 | 11.5 ± 0.2 | 19.0 ± 1.2 | 41.0 ± 2.7 |
| Dichlorobenzene | 5.2 ± 0.2 | 5.4 ± 0.2 | 3.6 ± 0.1 | 6.4 ± 0.5 | 9.5 ± 0.8 |
| *p*-Nitrotoluene | 7.0 ± 0.1 | 8.1 ± 0.1 | 138.0 ± 2.9 | 14.2 ± 0.9 | ND |
| Camphor | 3.5 ± 0.2 | 4.8 ± 0.1 | ND | 475.0 ± 8.4 | 420.0 ± 8.8 |
| Phenol | 10.4 ± 0.4 | 10.6 ± 0.4 | 28.0 ± 1.2 | 21.0 ± 0.7 | 185.0 ± 7.9 |
| Hydroquinone | 2.4 ± 0.1 | 3.5 ± 0.1 | 11.0 ± 0.6 | 31.5 ± 1.2 | 39.0 ± 1.6 |
| 4,6-Dinitro-*o*-cresol | 4.5 ± 0.2 | 5.2 ± 0.2 | ND | ND | ND |
| 2,4-Dinitrotoluene | 6.1 ± 0.4 | 6.8 ± 0.1 | 7.1 ± 0.3 | 35.0 ± 1.5 | 142.0 ± 2.6 |

$^A$Nominal membrane thickness 5 mil.

$^B$95% confidence limits of three experiments.

$^C$ND = no permeation detected.

**TABLE II. Steady-State Times (min)**

| *Compound* | *Latex*$^A$ | *PVC*$^A$ | *Polyurethane*$^A$ | *Neoprene*$^A$ | *Nitrile*$^A$ |
|---|---|---|---|---|---|
| Benzoquinone | 35 ± 3 | 59 ± 6 | 170 ± 10 | 300 ± 13 | T$^B$ |
| Naphthalene | 151 ± 9 | 160 ± 9 | 104 ± 8 | 205 ± 9 | T |
| Dichlorobenzene | 50 ± 4 | 152 ± 8 | 147 ± 9 | 165 ± 10 | 131 ± 8 |
| *p*-Nitrotoluene | 120 ± 10 | 146 ± 10 | T | T | T |
| Camphor | 44 ± 3 | 75 ± 8 | T | T | T |
| Phenol | 80 ± 5 | 103 ± 5 | 122 ± 7 | 135 ± 8 | T |
| Hydroquinone | 260 ± 10 | 300 ± 13 | 292 ± 22 | 250 ± 9 | T |
| 4,6-Dinitro-*o*-cresol | 110 ± 5 | 133 ± 10 | T | T | T |
| 2,4-Dinitrotoluene | 210 ± 8 | 239 ± 17 | 241 ± 17 | 360 ± 11 | 600 ± 28 |

$^A$95% confidence limits of three experiments.

$^B$T = Steady state was not reached in 650 min.

**TABLE III. Steady-State Permeation Rates for Solids (μg/min/cm$^2$)**

| *Compound (Solid)* | *Latex*$^A$ | *PVC*$^A$ | *Polyurethane*$^A$ | *Neoprene*$^A$ | *Nitrile*$^A$ |
|---|---|---|---|---|---|
| Benzoquinone | $1.60 \pm 8\times10^{-2}$ | $4.46 \pm 3\times10^{-2}$ | $1.73 \pm 1.4\times10^{-1}$ | $3.21\times10^{-1} \pm 1.9\times10^{-2}$ | ND$^B$ |
| Naphthalene | $1.13 \pm 7\times10^{-2}$ | $8.47\times10^{-1} \pm 7.1\times10^{-2}$ | $1.14 \pm 8\times10^{-2}$ | $1.79\times10^{-1} \pm 1.1\times10^{-2}$ | $3.27\times10^{-1} \pm 1.6\times10^{-2}$ |
| Dichlorobenzene | $8.07 \pm 3.1\times10^{-1}$ | $1.14 \pm 5\times10^{-2}$ | $5.00 \pm 2.1\times10^{-1}$ | $3.16\times10^{-1} \pm 2.2\times10^{-2}$ | $3.26\times10^{-1} \pm 1.3\times10^{-2}$ |
| *p*-Nitrotoluene | $2.04 \pm 1\times10^{-2}$ | $1.48\times10^{-1} \pm 1.0\times10^{-2}$ | $1.65 \pm 8\times10^{-2}$ | $5.03\times10^{-1} \pm 3.3\times10^{-2}$ | ND |
| Camphor | $1.11\times10^{-1} \pm 4\times10^{-3}$ | $4.03\times10^{-1} \pm 1.0\times10^{-2}$ | ND | $3.67\times10^{-1} \pm 1.79\times10^{-2}$ | $2.33\times10^{-1} \pm 1.01\times10^{-2}$ |
| Phenol | $1.51 \pm 9\times10^{-2}$ | S$^C$ | $2.33 \pm 1.2\times10^{-1}$ | $2.01\times10^{-1} \pm 1.4\times10^{-2}$ | $1.14 \pm 4\times10^{-2}$ |
| Hydroquinone | $1.52\times10^{-2} \pm 8\times10^{-4}$ | $9.58\times10^{-3} \pm 4.7\times10^{-4}$ | $1.31\times10^{-2} \pm 6\times10^{-4}$ | $9.05\times10^{-3} \pm 7.7\times10^{-4}$ | $8.22\times10^{-3} \pm 3.7\times10^{-4}$ |
| 4,6-Dinitro-*o*-cresol | $1.38\times10^{-3} \pm 7.7\times10^{-4}$ | $6.39\times10^{-3} \pm 3.7\times10^{-4}$ | ND | ND | ND |
| 2,4-Dinitrotoluene | $7.03 \pm 2.8\times10^{-1}$ | $5.79\times10^{-1} \pm 1.7\times10^{-2}$ | $8.13 \pm 6.3\times10^{-1}$ | $3.47\times10^{-1} \pm 2.0\times10^{-2}$ | $2.51\times10^{-1} \pm 1.4\times10^{-2}$ |

[A] 95% confidence limits of three experiments.

[B] ND = no permeation detected.

[C] S = membrane solubilized.

PVC exposure exhibited some "melting" or solubilizing of the membrane during the exposure. In the case of 4,6-dinitro-*o*-cresol, three of the materials exhibit no response, but for latex and PVC, steady-state permeation rates are slightly above the detection limit. The precision of the SSPR varies from 2% to 8.8%. The poorer precision can be attributed to the neoprene membranes, ranging from 6.0% to 8.8%, in which many of the membrane-solid pairs did not reach SST and had to be calculated at the default time of 650 min.

Some organic solids and glove pairs show a consistent deviation from the representative permeation curve. These trends were most evident for camphor/neoprene and hydroquinone/latex (Figure 6). With camphor/neoprene, an initial rapid increase is observed, followed by a slower, near-linear rise in response. Other workers have reported this phenomenon for liquid/membrane permeation and attributed it to membrane swelling during exposure.(6) For hydroquinone/latex, there is a steady decrease in response after reaching a maximum at 39.5 min. This phenomenon has been documented in samples that exhibit some physical deformation or degradation by chemical reaction of the membrane.(10) Similarly, these phenomena are also observed in permeation of solids and can be attributed to deformation or degradation of the test membrane. When either of these profiles was observed, the steady-state permeation rate for this solid/membrane pair was calculated at the default time of 650 min.

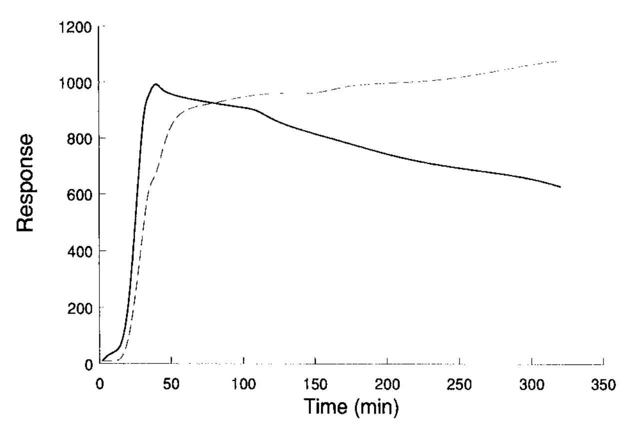

*FIGURE 6. Permeation curves: (——) hydroquinone versus latex; (- - -) camphor versus neoprene*

### *Effect of Direct Contact versus Vapor Phase*

Both vapor phase transfer and direct dissolution of a permeant can represent significant modes for initiating the permeation process. Studies were conducted to determine differences between these two modes for both phenol and *p*-dichlorobenzene. The cell was inverted, allowing the disk to be placed at known distances from the membrane, and the BT and SSPR were determined (Table IV). SSPR values were, overall, slightly higher than they were when the solids were in direct contact with the membranes. This can be attributed to each membrane exhibiting a bowing effect because of the lack of support nor-

**TABLE IV. BT for Direct Contact and Vapor Phase Transfer**

| | | *Phenol* | | *p-Dichlorobenzene* | |
|---|---|---|---|---|---|
| *Material* | *Distance (cm)* | *BT (min)* | *SSPR (μg/min/cm$^2$)* | *BT (min)* | *SSPR (μg/min/cm$^2$)* |
| Latex | 0 | 10.4 | 1.51 | 5.2 | 8.07 |
| | 1 | 19.5 | 1.69 | 8.0 | 8.87 |
| PVC | 0 | 10.6 | S$^A$ | 5.4 | 1.14 |
| | 1 | 15.0 | 0.371 | 5.8 | 1.15 |
| Nitrile | 0 | 185 | 1.14 | 9.5 | 0.327 |
| | 1 | 320 | 1.17 | ND$^B$ | |
| Urethane | 0 | 28.1 | 2.33 | 3.6 | 4.99 |
| | 1 | 54.9 | 4.98 | 10.5 | 5.90 |
| | 2 | 64.4 | | 11.7 | |
| | 3 | 71.4 | | 12.0 | |
| Neoprene | 0 | 21.0 | 0.201 | 6.4 | 0.316 |
| | 1 | 60.4 | | 9.7 | 0.499 |
| | 2 | 69.0 | | 18.5 | |
| | 3 | 79.2 | | 22.2 | |

[A] S = membrane solubilized.

[B] ND = not detected.

mally provided by the disk and plunger. The phenol versus PVC exposure exhibited some melting or solubilizing of the membrane during the exposure.

In the case of phenol versus neoprene and *p*-dichlorobenzene versus polyurethane, the experiment was completed at different distances to determine if the effect was caused by variations in concentration or in the time required to saturate the vapor phase. Breakthrough times increased when there was no contact. This indicates that the initial dissolution of the permeant greatly reduces the BT.

## CONCLUSIONS

By using a modification of a previously described method for the characterization of permeation by organic solvents, it has been demonstrated that organic solids permeate through common glove materials. SSPR values are low for solids, most likely because of lower vapor pressures. A significant variation in BT and SSPR between direct contact and vapor phase permeation demonstrates that permeation is not totally a vapor pressure phenomenon. The work reported shows that although some glove materials can offer adequate protection for specific chemical species, individual permeant evaluations may be required.

## ACKNOWLEDGMENT

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