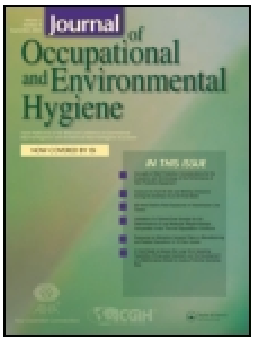

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Application of Colorimetric Indicators and Thermo-Hand Method to Determine Base Permeation Through Chemical Protective Gloves

Evanly Vo

National Institute for Occupational Safety and Health, National Personal Protective Technology Laboratory,
Pittsburgh, Pennsylvania

The aim of this study was to assess the use of colorimetric indicator pads and the thermo-hand method for detection of inorganic/organic base permeation of chemical protective gloves under simulated in-use conditions. Breakthrough times for four types of gloves were determined based on the color change of pads and ranged from 3 to 10 min for butylamine, from 4 min to >4 hours for diisopropylamine, from 6 min to >4 hours for triethylamine, and >4 hours for sodium hydroxide. Quantification was performed for butylamine, diisopropylamine, and triethylamine by gas chromatography following solvent desorption. These chemicals exhibited >99% adsorption on the pads at spiking levels of 1.08–1.11 :g for each base. The recovery for the system was calculated for each chemical, with results ranging from 50–74% (RSD $\leq$5%) for these bases over the spiking range 0.22–1.11 µg. The quantitative mass of the bases on the pads at the time of breakthrough detection ranged from 118–121, 117–120, and 109–116 µg/cm^2 for butylamine, diisopropylamine, and triethylamine, respectively. The thermo-hand test method and base indicators together should find utility in detecting, collecting, and quantitatively analyzing base permeation samples under simulated in-use conditions.

Keywords breakthrough times, chemical protective gloves, colorimetric indicators, quantitative analysis, safety, thermo-hand method

Address correspondence to: Evanly Vo, National Institute for Occupational Safety and Health, National Personal Protective Technology Laboratory, 626 Cochrans Mill Road, Pittsburgh, PA 15236; e-mail: eav8@cdc.gov.

Inorganic and organic bases are used widely in chemical synthesis and in the making of soap, paper, petroleum products, and metal cleaning agents.(1,2) These chemicals are capable of penetrating the skin;(1,3) thus, dermal exposure to neat or aqueous solutions of bases represents a potential route of chemical exposure in humans. These chemicals are used widely and represent a sufficient hazard by the inhalation route that guidelines for limiting exposure have been developed.(4,5) Butylamine is used as a chemical intermediate in the making of certain pharmaceuticals, dyestuffs, rubber chemicals, emulsifying agents, insecticides, and synthetic tanning agents.(1) Diisopropylamine is also used as a chemical intermediate.(3) Triethylamine is used in the manufacture of waterproofing agents, corrosion inhibitors, and propellants.(6) Sodium hydroxide (NaOH) is used in the manufacture of rayon, mercerized cotton, soap, paper, aluminum, petroleum products, metal cleaning agents, during electrolytic extraction of zinc, and as an oxide coating.(2)

Although substitution and administrative and engineering controls are considered first, chemical resistant gloves and protective clothing are routinely employed to prevent skin exposure of workers to hazardous chemicals in the workplace.(7) Chemical resistant gloves are typically selected based on performance characteristics determined during laboratory permeation testing, following the procedure known as the ASTM F-739 method, or a modification of this method.(8) This method provides breakthrough detection times and permeation rates for volatile chemicals so that one can choose the most appropriate glove type(s) for use in a particular application. However, many workplace variables influence glove performance, including flexing, increased temperature, mixtures of two or more chemicals, and differences between glove manufacturers and production lots.(9–11) Limited laboratory test data cannot address all these variables.(9–12)

Colorimetric indicator assays and spot tests for inorganic and organic bases have been developed and used successfully in a variety of biomarkers, enzyme assays, and identification of unknown chemicals.(13–15) Colorimetric indicator pads produce a visible color change when contamination occurs on the pads. Therefore, if workers wear the appropriate indicator pads they are able to detect contamination sooner and learn to protect themselves from increased skin exposure to toxic chemicals by changing their gloves. Recently, several laboratory studies on indicator development and performance have been published that might show promise for use of colorimetric indicators.(16–18) In addition to colorimetric indicators, dermal monitoring to simulate workplace conditions could be used to evaluate glove performance under in-use conditions and could bridge the gap between laboratory data and actual workplace performance. Herein, we report test results for an indicator pad for base permeation of chemical protective gloves under simulated in-use conditions using the thermo-hand test method.(19)

MATERIALS AND METHODS

Chemicals, Selected Gloves, Indicator Pads, and Other Apparatuses

Unless otherwise noted, all inorganic and organic bases used for this study were obtained from a commercial supplier (Aldrich Chemical, Milwaukee, Wis.). All organic bases were used as neat standard chemicals; NaOH was used as an aqueous solution (10 mL solution in water). The four types of gloves selected for this study are identified in Table I. Acid/Base indicator pads (rectangular-pad area: 2.0 cm × 2.2 cm; its thickness: 0.1 cm; containing thymol blue reagent) were purchased from Colormetric Laboratories (Des Plaines, Ill.). An Ames (Waltham, Mass.), 214-10 micrometer with a pressure foot of 1 cm was used to determine the thickness of each glove. A CEM (Matthews, N.C.) MES-100 microwave-extraction (ME) system was used to extract bases from pads. Savillex vials (3 mL vials; these vials could be used under heat condition while the vials were closed) were purchased from CEM. A Perkin-Elmer (Norwalk, Conn.) gas chromatographic system, which consisted of a PE Nelson Model 1022 Personal Integrator and an AutoSystem gas chromatograph with flame ionization detector, was used to analyze chemicals.

The thermo-hand apparatus (Figure 1), which consisted of a temperature-controlled incubator (model 550D, Fisher Scientific, Pittsburgh, Pa.), a multimixer shaker (model 4605, Lab-Line Instruments Inc., Melrose, Ill.), a 2.5 cm diameter glass cell and glass cylinder (AMK Glass Company, Vineland, N.J.), was used to determine breakthrough detection time for the glove materials. This apparatus simulates human hand temperature and hand movement.

Evaluation of Sorption Properties of Pads

Spiking Pads

New pads were removed from sealed packages and the adhesive areas removed with forceps. Known mass of each base (0.22–1.11 μg) was added directly to the surface of the pads. The pads were then inserted into 300 μL of distilled water (used as an extraction solvent for the entire base study) in 3-mL Savillex vials and the vials were closed with the vial caps. These vials were then used for the extraction process and GC analysis. Blank determinations were performed using the same procedure but without exposing the pads to any base chemical.

The ME Process and GC Analysis

The ME-GC procedure was run according to the method described by Vo et al.(20) as follows. The closed vials were immersed in 25 mL of water in extraction vessels, then the vessels were placed in the MES-100 system and heated for 5 min at 100°C, 70 psi, and 60% power. To obtain maximum recovery of these chemicals without evaporation loss, the extracted solutions were allowed to cool to room temperature for 15 min before opening the vial caps for GC analysis. The optimum GC conditions were as follows: column, 3.05 m × 3.18 mm i.d., 3% SP-1500 on 80-120-mesh Carbopack B (Supelco, Bellefonte, Pa.); helium flow rate, 25 mL min^{-1}; oven temperature, 210°C; column temperature, 210°C; temperature of the flame ionization detector, 240°C; data collection time, 0–20 min. Volumes of 5 μL of extracted samples of spiked bases were injected into the GC column using a syringe.

Efficiency of Adsorption During Base Spiking

To assess whether complete adsorption of each base is accomplished during spiking on pads, two Savillex vials were used. In the first vial, 1.5 μL (1.08–1.11 μg) of each base was added to a pad and the vial was closed with the vial cap. The vial was left at room temperature for 15 min before the pad was inserted in 300 μL of distilled water in the second vial. Then, 300 μL of distilled water was added directly to the first vial to extract excess base that did not adsorb on the spiked pad. These vials were then used for the extraction process and GC analysis.

Base Chemical Recovery

Standard curves were generated using the same spiked base procedure but without using pads. Known mass of each base (0.22–1.11 μg) was added directly to 300 μL of distilled water in the vials. These vials were then used in the extraction process and GC analysis. The recovery was calculated as: (100% [spiked peak area/standard peak area]).

Glove Thickness Test

The thickness test was performed at three positions on each glove: the palm, the entire middle finger, and the cuff (3 cm from the open end), as these represent the areas of highest contact and glove abrasion, which enhances chemical penetration.

TABLE I. Glove Materials Used in Base Permeation Study

Glove Brand Name	Glove Material	Model or Lot #	Manufacturer
Guardian (butyl-Std)	Butyl rubber (powder-free)	Model # 1-196	Guardian Company (Willard, Ohio)
Neoprene	Neoprene (powder-free)	Model # 29-845	Ansell-Edmont (Coshocton, Ohio)
Powdered nitrile (Box Ambi)	Nitrile rubber (powdered)	Model # 40101-410	VWR Scientific Products (West Chester, Pa.)
Sol-Vex	Nitrile rubber (powder-free)	Model # 37-145	Ansell-Edmont (Coshocton, Ohio)

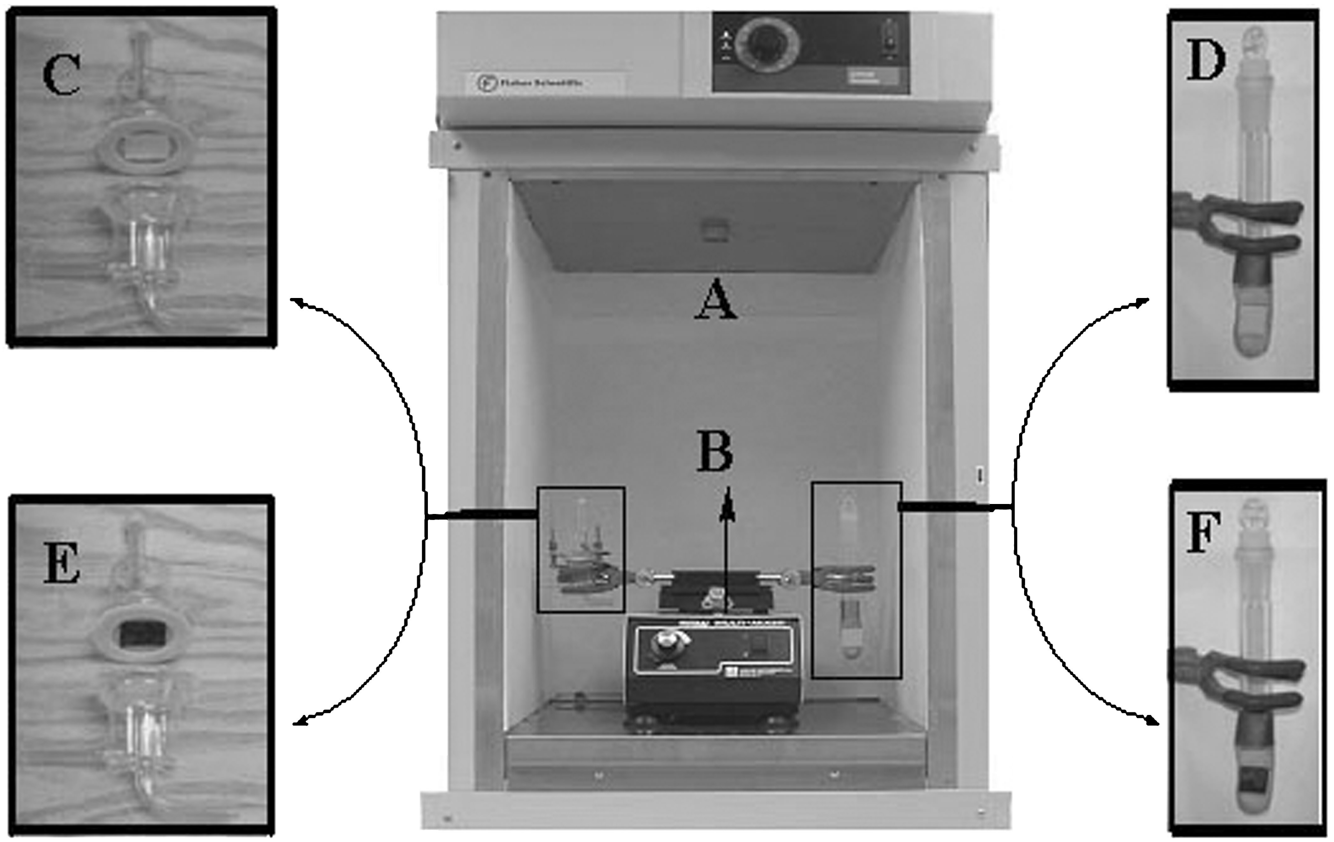

FIGURE 1. Chemical permeation test configuration under simulated in-use conditions: A temperature-controlled incubator (A) to simulate the hand's skin temperature at 34°C. A multimixer shaker to simulate hand movements (B). The 2.5 cm diameter permeation cell (C) used for breakthrough time testing for the palm and cuff of gloves. A glass cylinder used for breakthrough time testing for the finger of gloves (D). Base indicator pads with a yellow color (C and D); on exposure to bases, these pads changed color to blue (E and F).

Five thickness measurements for each position on the glove were recorded and the mean thickness and variation were calculated. All measurements were made to the nearest ±0.01 mm.

Glove Breakthrough Time Determination

Breakthrough times of glove sections from the palm and the cuff were determined according to the method described by Vo et al.(19) The 2.5-cm permeation cell is divided into a "challenge side" that contains the chemicals, and a "collection side" that contains an indicator pad (Figure1C). A glove membrane (sections from the palm and the cuff of gloves) separated the two sides of the permeation cell, with the outer surface toward the challenge side of the permeation cell. A pad was attached to the inner surface of the glove section with clear plastic tape (the parafilm was placed under the plastic tape so the pad could be easily removed from the glove for quantification; results from this study showed that using parafilm under the plastic tape did not affect the breakthrough detection time).

To simulate the hand's skin temperature under in-use conditions, the controlled incubator was used to conduct the experiment at 34°C (Figure 1A). Fifteen mL of each base was injected into the challenge side of the cell using a 30-mL glass syringe, and a multimixer shaker (Figure 1B) used to simulate hand movements(19) was operated (the glove materials in the thermo-hand method are simply shaken without stretching glove materials). Then, a timer was immediately started. Permeation of each base through glove sections from the palm and the cuff was detected by the change in color of the pads. Breakthrough time was recorded, and the pads were immediately removed from the glove and inserted into 300 μL of distilled water in 3-mL Savillex vials for the extraction process.

Breakthrough detection times of the finger portion of gloves were also determined under simulated in-use conditions according to the method described by Vo et al.(19) A finger of the gloves was turned inside out and a pad was attached to the finger. The glove finger was then secured to a glass cylinder (2 cm × 15 cm) with duct tape at the open end of the glove finger as the attachment area. The glass cylinder served as a fill tube (Figure 1D). A 10-mL volume of each base was injected into the glass cylinder, and a timer was immediately started. Permeation of each base through the glove finger was detected by the change in color of the pads. Breakthrough time was recorded, and the pads were immediately removed from the glove and inserted into 300 μL of distilled water in the vials for the extraction process.

Quantitative Determination of Bases

The closed vials of each base were extracted, and 5 μL of extracted samples were subjected to GC analysis as described above. The quantitative mass of each base was determined against a known mass of each spiked base based on its linear equation and the degree of each base recovery.

RESULTS

Evaluation of Sorption Properties of Pads

Spiking Pads

It was shown that a new base indicator pad is very sensitive to the detection of the presence of both organic and inorganic bases with small amounts of 0.22 μg of each base applied to the pads. Bases changed pH of the indicator (thymol blue) to contribute the color change from yellow to dark blue. None of the blanks (unexposed pads in the extraction solvent) produced chromatograms containing peaks corresponding to these bases used in this study. The GC retention times for the bases obtained under these conditions are given in Table II.

Efficiency of Adsorption as Determined by Spiking

Base quantity that did not adsorb on the spiked pads in the first vial was low, being <1% for all bases (the area peaks found in vial #2 were 0.15%, 0.12%, and 0.18% of the area peaks found in vial #1 for butylamine, diisopropylamine, and triethylamine, respectively). Generally, all bases exhibited >99% adsorption on the pads at spiking levels of 1.08–1.11 μg for each base.

Calibrations of Pads and Base Recovery

The relationship between signals (peak area) on the gas chromatograms and spiked mass levels of 0.22–1.11 μg (Table II) of each base applied to pads was analyzed using Microsoft Excel software. The best linear correlation for bases in each solution was obtained over the range 0.22–1.11 μg (n = 4). The best straight-line fit from linear analysis of the data is also obtained (diisopropylamine: $y_d = 18.4x_d - 1.68$, $R^2 = 0.987$; butylamine: $y_b = 28.6x_b - 1.65$, $R^2 = 0.995$; and triethylamine: $y_t = 38.2x_t - 1.57$, $R^2 = 0.997$). For experiments performed with extraction process and GC measurements, recoveries from 50% to 74% (RSD $\leq$%) were obtained over the range 0.22–1.11 μg of each base applied to the pads (Table II).

Glove Thickness Test

Tables III, IV, and V summarize the results obtained for the mean thickness of four thickness measurements for each type of gloves.

Glove Breakthrough Time Determination

Breakthrough time of triethylamine through the gloves was detected by the change in color of the pads before the IR analyzer detector responded;(16) therefore, the determination of breakthrough time of each base through the gloves is highly dependent on the sensitivity of the indicator detection method. Breakthrough time of each base through the gloves was detected by the change in color of the pads and showed in Tables III, IV, and V. Relatively slow permeation of diisopropylamine and triethylamine was observed through the nitrile glove material, while rapid permeation of the butyl rubber glove material was observed (Table III, IV, and V). Although slower permeation of diisopropylamine and triethylamine through the nitrile rubber material was observed, butylamine permeated all the materials in 10 min or less. For NaOH, no permeation was observed for all glove materials after 4 hours.

The breakthrough time data of triethylamine obtained under the thermo-hand method (34°C) were compared with breakthrough time data obtained at room temperature (22 ± 1°C;

TABLE II. Recoveries of Organic Bases

Bases (Retention Time)	Volume of Bases Applied to Pad (μL)	Mass of Bases Applied to Pad (ng)	Mean of Spiked Peak Areas (×100)	Recovery (%) (mean ± SD, n = 3)
Butylamine (2.18 min)	0.3	222	5.14	57 ± 3
	0.6	444	14.9	62 ± 2
	0.9	666	24.3	67 ± 3
	1.2	888	32.9	71 ± 3
	1.5	1110	41.5	74 ± 2
Diisopropylamine (2.29 min)	0.3	217	1.96	52 ± 2
	0.6	433	8.81	58 ± 2
	0.9	649	15.1	63 ± 3
	1.2	866	20.5	67 ± 3
	1.5	1083	26.2	71 ± 3
Triethylamine (2.49 min)	0.3	218	8.14	50 ± 2
	0.6	436	20.8	56 ± 2
	0.9	653	33.1	62 ± 3
	1.2	871	44.7	65 ± 3
	1.5	1089	55.9	68 ± 3

Notes: 0.3–1.5 μL of each base added pads, n = 4; 300 μL of distilled water was used to extract these bases; 5 μL of the extracted solutions was analyzed by gas chromotography.

TABLE III. Results of Glove Thickness, Breakthrough Time, and the Quantitative Mass Data of Test Butylamine on the Indicator Pads at the Breakthrough Time

Glove Type	Thickness[A] (Finger) (mm)	Breakthrough[B] Time of Butylamine (Finger)	Thickness[A] (Palm and Cuff) (mm)	Breakthrough[C] Tme of Butylamine (Palm and Cuff)	Mass of Butylamine[D] (μg/cm^2) (mean ± SD, n = 3)
Guardian (butyl-Std)	0.24 ± 0.01	3 ± 1 min	0.23 ± 0.01	3 ± 1 min	114 ± 6
Neoprene	1.25 ± 0.01	10 ± 1 min	1.24 ± 0.01	9 ± 2 min	112 ± 5
Powdered nitrile (Box Ambi)	0.12 ± 0.01	2 ± 1 min	0.11 ± 0.01	2 ± 1 min	115 ± 6
Sol-Vex	0.38 ± 0.01	9 ± 1 min	0.36 ± 0.01	8 ± 1 min	112 ± 4

[A] Mean thickness ± SD (n = 4).
[B] Breakthrough time ± SD (n = 4) detected by the change in color of indicators (finger permeation testing procedure).
[C] Breakthrough time ± SD (n = 4) detected by the color change of indicators (the palm-cuff permeation testing).
[D] The mean of mass permeated at all three locations: finger, palm, and cuff of gloves.

TABLE IV. Results of Glove Thickness, Breakthrough Time, and the Quantitative Mass Data of Test Diisopropylamine on the Indicator Pads at the Breakthrough Time

Glove Type	Thickness[A] (Finger) (mm)	Breakthrough[B] Time of Diisopropylamine (Finger)	Thickness[A] (Palm and Cuff) (mm)	Breakthrough[C] Time of Diisopropylamine (Palm and Cuff)	Mass of Diisopropylamine[D] (μg/cm^2) (mean ± SD, n = 3)
Guardian (butyl-Std)	0.24 ± 0.01	4 ± 1 min	0.23 ± 0.01	4 ± 1 min	119 ± 6
Neoprene	1.25 ± 0.01	30 ± 2 min	1.24 ± 0.01	30 ± 2 min	116 ± 4
Powdered nitrile (Box Ambi)	0.12 ± 0.01	33 ± 2 min	0.11 ± 0.01	30 ± 2 min	117 ± 4
Sol-Vex	0.38 ± 0.01	>4 hours	0.36 ± 0.01	>4 hours	nd[E]

[A] Mean thickness ± SD (n = 4).
[B] Breakthrough time ± SD (n = 4) detected by the change in color of indicators (finger permeation testing procedure).
[C] Breakthrough time ± SD (n = 4) detected by the color change of indicators (the palm-cuff permeation testing).
[D] The mean of mass permeated at all three locations: finger, palm, and cuff of gloves.
[E] Not detected for the Sol-Vex glove after 4 hours.

TABLE V. Results of Glove Thickness, Breakthrough Time, and the Quantitative Mass Data of Test Triethylamine on the Indicator Pads at the Breakthrough Time

Glove Type	Thickness[A] (Finger) (mm)	Breakthrough[B] Time of Triethylamine (Finger)	Thickness[A] (Palm and Cuff) (mm)	Breakthrough[C] Time of Triethylamine (Palm and Cuff)	Mass of Triethylamine[D] (μg/cm^2) (mean ± SD, n = 3)
Guardian (butyl-Std)	0.24 ± 0.01	6 ± 1 min	0.23 ± 0.01	6 ± 1 min	105 ± 5
Neoprene	1.25 ± 0.01	37 ± 2 min	1.24 ± 0.01	37 ± 2 min	102 ± 3
Powdered nitrile (Box Ambi)	0.12 ± 0.01	43 ± 2 min	0.11 ± 0.01	38 ± 2 min	103 ± 3
Sol-Vex	0.38 ± 0.01	>4 hours	0.36 ± 0.01	>4 hours	nd[E]

[A] Mean thickness ± SD (n = 4).
[B] Breakthrough time ± SD (n = 4) detected by the change in color of indicators (finger permeation testing procedure).
[C] Breakthrough time ± SD (n = 4) detected by the color change of indicators (the palm-cuff permeation testing).
[D] The mean of mass permeated at all three locations: finger, palm, and cuff of gloves.
[E] Not detected for the Sol-Vex glove after 4 hours.

the same glove batches and thickness).(16) It was found that rapid permeation times (PT) under the thermo-hand method were observed and were found to range from 44% to 46% faster than at room temperature for nitrile glove materials $[(PT_{22^\circ C} - PT_{34^\circ C})/PT_{22^\circ C}]$.

Quantitative Determination of Each Base

The volumes of each base on a pad at the point of breakthrough detection were determined, ranging from 0.40–0.41, 0.37–0.38, and 0.33–0.35 :L for butylamine, diisopropylamine, and triethylamine, respectively. These volumes represent from 118–121, 117–120, and 109–116 :g/cm^2 for butylamine, diisopropylamine, and triethylamine, respectively (based on their recoveries and linear equations). NaOH (an inorganic base) can be quantified using solvent desorption and HPLC analysis; however, no permeation of NaOH was observed for all glove materials after 4 hours.

DISCUSSION

The results obtained with the solvent desorption-GC procedure were consistent for reproducible recovery, with results ranging from 50–74% (RSD $\leq$ 5%) for these bases over the spiking range 0.22–1.11 μg. The recovery of each base was low. It is probable that the base bound strongly with indicator pads (thymol blue and glycerine) either binding or hydrogen bonding to give low desorbing mass. Recovery was dependent on the mass of each base applied to the pads, with increasing recovery observed with increasing mass. The recovery also depended on the volatility of the bases, with slight decreasing recovery observed with increasing base boiling points. It is probable that the base with the low boiling point could be extracted from the pad easier than other bases with higher boiling points. Excellent linear correlations were obtained for these bases over the spiking range 0.22–1.11 μg on pads using 5.0 :L of the extracted solutions for GC analysis.

The results of the permeation experiments demonstrated that within a given material type of the gloves tested, thickness had a primary effect on the breakthrough time of the test bases. The thicker gloves had the longer breakthrough time of the bases. Differences in glove materials or test bases yielded different breakthrough times. Based on our laboratory permeation testing, nitrile rubber glove materials are recommended for use when working with particular base chemicals. The results of the permeation experiments also demonstrated that within a given material type, but with different manufacturers, different breakthrough times were recorded. It is probable that the glove materials with powder and without powder would have different material densities. The results of differences in material density may yield different breakthrough times.

The quantitative mass data of test bases on the indicator pads, ranging from 118–121, 117–120, and 109–116 μg/cm^2 for butylamine, diisopropylamine, and triethylamine, respectively, indicated that these bases can be detected on the pads at the breakthrough times. The fact that the amounts of permeant detected in each glove test for each base were consistent indicates that the pads performed independently of the glove type in consistently collecting amounts of bases during the time before breakthrough was detected. Results provide further evidence that the use of the colorimetric indicator pad and the extraction process together yields consistent and reliable information due to the very small variations on repeated tests.

CONCLUSION

The results presented in this research indicate that the thermo-hand method and the colorimetric indicator pad together, evaluated as a glove performance test procedure, performed effectively for both organic and inorganic bases under simulated in-use conditions. The results indicate that pads exposed to the bases studied can be successfully analyzed under these conditions for the solvent desorption and GC analysis as described. Based on the promising results described, future studies will need to be conducted to determine whether pads placed on the inside of the glove and used by a worker in the actual workplace can be used to detect and quantify a permeating chemical. The backing of the pad used for this study is coated with plastic tape, so liquids such as sweat should not be able to reach to the other side of the pad and interfere with the color change. The mass of bases detected on the inside of gloves can be used to determine adequacy of protection, duration of acceptable use, or need for substitution of a more effective glove type(s). It is important to evaluate these pads for these uses in the workplaces.

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