

The Service Life of Respirator Cartridges with Binary Mixtures of Organic Vapors

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Abstract

The service life of organic-vapor cartridges was evaluated with acetone/*m*-xylene, acetone/styrene and toluene/*m*-xylene solvent mixtures. For almost all the tests, the total concentration was maintained at 1000 ppm. The solvent 1/solvent 2 concentration ratio was varied from 0.1 to 0.75. When possible, a fractional concentration of 0.9 was tested. Also, the effect of the variation in concentration of one of the solvents on the breakthrough time of the other solvent was studied. For all tests, a relative humidity of 40% was used. The results show that in general, the more volatile compound breaks through first, independent of the concentration ratio, and its breakthrough concentration exceeds the exposure concentration. In cases where the more volatile compound was at the lower concentration ratio (the more volatile compound of 100 ppm, and the less volatile compound at 900 ppm), the maximum breakthrough concentration was more than three times the exposure concentration. This maximum decreases when the fractional concentration of the more volatile compound increases. This phenomenon was interpreted in terms of desorption of the more volatile by the less volatile compound from the carbon surface. It was experimentally demonstrated that at the relative humidity used, adsorbed water molecules are desorbed by the less volatile solvent namely toluene, *m*-xylene or styrene. The service life of cartridges is discussed in terms of 10% breakthrough times. In all cases, the breakthrough of the more volatile compound determines the service life of cartridges and is dependent on the concentration of the less volatile compound. For acetone mixtures with *m*-xylene or styrene, non-significant differences were observed in the acetone 10% breakthrough time. This value stays close to the 10% breakthrough time for pure acetone at 1000 ppm. For toluene/*m*-xylene mixtures, the 10% breakthrough time for toluene corresponds to that for pure toluene at 1000 ppm. These results have been discussed in terms of an additive concentration effect of the less volatile solvent on the more volatile solvent.

1. Introduction

Adsorption type devices are the respiratory protection most often used when organic hazardous contaminants are found in the air. These respirators use cartridges with activated carbon of mineral or petroleum origin as adsorbent. The adsorption capacity and service life are dependent not only on the carbon surface area and the amount of carbon in the cartridge, but also on the contaminant concentration and the volatility of the contaminant. In spite of the fact that less volatile vapors result in a longer cartridge service life, no relationship has been found between solvent boiling points and breakthrough times (Nelson and Harder, 1976). Also, humidity is known to affect the service life of cartridges (Nelson *et al.*, 1974; Yoon and Nelson, 1988). With most contaminants, there are no indicators of the end of cartridge service life, and the odor of the compound is often the only indicator of cartridge saturation. This practice is dangerous because there are many compounds whose olfactory detection threshold is at a concentration higher than the TLV (Nelson and Correia, 1976).

The actual NIOSH test for approval of respirator cartridges for organic vapors is carried out with only one contaminant, carbon tetrachloride, at a constant concentration of 1000 ppm at 25°C and 50% humidity and an air flow of 64 L/min when the cartridges are tested as received (30 CFR Part 11, Subpart L, 11.162-8). Also, certification requires the same kind of tests on cartridges preconditioned for 6 hours at 25% and 85% relative humidity. The information generated from these tests cannot be used to estimate the service life of cartridges because only one compound is involved. Furthermore, it is known that there are many contaminants that are less well adsorbed than carbon tetrachloride in the respiratory cartridges (Nelson and Correia, 1976).

For several years now, extensive research has been carried out to characterize the filtration capacity of cartridges with various types of contaminants. The most important work is that of Gary Nelson *et al.* (1974-76) who have evaluated respiratory cartridges with 121 different solvents. In this case, the cartridges were exposed to a constant concentration of a single contaminant at a controlled humidity, air flow, and temperature. These authors also characterized the effects of contaminant concentration, temperature, and relative humidity on the service life of cartridges (Nelson and Harder, 1976; Nelson, Correia and Harder, 1976).

By characterizing the service life of cartridges at different concentrations of contaminant, curves of contaminant concentration versus time at a given breakthrough percentage (e.g., 10% breakthrough) can be generated. This type of curve could be useful in predicting the service life of cartridges in workplaces, provided the contaminant concentration is known. However, the practical use of these results remains limited because they are valid only for the case of one contaminant. In the workplace, the ambient air often contains more than two contaminants.

Despite the fact that some studies have been performed to evaluate the service life of cartridges with binary mixtures of contaminants (Jonas, 1972; Jonas *et al.* 1983 and 1986; Rudling, 1988; Siddiqi and Thomas, 1981; Swearengen and Weaver, 1988), no systematic research has yet been done. This type of research is very important in developing knowledge about the vapor-retention mechanisms of the activated carbon for these situations, in order to predict the service life of respirator cartridges used in the workplace. The objective of this study was to characterize the adsorption capacity of cartridges exposed to binary mixtures of organic vapors.

2. Experimental

2.1 Chemicals

The chemicals used to generate the contaminants were the following: acetone, styrene, and *m*-xylene of a certified quality, from Fisher Chemical Co., and toluene, of HPLC quality, from Baker Chemical Co.

2.2 Choice of Systems

The following binary mixtures were studied: acetone/*m*-xylene, acetone/styrene, and toluene/*m*-xylene were chosen as model systems having different physical-chemical properties; in the first case, the boiling point (BP) of acetone is 56.5°C

and the molar volume (V_m) is $72.9 \text{ cm}^3/\text{mole}$ and the BP for *ra*-xylene is 139°C and its V_m is $122.9 \text{ cm}^3/\text{mole}$; in the second case, the physical-chemical properties of the two substances are closer, with the BP of toluene being 110.4°C and its V_m being $106.4 \text{ cm}^3/\text{mole}$. In the case of acetone/styrene, the system was chosen for practical reasons; these contaminants are found in fiberglass industries. Furthermore, the physical-chemical properties resemble those of the acetone/*m*-xylene system; the BP for styrene is 146°C and the V_m is $114.8 \text{ cm}^3/\text{mole}$.

2.3 The Assembly

The experimental assembly for evaluating the service life of respirator cartridges is represented in Figure 1. The assembly has four parts:

1) Generation system composed of:

- a) a temperature, air-flow, and humidity controller from the Miller-Nelson Company, model HCS-301 (Miller-Nelson Research, Inc., Dublin, Calif.) [No. 1];
- b) a contaminant-injection system composed of two motor-controlled syringes, one for each solvent [No. 2];
- c) an exposure chamber consisting of a 120-L plexiglas box where up to four cartridges can be exposed simultaneously to the contaminants [No. 3].

2) Respiratory system:

The respiratory system consisted of a compressor working in aspiration mode at a constant flow [No. 4]. To ensure a constant flow, steel tubes with a critical orifice were installed in the pipe connecting each cartridge to the aspiration system. Flow meters were installed at the outlet of each cartridge. Most experiments were performed at a 24 L/min flow rate. However, in some cases for practical reasons, a 36 L/min flow rate was used.

3) Analytical system:

The analytical system consisted of:

- a) a computer-controlled valve selector [No. 6 and No. 9], allowing alternate

sampling of the air in the exposure chamber and the air passing through the cartridges;

- b) a Hewlett-Packard chromatograph, model 5890, series II, with a flame ionization detector (FID) [No. 5].

To separate the solvents in the mixture, a 25-cm column with an internal diameter of 2 mm filled with carbopack C/0.1% SP-1000 as adsorbent was used. The temperature of the detector was 250°C and the carrier gas was nitrogen. The flow of the carrier gas and the temperature of the oven were established for each system in order to obtain the best separation. For the acetone/*m*-xylene system, the nitrogen flow rate was 30 mL/min , and the oven temperature was 200°C ; for the acetone/styrene system, the nitrogen flow rate was 49 mL/min , and the oven temperature was 220°C ; and for the toluene/*m*-xylene system, the nitrogen flow rate was 30 mL/min , and the oven temperature was 185°C .

Calibration curves were prepared for each pair of solvents and each experimental condition. Standards for the pure solvents and for the solvent mixtures were prepared by diluting the liquid solvent with air in Tedlar® bags. For each test, a standard binary mixture with the same composition as that for the test was prepared and analyzed. A chart recorder and a signal integrator were used simultaneously to register the chromatographic peaks. The standard peak heights from the chart recorder serve as a reference to control the contaminant concentration in the chamber during the test. The contaminant concentration is calculated from the peak area values with the integrator.

4) Data acquisition and treatment system:

The acquisition system consisted of a Hewlett-Packard integrator, model 3395, [No. 7] and an IBM-PC computer. Data processing was carried out using SOLO software, version 3.0.*

* Hintze, J.L.: Solo Statistical Systems (Computer Software) BHDP Statistical Software, Inc., 1989.

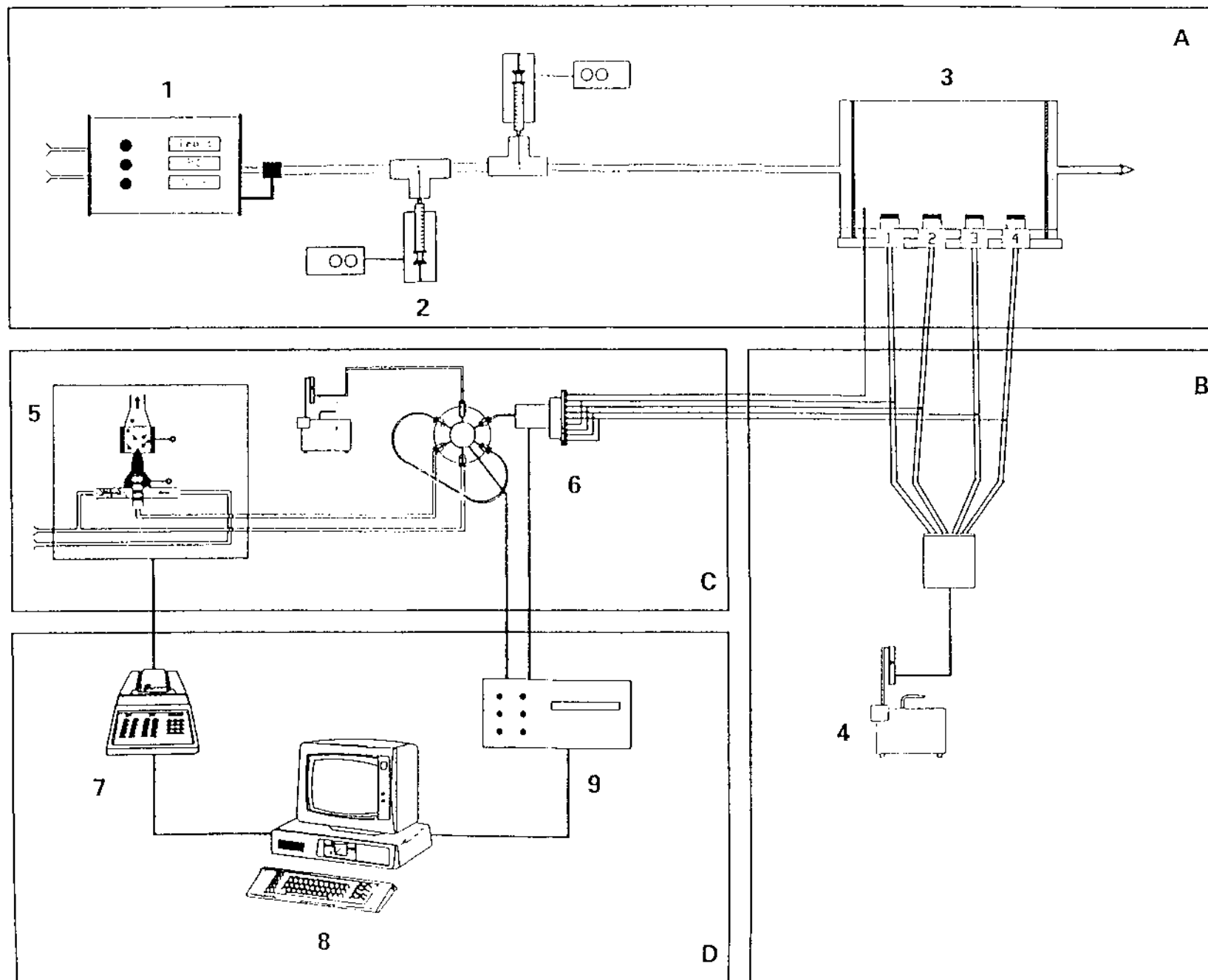


Figure 1. Diagram of the experimental assembly for evaluating the service life of respirator cartridges.

2.4 The Cartridges

The cartridges used in this study were from Scott Aviation (Lancaster, N.Y.), model 640 OV for organic vapors. These cartridges corresponded to lot nos. 06149, 06159, and 12198 whose weight of carbon was 50 ± 1 g and type of carbon was activated coconut at high temperature. The volume of carbon in the cartridges was 112 cm^3 , the diameter of the cartridges was 8 cm, and the depth 2.24 cm. The surface area of the activated carbon was $1280 \text{ m}^2/\text{g}$. The cartridges were tested as received. For each test, two or four cartridges were evaluated simultaneously. The weight of carbon in each cartridge was controlled immediately before and after the experiment.

2.5 Evaluation of the Service Life

The service life of the cartridges was characterized first using the single system, by exposing them to constant contaminant concentrations between 100 ppm and 1000 ppm. Subsequently,

the cartridges were tested with the binary system, at the following solvent 1 and solvent 2 concentrations; 100 ppm and 900 ppm; 250 ppm and 750 ppm; 500 ppm and 500 ppm; 750 ppm and 250 ppm; 900 ppm and 100 ppm. The total concentration of the contaminants was 1000 ppm.

In addition, the effect of the concentration of one solvent on the breakthrough of the other was analyzed. In this case, the concentration of one of the contaminants was kept constant at 500 ppm and the concentration of the other was varied between 100 ppm and 1000 ppm.

The tests were carried out at a temperature of $25 \pm 1^\circ\text{C}$ and a relative humidity of $40 \pm 1\%$.

The temperature and the humidity of the exposure chamber were measured with a Vaisale device (Helsinki, Finland) model HUI14.

3. Results and Discussion

3.1 Acetone/M-Xylene and Acetone/Styrene Systems

The tests with these systems were performed at a flow rate of 24 L/min, and for each experiment, four cartridges were tested simultaneously. In general, the trends of the breakthrough curves for both systems are similar. For this reason only curves for acetone/m-xylene will be presented.

3.1.1 The Single Systems

Figure 2 presents, as an example, the breakthrough curves for 107 ppm, 260 ppm, 501 ppm, 726 ppm and 1060 ppm of pure acetone. These concentrations correspond to the average values for the acetone in the exposure chamber. These curves are represented in terms of the breakthrough percentage as a function of time. The breakthrough curves have a sigmoidal shape, which is typical of the breakthrough of a pure gas through respirator cartridges. The shapes of the m-xylene and styrene breakthrough curves are similar, with longer breakthrough times than for acetone. This is consistent with the fact that the volatility of m-xylene and styrene is lower than that of acetone.

Also in Figure 2, the 10% breakthrough time values extracted from the breakthrough curves are represented in terms of the acetone concentration as a function of time. This type of curve is

used because of its practical utility in the workplace. Hence, by knowing the average concentration of a given pollutant, the service life at 10% breakthrough can be estimated. The results in Figure 2 show that there is no regular variation in the increasing 10% breakthrough time for acetone when the contaminant concentration decreases. The same kind of behavior was obtained with the other contaminants studied.

In Table 1, the 10% breakthrough time values for acetone, m-xylene, and styrene are presented. The results in this table show that the largest 10% breakthrough time is for styrene, followed by m-xylene and finally acetone. This order is the same as their decreasing order of solvent volatility.

Adsorption Capacity of the Cartridges

Yoon and Nelson (1984) stated that the maximum adsorption capacity of a cartridge for a given contaminant may be calculated from the following equation:

$$W_t = CF\tau \quad (1)$$

where

W_t = the maximum adsorption capacity of the respirator cartridge as a unit of weight (e.g., mg, g)

C = the concentration of the contaminant as a unit of weight (e.g., mg, g)/L

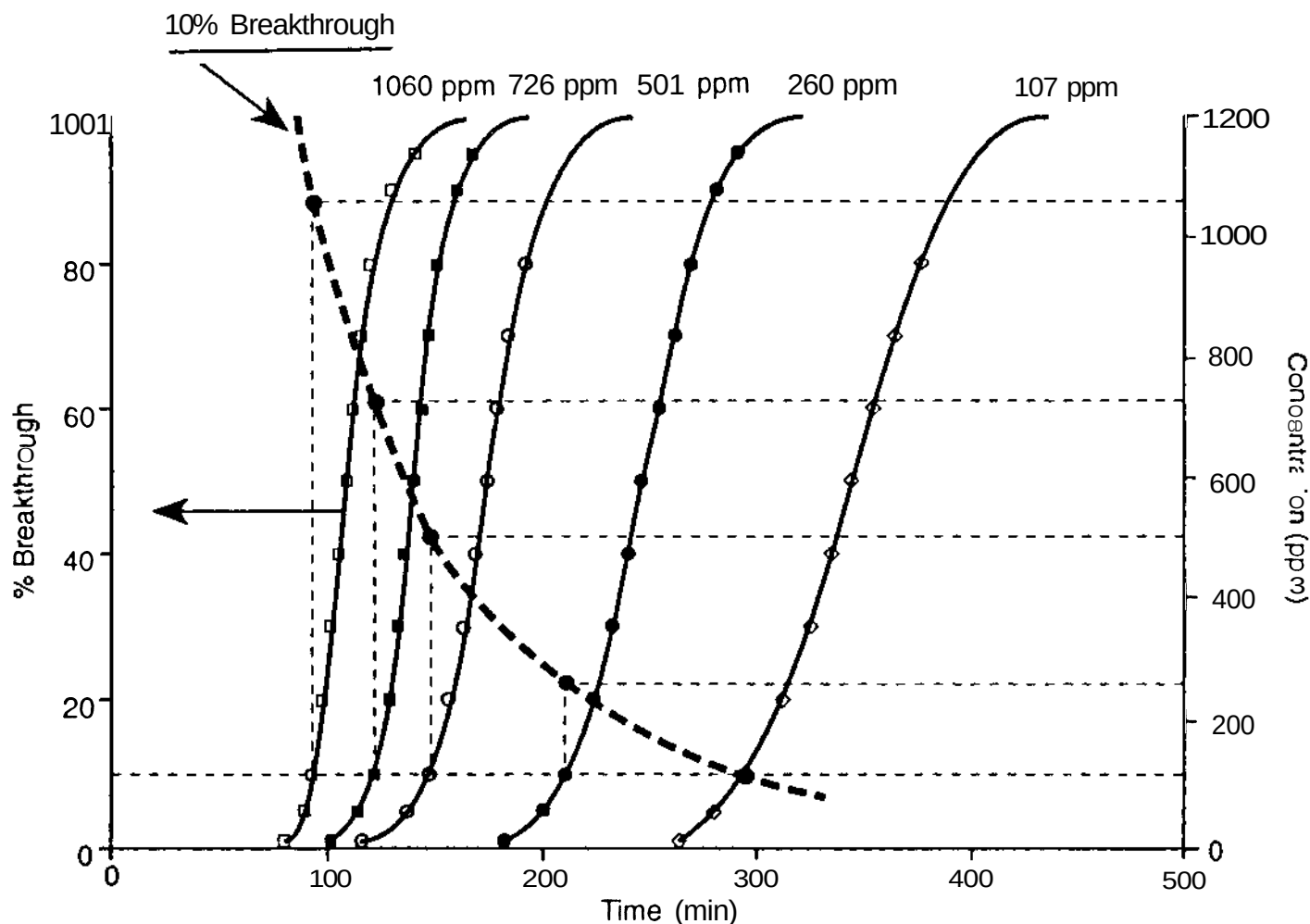


Figure 2. Breakthrough curves and 10% breakthrough curves for the different single acetone concentrations.

- F = the air flow passing through the cartridge in L/min
 T = the service life of the cartridge at 50% breakthrough in minutes.

From Eq. (1), the adsorption capacity of the cartridges with pure acetone, m-xylene, and styrene was calculated at different concentrations, and the results are presented in Table 1. The adsorption capacity is represented in terms of the total adsorption space on the carbon surface (W_v) occupied by a contaminant. These values were calculated from Wt/d , where d represents the contaminant liquid density (Jonas and Sansone, 1976).

The results in Table 1 show that, when compared at equivalent solvent concentrations, the cartridges have a higher adsorption capacity for

m-xylene and styrene than for acetone. For example, when the concentration of acetone is 1060 ppm, the adsorption capacity of the cartridge is 8.3 cm^3 . At a m-xylene concentration of 973 ppm, the adsorption capacity of the cartridge is 23.7 cm^3 , and for styrene at 1045 ppm, the adsorption capacity is 24.0 cm^3 . This means that the adsorption space on the carbon surface occupied by the contaminant is equivalent for m-xylene and styrene, and significantly lower for acetone. This agrees with the fact that at equivalent concentrations, the relative vapor pressures and molar volumes for m-xylene and styrene are very close and are significantly higher than those for acetone. These results agree with those of the Gary Nelson *et al.* studies which demonstrated that in general, the less volatile molecules are retained more by the activated carbon.

Table 1: Adsorption capacity and 10% breakthrough times for cartridges with the acetone, m-xylene, and styrene single systems. Flow rate of 24 L/min

Concentration (ppm)			Cartridge adsorption capacity						10% breakthrough time (min)		
acetone	m-xylene	styrene	acetone		m-xylene		styrene		acetone	m-xylene	styrene
			r (min)	W_v (cm ³ /c)	r (min)	W_v (cm ³ /c)	r (min)	W_v (cm ³ /c)			
107	93	254	340	2.7	1640	18.4	728	20.6	296	1449	678
260	257	481	270	5.1	702	21.8	418	22.4	210	646	390
501	446	497	170	6.4	386	20.8	393	21.7	147	356	335
726	857	725	140	7.4	215	22.3	270	21.8	122	195	250
1060	973	1045	109	8.3	202	23.7	206	24.0	94	185	196

W_v = total volume of solvent adsorbed per cartridge

T = 50% breakthrough time

Furthermore, the results in Table 1 show that for a given solvent, the cartridge adsorption capacity increases with solvent concentration, which is due to the relative pressure increasing with increasing contaminant concentration. This effect is important for acetone and less significant for m-xylene and styrene.

The Effect of Water

The difference in the adsorption capacity of the cartridges with acetone, m-xylene, and styrene may be interpreted in part as being due to the difference in solvent volatility. Furthermore, a simultaneous adsorption of water (air humidity is 10%) and solvent occurs. In fact, Nelson *et al.* (1976) demonstrated that the cartridge adsorption capacity with volatile water-soluble molecules like acetone decreases due to water adsorption. The water adsorption effect is more significant with more volatile solvents and especially with those that are water soluble. Inversely, with less volatile hydrophobic molecules, the adsorption of water molecules is very low. Nelson's cartridge tests were performed at 50% RH.

Despite the fact that in the present study the tests were performed at 40% RH, it was demonstrated that a significant amount of water is adsorbed in the cartridge in the first minutes following the moment at which the cartridge is first exposed to the contaminated air. The experiments we performed consisted in exposing the cartridge to the contaminant at 40% RH. A humidity sensor was placed inside the chamber and another sensor was placed at the outlet of the

cartridge in order to measure the humidity of the air coming out of the cartridge. One example of a test performed with 750 ppm m-xylene is shown in Figure 3.

Signal 1 in Figure 3 corresponds to the humidity of the air inside the exposure chamber, and signal 2 is the humidity of the air at the cartridge outlet. The beginning of the test (start) corresponds to the time at which the cartridge was installed on the test bench. The signal corresponding to air coming out of the cartridge shows that, at first, the air humidity decreases significantly. This is due to water molecules which are being retained by the dry activated carbon. Subsequently, the air humidity increases (water breaks through), exceeds the humidity of the air in the chamber, and reaches a plateau at its maximum concentration, to then decrease when the m-xylene breaks through the cartridge. The most probable interpretation is that the increase in humidity is because m-xylene displaces the water molecules adsorbed on the surface of the activated carbon.

Recently, Hall and Holmes (1991) published the results of a study of the degradative effect of water adsorption on the adsorption capacity of cartridges at different relative humidities from 40% to 85%. The contaminant was a hydrophobic molecule, trichloronitromethane (B.P. 112.3°C). They demonstrated that at 40% RH, a significant amount of water was adsorbed in the preconditioned cartridges (approximately 20% of the water adsorbed at 65% RH). Furthermore, the cartridge

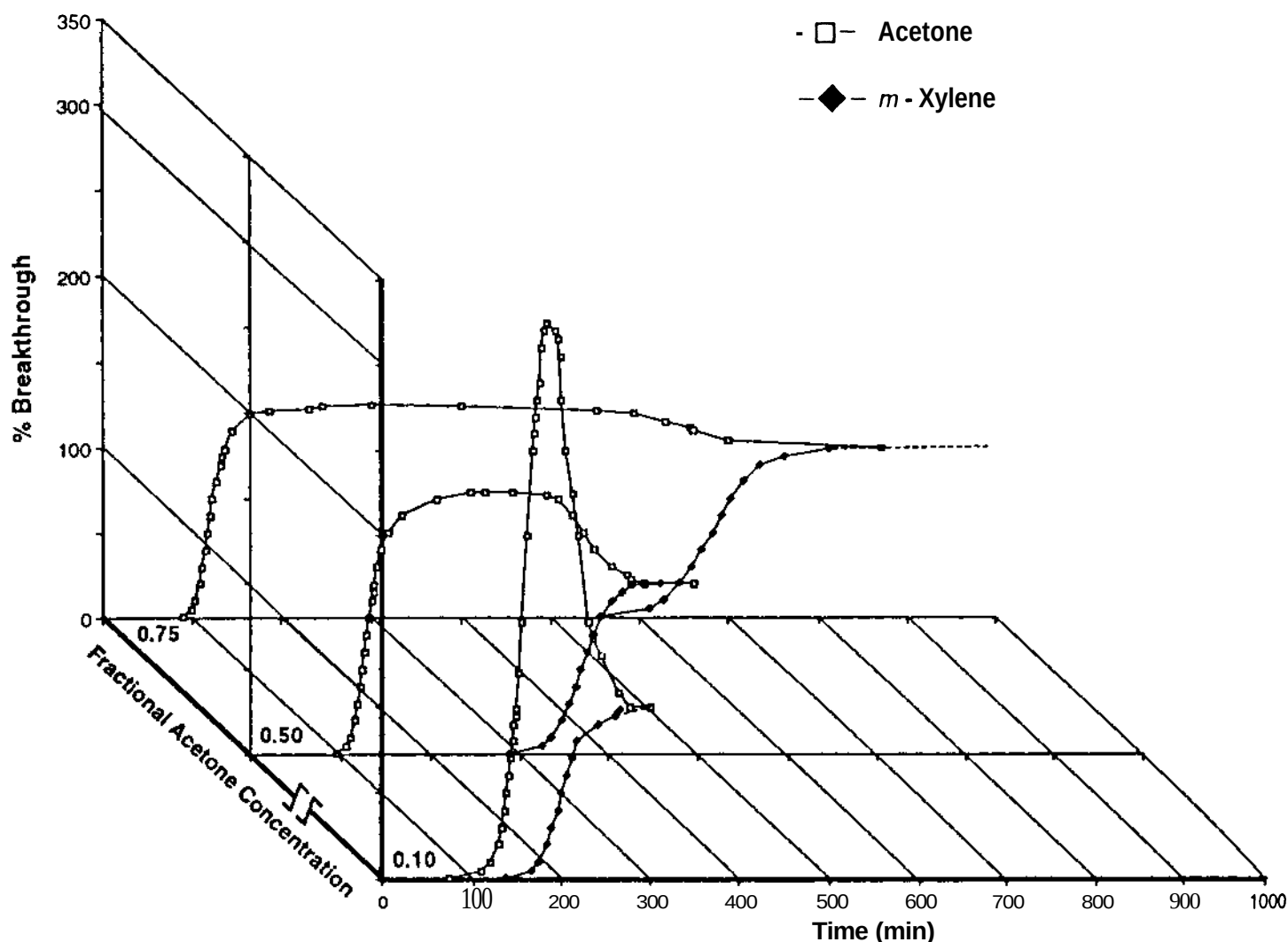


Figure 3. Water desorption curve. Numbers 1 and 2 in the figure correspond respectively to the chamber air humidity and the humidity of the air exiting from the cartridge. Start corresponds to the moment at which the cartridge was installed in the experimental chamber.

tests performed at a contaminant concentration of 5000 mg/m^3 (approximately 750 ppm) demonstrated that the adsorbed water was completely displaced from the carbon surface. The trends of the curves for the displaced water reported in the Hall and Holmes study are similar to that reported in Figure 3.

These results support our interpretation that water has a negative effect on acetone adsorption, whereas *m*-xylene and styrene, which are less volatile hydrophobic molecules, displaced the adsorbed water. However, Hall and Holmes demonstrated that the adsorption of less volatile hydrophobic molecules can be affected by water

adsorption at the highest RH's (for example, at 80% RH, the cartridges lost approximately 80% of their performance as compared with dry carbon).

3.1.2 The Binary Acetone/*M*-Xylene and Acetone/Styrene Systems

Cartridge service life was characterized with acetone/*m*-xylene and acetone/styrene mixtures at the compositions indicated in Table 2 and Table 3. In Figure 4, examples of the breakthrough curves for the 109/891 and 749/260 ppm mixtures of acetone/*m*-xylene, which correspond respectively to a fractional acetone concentration of 0.10, 0.50 and 0.75, are presented.

Table 2. Breakthrough values for the acetone/*m*-xylene system.

Concentration (ppm)		X_{acet}	Adsorption capacity (cm ³ /c)		10% breakthrough time (min)		Cm	η
acetone	<i>m</i> -xylene		acetone	m-xylene	acetone	m-xylene		
Part I. Flow rate of 24 L/min								
109	891	0.109	1.3	21.7	119	173	355	3.6
248	730	0.254	2.8	22.4	128	232	610	2.1
474	490	0.492	4.6	21.9	110	335	724	2.0
749	260	0.742	6.7	21.4	103	629	914	1.6
813	90	0.900	7.1	17.5	97	1448	871	1.6
Part II. Effect of variable acetone concentration. Flow rate of 36 L/min.								
96	503	0.160	1.5	23.0	140	227	280	2.7
260	496	0.344	3.4	24.5	105	240	491	2.1
432	520	0.454	4.7	-	85	-	740	1.7
758	500	0.600	6.2	22.8	64	229	1070	1.6

Table 3. Breakthrough values for the acetone/styrene system.

Concentration (ppm)		X_{acet}	Adsorption capacity (cm ³ /c)		10% breakthrough time (mm)		Cm	η
acetone	styrene		acetone	styrene	acetone	styrene		
Part I. Flow rate of 24 L/min								
97	892	0.098	1.1	22.1	125	197	353	3.5
234	739	0.240	2.2	21.6	114	245	492	2.9
249	755	0.248	2.5	23.0	111	247	513	2.9
479	507	0.486	4.8	21.2	109	330	763	1.8
746	256	0.745	6.8	22.2	106	698	895	1.7
Part II. Effect of variable acetone concentration. Flow rate of 24 L/min								
92	508	0.153	1.6	21.1	190	344	258	3.1
99	516	0.161	1.8	21.9	172	347	280	2.9
305	503	0.377	3.8	21.6	141	359	552	2.0
479	507	0.486	4.8	20.8	104	366	763	1.8
779	510	0.604	6.6	21.9	97	350	1080	1.7
998	506	0.665	7.3	22.3	85	368	1340	1.5
Part III. Effect of variable styrene concentration. Flow rate of 24 L/min								
479	507	0.486	4.8	20.8	104	366	763	1.8
492	733	0.402	4.4	22.0	99	247	926	1.7
494	1036	0.323	4.1	23.7	93	186	1111	1.7
490	1578	0.238	3.3	23.5	75	122	1470	1.7

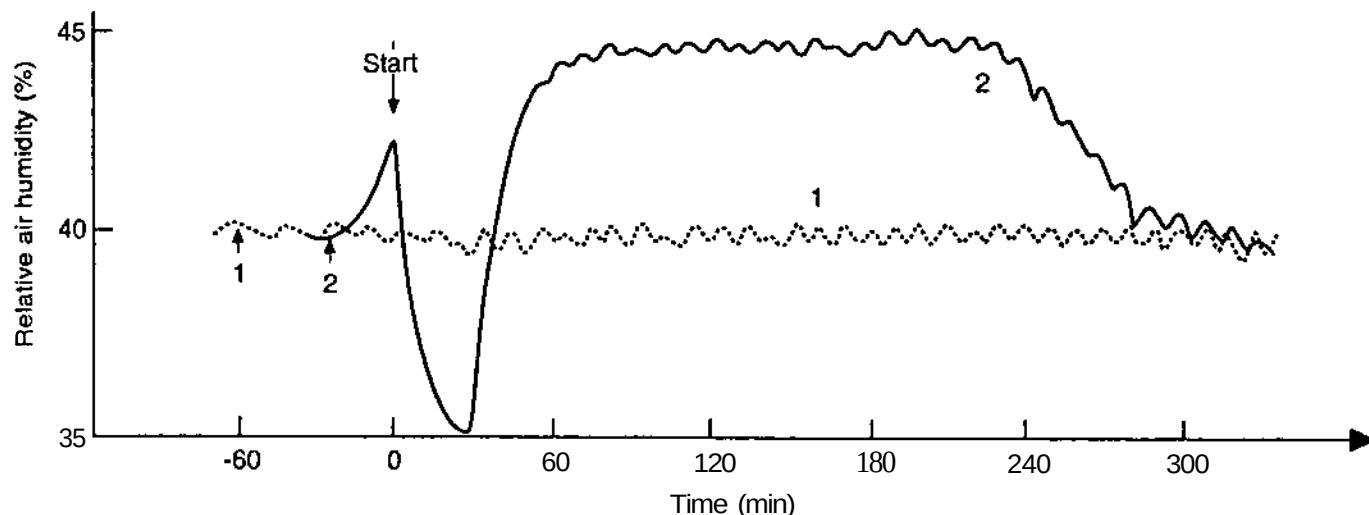


Figure 4. Three-dimensional representation of breakthrough curves for the acetone/*m*-xylene mixtures with a fractional acetone concentration of 0.10, 0.50 and 0.75.

The first curve shown in Figure 4 corresponds to the 109 ppm/891 ppm acetone/*m*-xylene mixture. From this curve, it can be seen that the most volatile solvent, acetone, breaks through first, despite the fact that it is present at a low concentration. The breakthrough concentration increases rapidly, to greatly exceed the acetone concentration in the exposure chamber. The maximum breakthrough concentration for acetone is reached around 355 ppm, more than 3 times the concentration in the exposure chamber. When *m*-xylene breaks through the cartridge, its concentration increases at the same time that the acetone breakthrough concentration decreases. When the *m*-xylene breakthrough concentration reaches the exposure concentration (100% breakthrough), the acetone breakthrough concentration decreases, to also reach the acetone's chamber concentration.

The second curve in Figure 4 corresponds to the 474 ppm acetone and 490 ppm *m*-xylene mixture, which is a 0.50 fractional acetone concentration. In this case, the acetone breaks through first, reaches a maximum at a concentration equivalent to 1.5 times the exposure concentration, and remains stable for a long period of time (more than one hour). The decrease in the acetone breakthrough concentration begins at the same time as the *m*-xylene breaks through.

Finally, the third curve presented in Figure 4 represents the breakthrough of the contaminants at a fractional acetone concentration of 0.75, which represents the 749 ppm acetone and 260 ppm *m*-xylene mixture. In this case, the acetone's maximum breakthrough concentration reached a

plateau at approximately 1.2 times the exposure concentration, and the breakthrough concentration remained constant for a very long period (around 8 hours). The start of *m*-xylene breakthrough coincided with the beginning of the decrease in the acetone breakthrough concentration.

The most probable interpretation is the following: for the acetone/*m*-xylene system, when the cartridges are exposed to the binary mixture in humid air, the acetone and *m*-xylene mixture (and also water) are adsorbed simultaneously on the activated carbon. When acetone begins to break through, the situation inside the cartridge would be the following: all the carbon surface is occupied by acetone and *m*-xylene (also water). To be adsorbed, the *m*-xylene must displace the adsorbed acetone molecules (also water molecules). The concentration of the acetone breaking through the cartridge is given by the sum of the acetone in the exposure chamber plus the acetone displaced from the surface of the carbon by the *m*-xylene. This phenomenon justifies the fact that the acetone breakthrough concentration exceeds the concentration in the exposure chamber. The same interpretation can be given for the acetone/styrene system.

The adsorbed acetone in the studied binary systems is completely displaced by the *m*-xylene or styrene. In fact, no acetone was detected in the analysis of the contaminant content on the carbon after completion of the experiments, using method No. 1300 of the National Institute for Occupational Safety and Health (1984). This method can detect 0.2 mg of acetone per gram of carbon, which corresponds to approximately 10 mg per cartridge.

The plateau observed at the maximum acetone breakthrough concentration probably represents an equilibrium in the *m*-xylene- or styrene-adsorption/acetone-desorption process.

Adsorption Capacity of the Cartridges in the Binary Systems

The adsorption capacity of the cartridges for each of the solvents in the acetone/*m*-xylene and acetone/styrene mixtures was calculated using Eq. (1). For acetone, the adsorption capacity was calculated from its 50% breakthrough time. At this moment, the *m*-xylene or the styrene still have not started to break through. These results are reported in Table 2 and Table 3.

It has already been shown that the cartridge's capacity with respect to acetone increases with the solvent's increasing concentration. The results in Tables 2 and 3 show that the capacity of the cartridge to adsorb acetone in the binary system also increases when the acetone concentration increases. However, the adsorption capacity is lower than that observed with pure acetone. Jonas *et al.* (1983) stated that the decreasing adsorption capacity of a solvent in a binary mixture should be proportional to the solvent mole fraction composition. However, this statement is not applicable to systems where the desorption phenomenon of the more volatile solvent is extremely important, as demonstrated in the present study.

On the other hand, the results in Tables 2 and 3 demonstrated that the presence of acetone has no significant effect on the capacity of the cartridge to adsorb *m*-xylene or styrene for all the studied solvent mixture compositions.

Variable Concentration Effect

The results of the effect of the variable acetone concentration on breakthrough times are presented in part II of Tables 2 and 3. In this case, the *m*-xylene or the styrene concentration was maintained at approximately 500 ppm and the acetone concentration was varied from approximately 100 to 1000 ppm. The results in these tables demonstrate that the acetone cartridge adsorption capacity increases when the acetone concentration increases. However, the adsorption capacity is always lower than the one at an equivalent concentration of pure acetone. On the other hand, the cartridge adsorption capacity for *m*-xylene and styrene is not affected by increasing acetone concentration.

In Table 3, part III, the effect of the variable styrene concentration at constant acetone concentration on the cartridge solvent adsorption capacity is presented. In this case, the acetone concentration was maintained at approximately 500 ppm and the styrene concentration varied from approximately 500 ppm to 1570 ppm. The results in this table show that the adsorption capacity for the acetone decreases when the styrene concentration increases.

Substitution of Acetone

It is possible to determine the approximate number of molecules of the less volatile contaminant (*m*-xylene or styrene) that replace a molecule of acetone adsorbed on the surface of the activated carbon. It involves subtracting the maximum concentration reached by the acetone (C_m) from the acetone concentration in the exposure chamber (C_a). This difference represents the concentration of acetone coming from molecules displaced by the *m*-xylene or the styrene.

$$C_m - C_a = \text{concentration of desorbed acetone} \quad (2)$$

When the acetone breakthrough concentration equals the concentration in the exposure chamber, all the surface of the carbon could be assumed to be saturated by acetone, *m*-xylene, or styrene (and probably some water molecules). Any new molecule of the less volatile contaminant will have to displace acetone (or water) to adsorb onto the carbon surface. Let η be the ratio of the number of molecules of the less volatile contaminant necessary to displace one adsorbed molecule of the most volatile contaminant. If the exposure concentration of the less volatile contaminant is C_z , the relationship between the molecules of *m*-xylene or styrene replacing a molecule of acetone will be represented by:

$$\eta = \frac{C_z}{C_m - C_a} \quad (3)$$

$$= \frac{\text{m-xylene or styrene exposure concentration}}{\text{concentration of acetone desorbed}}$$

Tables 2 and 3 present the η ratios for the different compositions of the acetone/*m*-xylene or acetone/styrene mixtures calculated using Eq. (3). The results in these tables show that in the case of the acetone/*m*-xylene system, the number of molecules of *m*-xylene necessary to replace an adsorbed molecule of acetone varies from 3.6 to 1.6 when the acetone/*m*-xylene mixture concentration

changes from 109 ppm/891 ppm to 749 ppm/260 ppm respectively. In the case of the acetone/styrene system, these values vary from 3.5 to 1.6 when the mixture concentration changes from 97 ppm/892 ppm to 746 ppm/256 ppm. At an equivalent concentration of acetone and *m*-xylene (or styrene), the η ratio is 1.6 to 1.8, which is the case for the 479/490 ppm acetone/*m*-xylene or for the 479/507 ppm acetone/styrene binary mixtures.

Higher acetone concentrations in the mixture result in minor changes in the η value. This is clearly demonstrated in Tables 2 and 3, part II, in the variable concentration effect, and especially in Table 3, part III, in the variable styrene concentration effect. Thus, part II of Tables 2 and 3 shows that when the acetone concentration exceeds approximately 500 ppm, which is the case in the acetone/*m*-xylene 758/500 ppm mixture or the acetone/styrene 779/510 ppm and 985/496 ppm mixtures, almost no change in the η value is observed when the acetone concentration increases. On the other hand, part III of Table 3 shows that when the acetone concentration is maintained at approximately 500 ppm and the styrene concentration changed from 507 ppm through 1570 ppm, no change in η is observed. This is the case with the acetone/styrene 492/733, 494/1036 and 490/1570 ppm mixtures where the η value stays at 1.7.

An example of the substitution process is the following: when the acetone and *m*-xylene concentrations are 474 ppm and 490 ppm respectively, 180 molecules of *m*-xylene will displace 100 molecules of acetone adsorbed into the surface of the activated carbon.

3.1.3 Service Life at 10% Breakthrough

Tables 2 and 3 present the 10% breakthrough values for acetone and *m*-xylene respectively for the binary systems. The 10% breakthrough times for the pure solvents are presented in Table 1. Results demonstrate that the cartridge's service life with acetone is greatly affected by the presence of *m*-xylene or styrene. For example, the service life at 10% breakthrough for pure acetone at a concentration of 107 ppm is 296 min. In the binary system, when the concentration of acetone is 109 ppm and that of *m*-xylene 891 ppm, the cartridge's service life at 10% acetone breakthrough decreases to 119 min. Other information which may be drawn from Tables 2 and 3 is that the service life at 10% breakthrough with acetone varies very little when the acetone concentration increases from 109 ppm to more than 800 ppm in

the binary system. For all cases the 10% breakthrough time values for acetone in the binary system stay close to the value for pure acetone at 1000 ppm, namely 97 min.

The effect of a variable acetone concentration (part II of Tables 2 and 3) or a variable styrene concentration (part III of Table 3) causes a decrease in the 10% breakthrough time for the acetone.

In the case of the less volatile contaminant, Tables 2 and 3 show that acetone has little effect on the 10% breakthrough time of *m*-xylene or styrene. This effect is more remarkable when the acetone concentration is varied as shown in Part II of these tables. For example, when the acetone concentration changes from 92 ppm through 985 ppm and the styrene concentration is maintained at approximately 500 ppm, the 10% breakthrough time for styrene always stays at a value close to the one for pure styrene at 500 ppm (Table 3, part II). This suggests that the entire carbon surface is occupied by *m*-xylene or styrene.

The results presented above demonstrate the existence of such an additive concentration effect on the acetone 10% breakthrough time. To estimate this 10% breakthrough time for the binary systems with *m*-xylene or styrene, the equivalent acetone concentration should be calculated with the following equation:

$$C_e = C_1 + C_2/\eta \quad (4)$$

where

- C_e = the equivalent acetone concentration
- C_1 = the acetone concentration
- C_2 = the *m*-xylene or styrene concentration
- η = the replacement ratio obtained at equilibrium (when the plateau in the acetone desorption curve is reached)

Using the calculated equivalent acetone concentration, the 10% breakthrough time can be estimated from the type of 10% breakthrough curve presented in Figure 2 for pure acetone. For example, for the 248/730 ppm acetone/*m*-xylene mixture, using the η value of 1.6 (the η value reported in Table 2 is 2.1, which was obtained when the acetone-desorption/*m*-xylene-desorption does not still reach an equilibrium), the equivalent concentration is 704 ppm and the experimental 10% breakthrough time is 128 min. The experimentally obtained 10% breakthrough time for pure acetone at an equivalent concentration, 726

ppm (from Table 1), is 122 min. Another example is the 779/510 ppm acetone/styrene mixture where the experimentally obtained 10% breakthrough for acetone is 97 min. The acetone equivalent concentration is 1079 ppm using the h value of 1.7. At an equivalent pure acetone concentration of 1060 ppm, the 10% breakthrough time obtained is 97 min. (Table 3, part II).

3.2 The Toluene/*M*-Xylene System

The evaluation of the service life of cartridges for toluene and *m*-xylene in the single and binary systems was carried out at a flow of 36 L/min. These experiments were performed with two cartridges simultaneously.

3.2.1 The Single System

With pure toluene, the tests were performed at concentrations of 244 ppm, 550 ppm, 773 ppm, and 1006 ppm. In the case of *m*-xylene, the tests were repeated at the new flow at concentrations of 247 ppm, 571 ppm, 747 ppm, and 1000 ppm. The 10% breakthrough time values for toluene and *m*-xylene are presented in Table 4. The values for toluene and *m*-xylene are very close, being slightly lower for toluene.

The cartridge adsorption capacity values for pure solvents calculated using Eq. (1) are presented in Table 5. These results show that the adsorption capacity of the cartridges (in terms of contaminant weight per cartridge or the adsorp-

tion space occupied by each contaminant) is slightly higher for *m*-xylene than for toluene. Toluene is more volatile than *m*-xylene.

3.2.2 The Binary System

The breakthrough of contaminants with the toluene/*m*-xylene mixture was characterized at the following concentrations: 95 ppm/875 ppm, 242 ppm/740 ppm, 516 ppm/507 ppm, 747 ppm/247 ppm, and 886 ppm/97 ppm. In all cases, since toluene is the more volatile solvent, it breaks through the cartridge first.

Figure 5 represents the breakthrough curves for the 95/875, 516/507, and 886/97 ppm mixtures, which correspond respectively to a fractional toluene concentration of 0.10, 0.50 and 0.75. The first curve represents the breakthrough when the fraction of toluene is 0.10. In this case, the breakthrough times for the two solvents are very close. For example, at 10% breakthrough, the service life is 100 min for toluene, and 112 min for *m*-xylene. The breakthrough concentration for toluene increases very rapidly, to exceed the concentration in the exposure chamber. The maximum breakthrough concentration for toluene is around 3.4 times the exposure concentration. At this composition of the mixture, *m*-xylene breakthrough occurs much before the breakthrough concentration of toluene reaches its maximum level. The same phenomenon was observed for the mixture composed of 242 ppm/740 ppm of toluene/*m*-xylene. The plateau, which

Table 4. Adsorption capacity and 10% breakthrough times for the toluene and *m*-xylene single systems. Flow rate of 36 L/min

Concentration (ppm)		Cartridge adsorption capacity				10% breakthrough time (min)	
toluene	<i>m</i> -xylene	toluene		<i>m</i> -xylene		toluene	<i>m</i> -xylene
		T (min)	W_v (cm ³ /c)	T (min)	W_v (cm ³ /c)		
244	247	522	19.9	557	24.9	484	509
513	571	273	21.8	241	24.9	239	206
746	747	204	23.4	189	25.6	187	173
773	-	206	24.4	-	-	179	-
1138	1093	147	26.0	118	23.4	135	108

Table 5. Breakthrough values for the toluene/*m*-xylene system.

Concentration (ppm)		X _{tol}	W _r (cm ³ /c)		10% breakthrough tune (min)		C _m	η
toluene	<i>m</i> -xylene		toluene	m-xylene	toluene	m-xylene		
Part I. Flow rate of 36 L/min.								
95	875	0.098	1.6	20.8	100	112	320	3.9
245	725	0.253	5.2	23.6	127	148	720	1.5
242	740	0.246	5.1	23.4	126	149	800	1.3
516	507	0.504	11.0	20.3	125	184	1039	1.0
747	247	0.752	17.1	17.4	137	303	994	1.0
886	97	0.901	19.9	10.2	128	422	983	1.0
Part II. Effect of variable toluene concentration. Flow rate of 36 L/min.								
94	507	0.156	2.9	21.3	182	205	310	2.3
249	510	0.328	6.5	21.4	154	194	650	1.3
516	507	0.504	11.0	20.3	125	184	1039	1.0
757	505	0.600	12.9	17.7	98	156	1275	1.0
1026	524	0.662	14.5	17.9	84	145	1550	1.0

probably corresponds to the equilibrium in the adsorption-desorption process, was not observed with the 95/875 and 242/740 ppm mixtures.

The second curve presented in Figure 5 corresponds to the mixture composed of 516 ppm toluene and 507 ppm *m*-xylene (toluene fraction of 0.50). In this case, the service life at 10% breakthrough is 100 min for toluene, and 180 min for *m*-xylene. The maximum breakthrough concentration for toluene corresponds to approximately 2 times the exposure concentration. The beginning of the decrease in toluene breakthrough concentration coincides with the start of *m*-xylene breakthrough. However, once the maximum toluene breakthrough concentration is reached, the concentration decreases without reaching the plateau.

The third curve corresponds to the 747 ppm/247 ppm toluene/*m*-xylene mixture. In this case, a plateau is observed at the maximum toluene breakthrough concentration. The maximum

toluene breakthrough concentration corresponds to approximately 1.4 times the exposure concentration, and the duration of the plateau is around two hours.

The Adsorption Capacity of the Cartridges in the Binary System

The adsorption capacity of cartridges for toluene and *m*-xylene was calculated using Eq. (1) and the results are presented in Table 5. In this case, the cartridge adsorption capacity for toluene was calculated from the 50% breakthrough time value, independent of the fact that in several mixtures at this time, the *m*-xylene has already broken through the cartridge. The results show that the cartridge's adsorption capacity with respect to either of the two solvents in the binary system depends to a great extent on the toluene/*m*-xylene concentration ratio. When the concentration of one of the solvents increases, the cartridge's adsorption capacity with respect to the other solvent decreases. However, it appears that

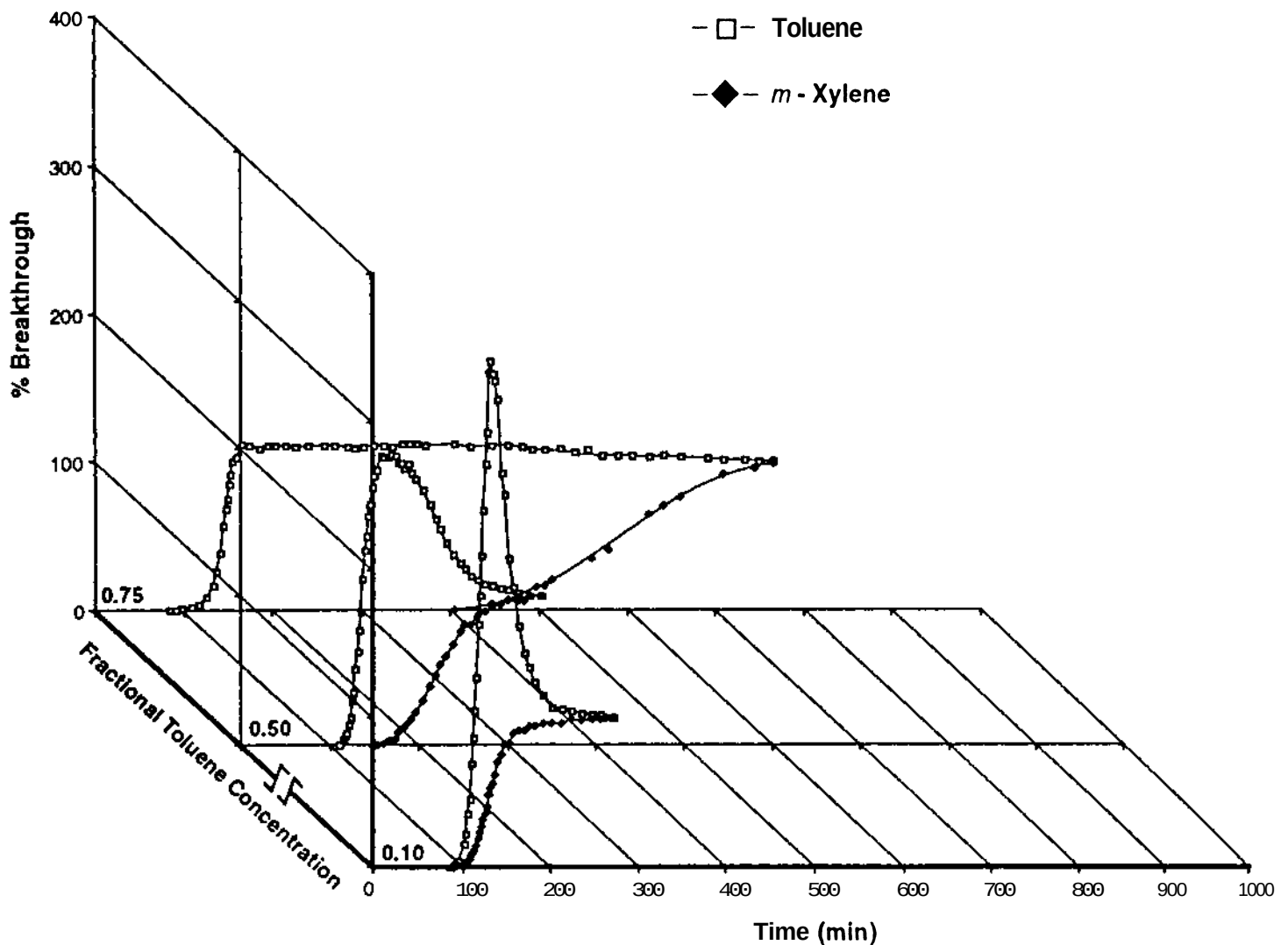


Figure 5. Three-dimensional representation of breakthrough curves for the toluene/m-xylene mixtures with a fractional toluene concentration of 0.10, 0.50 and 0.75.

the effect is much more marked for toluene than for m-xylene. Hence, when the toluene and m-xylene concentrations are nearly equivalent, as for example in the case of a mixture of 516 ppm toluene and 507 ppm m-xylene, the cartridge's adsorption capacity is 9.5 cm³ for toluene (compared to 21.8 cm³ with pure toluene at 513 ppm), while it is 17.5 cm³ for m-xylene (compared to 24.9 cm³ with pure m-xylene at 571 ppm). When the toluene is at lower concentration, as in the case of the mixture of 242 ppm toluene and 740 ppm m-xylene, the adsorption capacity is 5.1 cm³ of toluene (compared to 19.9 cm³ with pure toluene at 244 ppm), whereas the adsorption capacity is 23.4 cm³ for m-xylene. Finally, when the m-xylene is at low concentration, which is the case in the mixture of 747 ppm toluene and 247 ppm m-xylene, the

cartridge's adsorption capacity is 17.1 cm³ for toluene (compared to 23.4 cm³ with pure toluene at 746 ppm), and 17.4 cm³ for m-xylene (compared to 24.9 cm³ with pure m-xylene at 247 ppm).

The most probable interpretation is the following: the two solvents would adsorb simultaneously at the carbon surface. When the activated carbon is saturated, the m-xylene displaces the adsorbed molecules of toluene. However, this displacement would only be partial and would depend on the concentration ratio of the binary system.

To have an approximate idea of the quantity of toluene retained in the cartridge, the following assumption could be made: the difference be-

tween the total quantity of *m*-xylene adsorbed in the single system (W_{txyl}) and the quantity adsorbed in the binary mixture (W_{xyl}) represents the quantity of *m*-xylene not adsorbed by the cartridge because of toluene (ΔW_{xyl}).

$$W_{txyl} - W_{xyl} = \Delta W_{xyl} \quad (5)$$

where

W_{txyl} = total adsorption capacity for pure *m*-xylene

W_{xyl} = adsorption capacity for *m*-xylene in the binary system

Given that there is a difference between the adsorption capacity for toluene and that for *m*-xylene, ΔW_{xyl} must be multiplied by a factor W_{ttol}/W_{txyl} to obtain the amount of toluene remaining in the cartridge after saturation with *m*-xylene.

$$\Delta \Omega_{tol} = \Delta W_{xyl} \times \frac{W_{ttol}}{W_{txyl}} \quad (6)$$

where

W_{tol} = grams of toluene adsorbed in the binary system

W_{ttol} = total adsorption capacity for pure toluene

Using Eq. (6), the amounts of toluene that remain once the cartridge is saturated with the binary mixture were calculated. These values are presented in Table 6. This table shows that almost all the toluene is desorbed at the lowest toluene/*m*-xylene concentration ration studied. However, the quantity of residual toluene in the cartridge increases with an increasing toluene concentration.

To verify the validity of this statement, the calculated weight of *m*-xylene retained by the cartridge in the binary mixture presented in Table 5 was added to the weight of toluene remaining in the cartridge presented in Table 6. This value is compared to the quantity determined experimentally by weighing the cartridge at the end of each test. These results are also presented in Table 6.

The results in Table 6 show the quantities of solvent retained by the cartridge (calculated from the breakthrough time results) and the quantities determined by weighing are very close. Furthermore, these results support the interpretation given for the adsorption of solvents in the binary system.

Substitution of Toluene by *M*-Xylene

To obtain information on the number of molecules of *m*-xylene displacing the molecules of adsorbed toluene, calculations were carried out using Eq. (3) with the toluene/*m*-xylene mixtures. These results are presented in the last column in Table 5. This table shows that when the toluene concentration is lower than 500 ppm, which is the case for the 94/507, 95/875, 242/740, 245/725, and 249/510 ppm toluene/*m*-xylene mixtures, the substitution value η decreases from 3.9 to 1.3 when the toluene concentration increases. When the toluene concentration in the mixture is approximately 500 ppm or higher, η reaches a value of 1.0. This value does not change when the toluene concentration exceeds 500 ppm as observed in the toluene/*m*-xylene 516/507, 747/247, 886/97, 757/505, 1029/524 ppm mixtures.

Table 6. Weight of remaining toluene ($A W_{tol}$) calculated from equation 5, and calculated and measured total weight of toluene and *m*-xylene adsorbed after cartridge saturation with mixtures

Concentration (ppm)		ΔW_{tol} (g/c)	Measured weight (grams)	Calculated weight (grams)	% Dev.
Toluene	<i>m</i> -xylene				
242	740	1.1	21.6	21.3	1.4
245	724	0.9	*	21.3	-
501	506	2.8	21.3	21.1	1.0
516	507	3.3	*	21.0	-
747	247	5.8	21.8	20.8	4.7
886	97	11.4	20.5	20.2	1.4

$$W_{tol}/W_{txyl} = 0.91$$

$$W_{txyl} = 21.34$$

* = experiment not completed at 100% *m*-xylene breakthrough

These results demonstrated that when the toluene concentration is higher than approximately 500 ppm, one molecule of m-xylene displaces one molecule of toluene.

3.2.3 Variable Concentration Effect

The effect of varying the concentration of one solvent in relation to the other was verified by keeping the m-xylene concentration constant at approximately 500 ppm and by varying that of toluene between approximately 100 ppm and 1000 ppm. The results obtained are presented in Table 5, part II. This table shows that the adsorption capacity for m-xylene decreases when the toluene concentration increases. Thus, the cartridge adsorption capacity for m-xylene decreases from approximately 21 cm³ when toluene is at 94 ppm to approximately 17.9 cm³ when the toluene concentration is 1026 ppm, as compared to 24.9 cm³ for pure m-xylene.

The effect of variable toluene concentration on the 10% breakthrough time of m-xylene is that its value decreases when the toluene concentration increases. For example, when the toluene concentration is 94 ppm, the 10% breakthrough time for m-xylene is 205 minutes, and when the toluene concentration increases to 1026 ppm, this value decreases to 145 minutes. At the same time, the 10% breakthrough time for toluene decreases when its concentration increases. For example, when the toluene concentration in the mixture is 249 ppm, its 10% breakthrough time is 154 minutes, as compared to 484 minutes for pure toluene at 244 ppm.

The effect of variable toluene concentration demonstrated that the substitution ratio η of the adsorbed toluene by m-xylene is a function of the toluene concentration rather than the concentration ratio of the mixture. This behavior is similar to the one observed in the previously discussed mixtures with acetone.

Service Life at 10% Breakthrough

The 10% breakthrough time values for toluene and m-xylene are presented in Table 4 for the single solvents and in Table 5 for the binary mixtures. In the case of the single toluene or m-xylene systems, the 10% breakthrough time increases exponentially with decreasing solvent concentration in a similar manner to those represented in Figure 2 for pure acetone. However, in the case of the binary mixture, the 10% breakthrough time for toluene remains almost constant at any toluene/m-xylene concentration ratio.

These results demonstrate that the toluene and m-xylene concentrations are additive with respect to the toluene 10% breakthrough time. In other words, when the composition of the mixture is, 250 ppm toluene and 750 ppm m-xylene, the breakthrough time for toluene would be that of pure toluene at a concentration of 1000 ppm. This justifies the fact that the value of 10% breakthrough time for toluene remains practically unaffected at the different toluene/m-xylene concentration ratios.

On the other hand, the 10% breakthrough times for m-xylene in the binary system are shorter than those obtained with the single solvent. This means that in this case, the service life with m-xylene is greatly reduced by the presence of toluene, which differs from the observed effect of acetone on the breakthrough of m-xylene.

The most interesting information presented in Table 5 is that the 10% breakthrough time for toluene remains almost constant at all concentration ratios. In the case of m-xylene, however, the 10% breakthrough time increases when the concentration decreases.

To estimate the 10% breakthrough time for toluene in the binary system, the equivalent toluene concentration can be calculated using Eq. (4). However, given that for this system the η value is 1.0, the additive concentration effect on the toluene's 10% breakthrough time applies.

4. Conclusion

Ten-percent breakthrough time values were used to compare the service life of the respirators. Beyond 10%, the concentration of the contaminant that breaks through the cartridge increases very rapidly. This is critical in the case of binary mixtures of contaminants, because the break-

through concentration of the most volatile component increases even more rapidly than in the case of the pure solvent, and may exceed by several times the concentration of the contaminant in the air.

The results of this study have demonstrated that the solvents with the lower volatility, namely styrene and m-xylene, are the ones retained to the greatest extent in the respirator cartridges. The next is toluene, and lastly, the solvent least retained by the cartridges is acetone, which is the most volatile.

For the binary systems studied, it was found that the service life of respirator cartridges is determined by the breakthrough time for the more volatile contaminant. This contaminant always breaks through first, and the breakthrough concentration may exceed the contaminant's exposure concentration by several times.

The fact that the more volatile contaminant's breakthrough concentration exceeded the exposure concentration was interpreted in terms of displacement of the adsorbed volatile contaminant by the less volatile one at the carbon surface. The concentration of the contaminant desorbed from the surface of the activated carbon is added to the concentration of the contaminant in the air.

In the case of the acetone/m-xylene and acetone/styrene systems, it was shown that all the acetone adsorbed on the activated carbon was totally displaced by the m-xylene or the styrene. The displacement ratio of m-xylene or styrene is approximately 1.6 to 1.7. In the case of the toluene/m-xylene system, it was shown that

molecules of m-xylene displaced adsorbed molecules of toluene, but that this substitution was only partial. The displacement ratio for this system is 1.0.

The concentration effect on the 10% breakthrough time of the most volatile contaminant is additive and related to the substitution ratio. When the substitution ratio is 1, which is the case in the toluene/m-xylene mixture, the concentration of the solvents can be added. The resulting concentration should be attributed to toluene. In this case, to estimate the service life of cartridges at 10% breakthrough, the experimental information obtained with pure toluene can be used. For the acetone/m-xylene or acetone/styrene mixtures, the concentration of the less volatile contaminant (m-xylene or styrene) must be converted into an equivalent acetone concentration using the substitution ratio. The acetone concentration and the acetone equivalent concentration are added and the resulting concentration is attributed to the acetone. Then, the 10% breakthrough time is estimated from the 10% breakthrough time curve experimentally obtained with pure acetone.

Consequently, the service life data obtained with pure solvents could be used to predict the service life of respirator cartridges in an industrial environment, provided that the concentrations of the contaminants and the humidity are known.

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