# Thiocompounds As Simulants of Sulphur Mustard for Testing of Protective Barriers

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**ABSTRACT:** The protective potential of protective devices such as respirators, suits, gloves, and overboots is widely evaluated using the standard colorimetric test (spot disc breakthrough time test, also called SD BTT) involving sulfur mustard (SM) as the challenge chemical. The vesicant nature of SM makes the test inconvenient and poses stringent safety demands. Moreover, such tests are allowed only at a limited number of facilities, causing delay in product development and supply. This prompted the present study on the search for suitable SM simulant responsive to SD BTT test. The diffusivities at BTT ( $D_{BTT}$ ) of 10 commercially available thiocompounds through butyl rubber (IIR) were compared vis-a-vis  $D_{BTT}$  of SM. For three representative thiocompounds, namely methyl (phe-

### **INTRODUCTION**

Protection against toxic chemicals in the event of chemical contamination or chemical warfare is generally afforded by elastomeric barriers in the form of protective ensemble such as respirators, suits, gloves, and overboots. Performance evaluation of these devices involves testing of breakthrough time (BTT) against the challenge chemical. For the widely adopted standard spot disc BTT method (known as SD BTT)1-5 that is based on the oxidation of thioethers with an active chlorine-containing compound,<sup>5</sup> the challenge chemical with thio moiety is needed. The military protective gear is generally challenged against the blister agent sulfur mustard (SM), since SM permeates most materials including human skin, causing deleterious effects.<sup>6,7</sup> Testing the effectiveness of protective devices against SM is however extremely inconvenient because of its vesicant action. Moreover, such tests can only be conducted at a limited number of facilities permitted under the Chemical Weapons Convention.<sup>8</sup>

nyl thio)acetate, 2-chloroethyl phenyl sulfide (2-CEPS) and phenyl-*n*-propyl sulfide (PNPS), the transport parameters through IIR were obtained. PNPS and 2-CEPS were further compared with respect to  $D_{BTT}$  in elastomers such as IIR, ethylene–propylene–diene methylene rubber, polydimethylsiloxane, nitrile rubber, polybutadiene, and natural rubber. 2-CEPS showed generally same order of  $D_{BTT}$  as SM implying its potential use as a simulant. The transport parameters for various 2-CEPS/elastomer systems were also determined. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 111: 928–933, 2009

Key words: simulant; sulfur mustard; barrier; elastomer; diffusion

The use of simulant drastically reduces the risk with live agent testing, thereby minimizing occupational hazard. The desirable simulants are less hazardous molecular analogs of toxic chemicals, which mimic the diffusion properties of such vesicants. Lower toxicity and commercial availability of simulants allow their use at manufacturer's site. Consequently, product development time and cost are reduced, thereby expediting the supply to user(s). Another reason that prompted the search of simulants is the ratification of Chemical Weapons Convention<sup>8</sup> by most nations, according to which the development, production, stock piling, and use of chemical warfare agents (CWA) such as SM is prohibited. With suitable simulants of CWAs, research efforts for the development of defense countermeasures and testing of protective devices can continue uninhibited without violation of the convention. An additional advantage in using the simulant is that the liquid permeation can also be studied easily, unlike for toxic chemicals which poses practical difficulties, because of the risk in handling.

Realizing the importance of simulants, US tasked its Agent Simulant Knowledge advisory office to identify past work with simulants. National Institute of Occupational Safety and Health (NIOSH) also compiled the list of such studies<sup>9</sup> based on which projects were funded to various agencies for further

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exploring the potential simulants for testing of filters and barrier materials. Some of the studies originating from NIOSH projects<sup>9,10</sup> have recommended 1,6dichlorohexane, bis(4-chlorobutyl) ether, 2,4-dichlorophenol as potential simulants. Oxygen analog of SM, namely bis(2-choroethyl) ether, has also been extensively investigated as a simulant.<sup>3,4</sup> However, none of these simulants contain thio moiety, and hence are not amenable to SD BTT test.

Very few reports exist in the open literature on the permeation of thiocompounds in barrier polymers. Pal et al.<sup>11</sup> used di-*n*-butyl sulfide (DNBS) as a SM simulant for permeation measurement in different polymeric materials such as teflon, polyester, nylon, viton, and rubbers like nitrile, butyl, neoprene, and chloroprene. Vojkovic et al.<sup>12</sup> reported a method for monitoring permeation of DNBS through clothing materials. However, DNBS is highly obnoxious and volatile, thus limiting its desirability. Lewis et al.<sup>13</sup> used 2-chloroethyl ethyl sulfide (2-CEES), 2chloroethyl methyl sulfide (2-CEMS), and 1,5dichloropentane for testing transparent materials like polycarbonates, polymethyl methacrylate, and polyurethane while Bodnar et al.<sup>14</sup> used 2-CEMS, 2-CEES, and 3-chloropropyl thioacetate (3-CPTA) for testing butyl rubber (IIR). It was reported that<sup>13,14</sup> BTT of 2-CEMS is more consistent to that of SM, whereas 2-CEES has slightly shorter BTT than SM, but both the simulants caused crazing of the samples and hence are not desirable for testing purposes. Moreover, 2-CEMS shows vesicant action; hence, it cannot be used as a safe simulant. One another reason for limiting the use of 2-CEMS and 2-CEES as simulants is that they evaporate more quickly than SM. Therefore, if one used 2-CEMS and 2-CEES as SM simulants, a barrier may be needed to prevent their evaporation. On the other hand, 3-CPTA is less volatile, but shows much longer BTT than SM,14 and hence cannot realistically mimic the diffusion behavior of SM.

Based on the hypotheses that instead of chloropropyl moiety in thioacetate, methyl phenyl group may help to approach the BTT of SM, methyl (phenyl thio)acetate (MPTA) was chosen as one of the candidate simulants in the present study. The BTT of MPTA in IIR membranes was compared vis-a-vis other commercially available thiocompounds such as 2-chloroethyl phenyl sulfide (2-CEPS), phenyl-npropyl sulfide (PNPS), DNBS, ethyl 2-mercaptocetate, diisopropyl sulfide, thiophene-2-thiol (T2T), Sethyl thioacetate, ethyl sulfide (ES), and ethyl isothiocyanate. The permeation parameters for three short-listed compounds (MPTA, PNPS, and 2-CEPS) were obtained using gravimetric method. PNPS and 2-CEPS were further compared with respect to their  $D_{\rm BTT}$  in different elastomers such as IIR, ethylene– propylene-diene methylene rubber (EPDM), polydimethylsiloxane (PDMS), nitrile rubber (NBR), polybutadiene (PB), and natural rubber (PI). 2-CEPS showed generally the same order of  $D_{BTT}$  as SM. The transport parameters for 2-CEPS/elastomer systems indicate 2-CEPS as a potential simulant of SM for testing of materials made from IIR, PI, and PB.

#### **EXPERIMENTAL**

#### Materials

All simulants were used as received from Lancaster, Lancashire, U.K. The details of rubbers and chemicals used are as reported earlier.<sup>15</sup> Elastomeric membranes of PI, IIR, PB, NBR, and EPDM were prepared by casting from 10% w/v solution of the rubber in toluene using the reported recipe.<sup>15,16</sup> PDMS was synthesized in laboratory using the standard procedure.<sup>16</sup> The thickness of the membranes was maintained at 0.26  $\pm$  0.05 mm. The membranes were dried in a vacuum oven for 24 h at 100°C to remove surface-absorbed moisture prior to testing.

#### Diffusivity at breakthrough $(D_{BTT})$

The BTT of test chemical was determined using a standard color reaction test (SD BTT) based on oxidation of thioethers with 2,4-dichlorophenylbenzoyl chloroimide—an active halogen containing compound.<sup>1–5</sup> The time taken for the first appearance of blue color on freshly prepared detector paper (Congo-red paper dotted with 2,4-dichlorophenylbenzoyl chloroimide) from the time of initial application of challenge chemical was termed the BTT. An average of three readings was reported. For a given thickness (*h*) of the sample and temperature, the higher the BTT value, the lower is the diffusivity of the chemical. The diffusion coefficient *D* at break-through ( $D_{\text{BTT}}$ ) was calculated from the relation<sup>2–4</sup>:

$$D_{\rm BTT} = \frac{h^2}{\pi^2({\rm BTT})} \tag{1}$$

#### Vapor sorption

A gravimetric method<sup>2–4</sup> was used to determine the vapor sorption of simulants. Membranes were exposed to the vapors of the challenge chemical in a closed chamber at 30°C  $\pm$  2°C. The specimens were taken out periodically and weighed on an analytical balance (Shimadzu, Japan) with an accuracy of 0.1 mg. The mean of three readings was reported; the standard deviation ranged from 1.8 to 6.9. The mole % vapor sorption  $Q_t$  obtained from the gain in weight of the specimen was related to

Candidate Simulants and Their D <sub>BTT</sub> Through IIR Membranes								
Chemical			Boiling point (°C)	BTT				
	CAS no.	Molecular weight		Mean (s)	$SD^{a}$	$D_{\rm BTT}~({\rm m}^2/{\rm s})$		
MPTA	17277-58-6	182	104–105/4 mm	270,000	745	$2.5 \times 10^{-14}$		
2-CEPS	5535-49-9	172	90–91/1 mm	68,400	730	$9.9 \times 10^{-14}$		
SM	505-60-2	159	228	90,000	800	$7.5 \times 10^{-14}$		
PNPS	874-79-3	152	215-220	4,200	169	$1.6 \times 10^{-12}$		
DNBS	544-40-1	146	188–189	600	40	$1.1 \times 10^{-11}$		
EMA	623-51-8	120	155-161	2,400	109	$2.8 \times 10^{-12}$		
DIPS	625-80-9	118	_	960	25	$7.1 \times 10^{-12}$		
T2T	7774-74-5	116	_	n.d. <sup>b</sup>	_	_		
SETA	625-60-5	104	116–117	18,000	100	$3.8 \times 10^{-13}$		
ES	352-93-2	90	90-92	300	15	$2.2 \times 10^{-11}$		
EITC	542-85-8	87	130–132	12,000	160	$5.7 \times 10^{-13}$		

TABLE I

<sup>a</sup> Standard deviation.

<sup>b</sup> Not detected upto 30 h.

equilibrium sorption uptake,  $Q_{\infty}$ , through an empirical relation<sup>2-4,15-17</sup>:

$$\frac{Q_t}{Q_\infty} = Kt^n \tag{2}$$

where *K* is a constant characteristic of the polymerpermeant interaction, t is the time of exposure, and n is the transport coefficient that describes the nature of sorption. For  $n \leq 0.5$ , diffusion is Fickian, while for n > 0.5, it is non-Fickian. K and n are obtained from the intercept and slope, respectively, of the plot of log  $Q_t/Q_{\infty}$  versus log t. The experimental data considered to obtain the values of K and n are generally all those for which  $Q_t/Q_{\infty} < 0.60$ .

## Solubility of simulants

A more specific indication about the polymer-permeant interaction is given by the solubility S which was determined by the immersion method. Membrane specimens of predetermined weight were immersed in the simulants. After equilibration for 72 h at room temperature, the membranes were removed and the superfluous liquid was wiped with tissue paper. The weight of the swollen membrane was determined using an analytical balance. From the gain in weight of the membrane, the solubility coefficient (S) was determined by computing the number of moles of the chemical sorbed per unit volume of the membrane at atmospheric pressure. The mean of three readings was reported; the standard deviation ranged from 2.6 to 9.5.

# **RESULTS AND DISCUSSION**

The molecular weights of the chosen thiocompounds ranged from 87 to 182, and their boiling points varied from 90 to 220°C. SM with a molecular weight of

159 boils at 228°C. The average BTT of SM through IIR membranes (thickness 0.26  $\pm$  0.05 mm) determined using SD BTT test was found to be 25 h with corresponding  $D_{\rm BTT}$  of 7.5  $\times$  10<sup>-14</sup> m<sup>2</sup>/s. The  $D_{\rm BTT}$ for the thiocompounds (Table I) was found to be of the order of  $10^{-11}$  to  $10^{-14}$  m<sup>2</sup>/s.

Molecular size and weight of diffusant molecules are important parameters governing the diffusivity. The bulkier compound, MPTA (molecular weight, 182), showed comparatively sluggish diffusion than all other compounds, presumably due to the lower mobility.18,19 It was observed that the lighter compounds DNBS and ES diffuse most rapidly;  $D_{BTT}$  is of the order of  $10^{-11}$  m<sup>2</sup>/s. Some workers<sup>9,10</sup> have used DNBS as a simulant for SM; however, because of its obnoxious odour and higher volatility, it may be unacceptable to most industrial workers. No BTT was observed for T2T up to 30 h, probably because of its highly viscous nature that impedes diffusion.  $D_{\rm BTT}$  of 2-CEPS approaches the value observed with SM.

Although BTT provides an estimate of the duration for which the protective gear can safely be used, knowledge of transport coefficients is necessary to understand and elucidate the diffusion mechanism as well. Three representative thiocompounds, namely MPTA, 2-CEPS, and PNPS, were shortlisted for studying the vapor sorption and diffusivity through IIR. MPTA and 2-CEPS were selected because of the agreement of their order of  $D_{BTT}$  with that of SM  $(10^{-14} \text{ m}^2/\text{s})$ , while PNPS was chosen because of its molecular weight and boiling point being closer to SM. While diffusion behavior of MPTA and PNPS has not been studied hitherto, the only study on diffusivity of 2-CEPS through elastomers has been reported by Rivin et al.<sup>10</sup>

The vapor sorption of MPTA, 2-CEPS, and PNPS as a function of time was studied at  $30^{\circ}C \pm 2^{\circ}C$ through IIR membranes of thickness  $0.24 \pm 0.05$  mm



Figure 1 (a) Sorption plot for PNPS/IIR system at  $30^{\circ}C \pm 2^{\circ}C$ . (b) Sorption plot for MPTA/IIR system at  $30^{\circ}C \pm 2^{\circ}C$ . (c) Sorption plot for 2-CEPS/IIR system at  $30^{\circ}C \pm 2^{\circ}C$ .

using the weight-gain method. The sorption plots obtained from these data are shown in Figure 1. It was observed that the mol % sorption was lowest for MPTA ( $2.9 \times 10^{-3}$  mol %) and highest for PNPS ( $154.9 \times 10^{-3}$  mol %). Within the limits of experimental error, the sorption plot for PNPS/IIR system is almost linear. A shift in the linearity was observed in MPTA/IIR and 2-CEPS/IIR systems, with the former assuming an almost inverted "S" and the latter an inverted "L" profile. The transport coefficient *n* was found to be greater than 0.5 with all the three compounds (Table II), indicating their non-Fickian diffusion through IIR. No attempt was therefore

made to determine the diffusion coefficients which would require a detailed analysis of relaxation and swelling effects which is beyond the scope of the present work.

The extent and mode of sorption of a compound in a polymer is dependent upon the relative strengths of the interactions between the permeant molecule and the polymer or between the polymer molecules themselves within the matrix. A comparatively higher value of *K* for PNPS (Table II) indicates a relatively higher interaction of IIR with PNPS. The sorption coefficient *S* of the polymer can also give a fair idea of the polymer-permeant interactions. The trend in *S* values as depicted in Table II followed the order PNPS > 2-CEPS > MPTA, indicating a higher interaction of IIR with PNPS when compared with other simulants.

PNPS and 2-CEPS, being two of the simulants which show higher interaction with IIR, were further compared with respect to their  $D_{BTT}$  in other elastomers. The  $D_{BTT}$  values of these thiocompounds along with that of SM determined from the triplicate sample using the standard SD BTT test are shown in Table III.  $D_{BTT}$  for SM/elastomer systems followed the trend PDMS  $\sim$  EPDM > PI > PB > NBR > IIR. It was further observed that IIR offers the highest resistance to the diffusion of PNPS and 2-CEPS as well, thus indicating its potential as a protective barrier. In contrast, PDMS provides the highest diffusivity, thus acting as an open window. Lower diffusivity of 2-CEPS in IIR as compared to PDMS and NBR was also reported by Rivin et al.<sup>10</sup> Wilde and Bodnar<sup>20</sup> also reported similar results for transport of 2-CEES in PDMS (45.6 mil), IIR (33.4 mil), and NBR (41.9 mil) rubbers; BTT varied in the order IIR (76.6 min) > NBR (48.5 min) > PDMS (6.6 min).

The observed trend in diffusivity and permeability of the simulants in various elastomers is a complex function of various factors, such as chain flexibility, segmental mobility, intermolecular interaction, phase and aggregation state of a polymer, density of macromolecular packing, extent of crosslinking, and free volume available for diffusional jump. Within the same physical state, the packing of macromolecules assumes greater importance in determining the extent of free volume and hence the diffusivity. The highly saturated and closely packed isobutylene

TABLE II Transport Parameters for Simulant Vapor/IIR Systems at  $30^{\circ}C \pm 2^{\circ}C$ 

		$50 C \pm 2 C$			
Simulant	п	$K (g/g min^n)$	$S \pmod{\mathrm{m}^{-3} \mathrm{Pa}^{-1}}$		
MPTA	0.62	0.018	0.00053		
2-CEPS	0.68	0.023	0.0033		
PNPS	2.3	0.029	0.0204		

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Comparison of $D_{BTT}$ of Simulants and SW in Elastomers									
	SM			PNPS			2-CEPS		
Code	h <sup>a</sup> (mm)	BTT (s), mean	D <sub>BTT</sub> (m²/s)	h <sup>a</sup> (mm)	BTT (s), mean	D <sub>BTT</sub> (m²/s)	h <sup>a</sup> (mm)	BTT (s), mean	D <sub>BTT</sub> (m <sup>2</sup> /s)
IIR PDMS NBR EPDM PB	0.26 0.84 0.28 0.20 0.29	90,000 (244) <sup>b</sup> 960 (18) 2,880 (90) 360 (08) 300 (04)	$\begin{array}{c} 7.5 \times 10^{-14} \\ 7.4 \times 10^{-11} \\ 2.7 \times 10^{-12} \\ 1.1 \times 10^{-11} \\ 2.9 \times 10^{-12} \end{array}$	0.26 0.42 0.28 0.20 0.24	1,800 (90) 720 (13) 2,880 (88) 540 (21) 360 (10)	$\begin{array}{l} 3.8 \times 10^{-12} \\ 2.4 \times 10^{-11} \\ 2.9 \times 10^{-12} \\ 7.5 \times 10^{-12} \\ 1.6 \times 10^{-11} \end{array}$	0.26 0.62 0.28 0.20 0.24	68,400 (282) 1,200 (61) 7,620 (99) 840 (25) 780 (18)	$\begin{array}{c} 9.9\times10^{-14}\\ 3.2\times10^{-11}\\ 1.0\times10^{-12}\\ 4.8\times10^{-12}\\ 7.5\times10^{-12}\end{array}$

 TABLE III

 Comparison of  $D_{BTT}$  of Simulants and SM in Elastomers

<sup>a</sup> Average thickness of five readings at five different points measured using a micrometer screw gauge with an accuracy of  $\pm 0.01$  mm.

<sup>9</sup> Values in parentheses indicate SDs (standard deviations).

chains in IIR show lower diffusivity in contrast to PDMS and other elastomers.

The latter elastomers also have a comparatively higher free volume<sup>2–4</sup> facilitating diffusion. Because of segmental mobility at temperatures above the glass transition temperature, the dipolar interaction bonds between the nitrile groups in NBR may break or reform, creating transient space for the diffusional jump.<sup>2</sup> However, in the case of IIR, the pendant



Figure 2 (a) Sorption plots for 2-CEPS/elastomer systems (PDMS, EPDM, and NBR) at  $30^{\circ}C \pm 2^{\circ}C$ . (b) Sorption plots for 2-CEPS/elastomer systems (IIR, PI, and PB) at  $30^{\circ}C \pm 2^{\circ}C$ .

methyl groups are symmetrically substituted on the same carbon atom in the main chain. Therefore, during segmental mobility caused by bond rotation, the probability of an interchange of these methyl groups is significant, rather than the creation of free volume,<sup>2</sup> thereby rendering IIR relatively impermeable.

A further comparison of  $D_{BTT}$  values in Table III also shows that, with the exception of EPDM, PNPS diffuses faster than SM in other elastomers. The  $D_{\rm BTT}$  values are less by one order of magnitude for PNPS in PB and PI, while they differ by two orders of magnitude in case of IIR. In contrast, the agreement in the order of magnitude of  $D_{BTT}$  for SM and 2-CEPS was similar in all elastomers except EPDM. It was also observed that 2-CEPS diffuses slightly faster than SM in IIR, PB, and PI. This implies that 2-CEPS is most suitable for testing of protective devices especially made from IIR, PI, or PB since the formulation providing resistance to diffusion of 2-CEPS would also afford protection against SM. It is however cautioned that, for testing of products made from PDMS, NBR or EPDM, 2-CEPS may not be a suitable simulant as it diffuses slightly slower than SM and may therefore provide overestimated values of protective potential.

The nature of the polymeric barrier is thus of paramount importance while choosing a suitable simulant. In view of the earlier results, it was considered worthwhile to investigate the sorption behavior of

TABLE IV Transport Parameters for 2-CEPS Vapor/Elastomer Systems at 30°C ± 2°C

Elastomer	Density (g/cm <sup>3</sup> )	п	$K (g/g min^n)$	$\begin{array}{c} S \ (\mathrm{mol} \\ \mathrm{m}^{-3} \ \mathrm{Pa}^{-1}) \end{array}$		
PDMS	0.86	0.60	0.173	0.0050		
NBR	0.91	0.96	0.108	0.0943		
EPDM	0.82	1.66	0.220	0.1031		
PB	0.87	0.63	0.257	0.1003		
PI	0.89	0.36	0.331	0.1440		
IIR	0.89	0.68	0.02	0.0030		

2-CEPS in various elastomers. The sorption plots for the elastomers exposed to the saturated atmosphere of simulant vapor in a closed environment at 30°C  $\pm$ 2°C are shown in Figure 2. The transport parameters obtained from these plots are shown in Table IV. Except for PI, the 2-CEPS diffusion is non-Fickian in all elastomers. The solubility of 2-CEPS showed a good consistency with *K* values. A higher *K* (for instance in PI) implies a higher polymer-permeant coupling and, consequently, a higher solubility. PDMS, NBR, and IIR with lower  $K \sim 0.1$  showed lower solubility of 2-CEPS than in other elastomers.

As a further scope of the work, it would be interesting to derive the diffusion coefficients for non-Fickian diffusion taking into account simulantinduced swelling and relaxation in elastomers. The overall effect of diffusivity and solubility could provide insights to the permeability of simulants in elastomers.

#### CONCLUSIONS

Ten thiocompounds ranging in molecular weights from 87 to 182 were screened as simulants of SM for testing of protective barriers using SD BTT test. Based on the  $D_{BTT}$  values, 2-CEPS appears to be a promising simulant for testing of protective devices formulated from IIR, PI, or PB. It was found that while the BTT of 2-CEPS is lower, its diffusivity is greater in comparison to SM in these elastomers. Hence, the formulation providing resistance to permeation of 2-CEPS would also afford protection against SM. It is however cautioned that, for testing of products made from PDMS, NBR, or EPDM, 2-CEPS may not be a reliable simulant, as it diffuses slightly slower than SM in these polymers and may therefore provide overestimated values of protective potential. The nature of the polymeric barrier is thus of paramount importance while choosing a suitable simulant.

The study thus indicates that there is no "universal simulant" viz a simulant independent of the nature of polymeric barrier. Simulants could be used to provide preliminary information. Nevertheless, the final evaluation and certification of protective devices has to be based on results obtained using tests with live chemical agents.

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