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RESPIRATOR CARTRIDGE EFFICIENCY STUDIES: VII. EFFECT OF RELATIVE HUMIDITY AND TEMPERATURE

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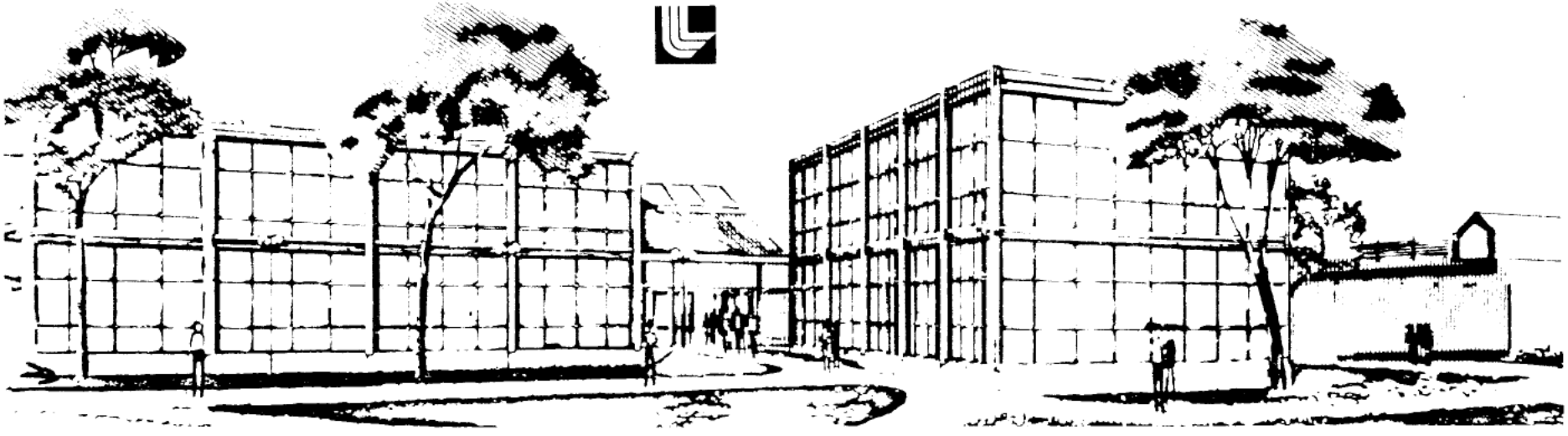

Abstract

Storage and use relative humidity above 65% greatly decreases cartridge service life. Temperature, however, does not significantly affect cartridge performance.

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RESPIRATOR CARTRIDGE EFFICIENCY STUDIES: VII. EFFECT OF RELATIVE HUMIDITY AND TEMPERATURE*

Introduction

This is the seventh paper in a series of reports assessing the service life of organic vapor respirator cartridges. We previously described our program goals,[1] testing apparatus,[2] development of a mechanical breathing simulator,[3] comparison of steady-state and pulsating flow,[4] and the effects of solvent vapor[5] and concentration.[6] This report describes how the service life varies with relative humidity.

Although activated carbon traditionally exhibits a minimal affinity for water vapor, preliminary tests have shown that high humidities can significantly depress the effective service life.[6] This investigation will show to what extent both the storage and use humidity play in cartridge performance for seven solvent vapors. The observed breakthrough times will also be compared with those calculated from the adsorption isotherm and the Mecklenburg and modified Wheeler equations. The effect of temperature will also be calculated from these expressions.

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Summary of Previous Results

Effect of Flowrate

Cartridge life is inversely proportional to flowrate. If the flowrate is doubled, the breakthrough time will be halved. A mechanical breathing simulator was designed to measure differences between pulsating and steady-state flow patterns. No significant difference was observed between these two techniques at concentrations of 1000 ppm and flowrates between 14 and 71.4 liters/min.

Effect of Solvent Vapor

Breakthrough curves were generated for 131 different solvent vapors at 1000 ppm. The service life cannot be attributed to a single solvent vapor property, but is a complex function of the nature of the solvent vapor and the adsorbing media as well as the experimental conditions. In general, the less volatile materials are adsorbed to a greater degree than those solvents with low boiling points.

Effect of Concentration

Experiments showed that the time to any chosen breakthrough percent (t_b) conformed to the empirical expression $t_b = aC^b$, where C is the concentration and a and b are constants for a given set of experimental conditions. The average value for b at 10% breakthrough was determined

to be -0.67. This indicates that if the concentration is reduced by a factor of 10, the service life will be extended by a factor of 4 or 5. The constant b is not affected by flowrate, but can be altered to some extent by relative humidity.

Calculation of Breakthrough Times

Calculation of breakthrough times using the adsorption isotherm and Mecklenburg equation agree reasonably well with experimental values below 20% breakthrough. Although calculations of this type are somewhat complicated, use of the modified Wheeler equation discussed later in this paper simplifies the mathematical operations. To date no satisfactory method has been found to predict the entire "S" shaped breakthrough curve or to account for the presence of water vapor.

Experimental

Apparatus

Reference 5 shows the apparatus generating and monitoring the test concentrations. Compressed air passes into a water reservoir where it is automatically humidified by a humidity controller. The air flow is measured with a mass flowmeter. A modified reciprocal syringe pump injects the solvent that is vaporized by pumping the liquid through a stainless steel needle in contact with a heated aluminum block. We have

experimented with several other types of pulseless high-pressure liquid chromatography pumps (10-5000 μliters/min range) and found them to be satisfactory. Two flame ionization detectors monitor the upstream and downstream solvent vapor concentration.

Water adsorption and desorption characteristics of the cartridges were determined with the apparatus shown in Figure 1. Here, humidification was accomplished in the usual manner and another relative humidity detector was placed downstream from the cartridges. A recorder yielded the effluent relative humidity as a function of time.

Cartridge Specifications

Cartridges selected for testing were the Mine Safety Appliance Co. (MSA) Model 44135* and the American Optical Corp. (AO) Model R-51. In the previous reports these were referred to as the Type 1 and Type 3 cartridges. Their specifications are shown in Reference 6. For a given carbon base and mesh size the only characteristics that are likely to change between cartridge batch or lot number are the maximum volume of

*Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Energy Research and Development Administration to the exclusion of others that may be suitable.

the adsorption space, W_o, and the microporosity, B. These two carbon properties can be determined experimentally by the method outlined in Reference 6.

Cartridge Testing

All cartridges were vacuum desiccated at 110°C for at least 24 hours to remove moisture. They were then placed in a cabinet and preconditioned at the humidity of interest. It was originally thought that the cartridge should achieve equilibrium in at least 2 days. However, it was subsequently learned that at least 7-10 days were required at static storage humidity greater than 65%.

All cartridges were tested at 1000 ppm, 20°C and 53.3 liters/min at 0, 20, 50, 65, 80, or 90% relative humidity. Seven solvents were chosen to represent a cross section of vapors that could be encountered under field conditions. These materials included isopropanol, acetone, carbon tetrachloride, benzene, hexane, 1-chlorobutane, and ethyl acetate.

Results

Effect of Preconditioning Humidity

The results of the static preconditioning tests are shown in Figure 2 for MSA and AO cartridges. Note that as the humidity increases, the amount of water adsorbed increases exponentially. Most of the

cartridges tested contain about 5% by weight adsorbed water. However, if cartridges are stored in a relatively damp area for extended time intervals, moisture contents of 40% or greater can be reached.

Although considerable water can be adsorbed by the activated carbon, one might intuitively suspect that any organic vapors passing through the cartridge would displace the water vapor and be preferentially adsorbed. This in not the case, however, as shown in Figures 3 and 4. Here cartridges are subjected to 1000 ppm benzene and hexane at a 50% use humidity with varying preconditioning moisture conditions. Note that the breakthrough curves exhibit only a slight shift at humidities less than 50%. However, above 50% the initial breakthrough times are shortened considerably. Also note in the case of benzene that the characteristic asymmetric "s" shaped breakthrough curve was distorted almost to a straight line at 90% relative humidity. This shift and distortion can possibly be explained by competition of water vapor for the available adsorption site or by a temporary blockage of the micropores by condensed water vapor.

Figure 5 gives additional moisture adsorption information after the cartridge was saturated with solvent vapor. Here the weight of water adsorbed (calculated by the method outlined in Reference 2) is shown as a function of preconditioning humidity for seven solvents. Oddly enough the water content drops slightly until about 50% relative humidity. After 50%, however, the water content rises sharply and probably accounts for the decreased breakthrough times.

Effect of Use Humidity

In addition to the water normally taken up by the cartridge during storage, moisture can reenter the cartridge during field use and further modify its performance. This is demonstrated in Figure 6, which shows the breakthrough curves for 1-chlorobutane at use humidities ranging from 20 to 90%. The cartridges were all preconditioned at 50% relative humidity. The breakthrough curves for 20 and 50% are practically coincident but the 65, 80, and 90% curves show a substantial shift toward lower cartridge service life. Figure 7 shows the weight water adsorbed as a function of use humidity after the cartridge was saturated with solvent vapors. The moisture uptake is almost an exponential function of use humidity rather than the much flatter response shown for preconditioning humidity.

Combined Effect of Preconditioning and Use Humidity

The previous sections indicate that both the storage and use relative humidity tend to decrease service life at higher water vapor concentrations. Figure 8 shows a typical example of the combined effects on MSA cartridges preconditioned at 50, 65, or 80% and tested at 20, 50, 65, 80, or 90% relative humidity with 1000 ppm carbon tetrachloride at 53.3 liters/min. Note that in Figure 8a at 50% preconditioning the curves are only slightly separated. Figure 8b, at 65% preconditioning, shows a larger separation and a decrease in the breakthrough times. Figure 8c, at 80% preconditioning, shows the largest separation and breakthrough time decrease.

Figures 9 and 10 show the 10% breakthrough time as a function test humidity for various preconditioning humidities. These examples represent the cartridges most (carbon tetrachloride) and least (acetone) affected by moisture of the seven solvents tested.

A typical example of water uptake at differing equilibrium and use humidity is shown in Figure 11. Isopropanol as well as the six other solvents show that the test or use humidity plays a larger role in moisture adsorption than does the preconditioning humidity.

A summary of all the humidity data for the seven solvent vapors tested is shown in Table I. Here the data was normalized to the 50% preconditioning and use humidity. If the breakthrough time is known at the normalized conditions, service life times at other moisture conditions can be estimated by using the appropriate correction factor obtained from Table I.

It must be emphasized that these factors were determined from a rather small cross section of solvent vapors and cartridge types as well as only one concentration. At lower concentrations past data (Reference 6) indicate that these multiplication factors increase as the concentration decreases below 1000 ppm.

From these data several important facts emerge:

1. Both the preconditioning and use humidity alter the cartridge service life.
2. The use humidity has a greater effect than the preconditioning humidity.

3. Service life is approximately the same between 0 and 50% humidity.
4. Humidity has a greater effect on cartridge performance at lower concentrations.

Calculations

Estimations of the breakthrough time can be obtained from the adsorption isotherm and the Mecklenburg or modified Wheeler equations.[7,8]

The adsorption isotherm yields the maximum weight of solvent adsorbed at total carbon saturation and is calculated by

$$w_s = \rho W_o \exp\left[-\frac{BT^2}{\beta^2} \log (p_s/p)^2\right], \qquad (1)$$

where

w_s = equilibrium static adsorption capacity per unit weight of carbon (g/g)

ρ = density of solvent (g/cm^3)

W_o = total volume of the adsorption space (cm^3/g)

B = microporosity constant for the carbon

T = temperature (K)

β = affinity coefficient of solvent vapor for activated carbon

p_s = saturated vapor pressure for the solvent at the temperature T (Torr)

p = equilibrium partial pressure of the solvent vapor (Torr)

Calculations using the adsorption isotherm with the Mecklenburg equation are shown in some detail in References 5 and 6.

Use of the modified Wheeler equation to calculate service life times once the adsorption capacity is known has enjoyed considerable success. This equation takes the form

$$t_b = \frac{w_s}{C_o Q}\left[w_c - \frac{\rho_c Q}{k_v}(\ln C_I/C_b)\right], \qquad (2)$$

where

$$C_o = \frac{MC_I}{24.1 \times 10^9}, \qquad (3)$$

$$k_v = 14.4\, v_1^{1/2}\, d^{-3/2} = 14.4\, (Q/nA)^{1/2}\, d^{-3/2}, \qquad (4)$$

and

C_o = assault concentration (g/cm^3)

Q = flowrate (cm^3/min)

w_c = weight of carbon (g)

ρ_c = bulk density of carbon (g/cm^3)

k_v = adsorption rate constant (min^{-1})

C_I = assault concentration (ppm)

C_b = breakthrough concentration (ppm)

M = molecular weight (g/mole)

v_1 = superficial velocity across carbon particle (cm/min)

d = particle diameter (cm)

n = number of cartridges

A = cross sectional area of each cartridge (cm^2)

This expression uses the kinetic approach and is based on the use of the pseudo first-order adsorption rate constant.

A comparison of experimental and calculated breakthrough times are shown in Table 2. In general, the Table 2 values calculated from the Wheeler equation showed the best agreement.

Neither of these expressions, however, correct for the presence of water vapor and thus the deviation from the experimental values is larger at higher humidities.

Effect of Temperature

Our testing apparatus did not permit us to measure cartridge service life at temperatures other than 20-25°C. Many adsorption studies, however, were done at other temperatures using undiluted solvent vapors.[9] The adsorption isotherm calculation was proven both useful and accurate in predicting the behavior of activated carbon.

Figure 12 shows the calculated 10% breakthrough time for the seven solvents. In general, every 10°C rise in temperature will reduce the service life from 1-10% depending on the solvent in question.

Summary

The service life of organic vapor respirator cartridges tested at 1000 ppm solvent vapor and 53.3 liters/min is dramatically reduced at high

storage and use humidities. Below 50% relative humidity, the effect is relatively slight. This moisture effect becomes more noticeable, however, as the solvent vapor concentration decreases.

Using the modified Wheeler equation instead of the Mecklenburg equation greatly simplifies as well as improves the accuracy of breakthrough time calculation.

Temperature is not a dominant factor when estimating cartridge service life. Calculations indicate that the breakthrough times are diminished from 1-10% with each 10°C rise in temperature.

Acknowledgments

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TABLE I

Breakthrough time correction factor at various humidities at 1000 ppm, 53.3 liters/min, and 22°C for a pair of cartridges containing coconut or petroleum base carbon. The data was normalized to the 50% preconditioning and test relative humidity. The footnotes indicate the test vapor employed and the carbon type. The number in parentheses is the standard deviation.

TEST RELATIVE HUMIDITY (%)	BREAKTHROUGH TIME MULTIPLIER PRECONDITIONING RELATIVE HUMIDITY (%)					
	0	20	50	65	80	90
0	0.94 (0.08)[a,b]	0.95 (0.04)[a-c]	0.99 (0.07)[a-c]	0.97 (estimated)	0.95 (0.07)[a-c]	0.95 (-)[a]
20	1.02 (0.06)[a,b]	1.02 (0.03)[a,b,d-g]	1.03 (0.04)[a,b,d-g]	1.04 (0.03)[d-g]	1.01 (0.05)[a,b,d-g]	1.00 (0.05)[a,d-g]
50	0.98 (0.07)[a-c]	0.99 (0.03)[a,b,d-g]	1.00 (0.05)[a-g]	0.99 (0.04)[d-g]	0.95 (0.08)[a-g]	0.77 (0.20)[a,c-g]
65	0.97 (0.04)[a,b]	0.98 (0.04)[a,b,d-g]	0.99 (0.05)[a,b,d-g]	0.94 (0.04)[d-g]	0.84 (0.10)[a,b,d-g]	0.66 (0.25)[a,d-g]
80	0.87 (0.06)[a-c,e]	0.91 (0.05)[a,b,d-g]	0.88 (0.04)[a-g]	0.83 (0.09)[d-g]	0.72 (0.16)[a-g]	0.50 (0.27)[a,c-g]
90	0.84 (0.03)[a,b,c]	0.85 (0.04)[a,b,d-g]	0.83 (0.06)[a,b,d-g]	0.78 (0.09)[d-g]	0.67 (0.13)[a,b,d-g]	0.48 (0.20)[a,d-g]

[a] Isopropanol, coconut base.
[b] Hexane, coconut base.
[c] Benzene, petroleum base.
[d] Acetone, petroleum base.
[e] Carbon tetrachloride, coconut base.
[f] 1-Chlorobutane, coconut base.
[g] Ethyl acetate, petroleum base.

TABLE II.

Comparison of experimental breakthrough times with those calculated from the Mecklenburg and modified Wheeler equations.

SOLVENT VAPOR	CARTRIDGE TYPE	FLOW RATE (liters/min)	CONC. (ppm)	USE RELATIVE HUMIDITY (%)	10% BREAKTHROUGH TIME EXPERIMENTAL (min)	MECKLENBURG (min)	DEVIATION FROM OBSERVED (%)	WHEELER (min)	DEVIATION FROM OBSERVED (%)
Benzene	MSA	53.3	125	50	355	440	24	418	18
			500		134	169	26	161	20
			2000		42	59	40	56	33
Benzene	AO	53.3	1000	20	101	114	13	110	8.7
				50	101	114	13	110	8.7
				80	87	114	31	110	26
Toluene	MSA	20.6	1000	50	288	328	14	322	12
		53.3			114	121	6.1	116	4.7
		71.4			82	89	8.7	84	2.8
m-Xylene	MSA	53.3	1000	50	116	114	-1.6	112	-3.5
p-Cymene	MSA	53.3	1000	50	93	93	0.5	92.2	-0.7
Methanol	MSA	53.3	1000	50	3.2	8.6	168	7.9	145
Isopropanol	MSA	53.3	500	50	126	170	35	160	27
			2000	50	55.7	75	37	63	16
Butanol	AO	71.4	500	50	177	197	12	190	7.2
			3000	50	31	36	16	35	12
Butanol	MSA	53.3	1000	50	141	150	6.1	143	1.5
Vinyl Chloride	AO	40	50	50	77	99	29	96	25
			100		62	81	30	78	26
			250		53	58	12	57	8.1
			500		31	44	41	43	37
			1000		23	32	43	31	38
Ethyl Chloride	MSA	53.3	1000	50	11	22	101	19	73
1-Chlorobutane	MSA	53.3	1000	20	86	90	4.7	87	1.0
				50	87	90	3.5	87	-0.1
				90	68	90	32	87	28
1-Chlorobutane	AO	53.3	500	50	120	135	12	131	8.8
			4000		17	21	21	20	17
Chlorobenzene	MSA	53.3	1000	50	131	132	0.9	128	-2.2
o-Chlorotoluene	MSA	53.3	1000	50	122	125	2.7	122	0.4
Dichloromethane	MSA	53.3	500	50	30	30	1.0	29	-4.9
			2000		17	21.1	21	20	14
1,1-Dichloroethane	MSA	53.3	1000	50	40	73	82	70	73
o-Dichlorobenzene	MSA	53.3	1000	50	132	132	—	130	-1.5
Chloroform	MSA	53.3	1000	50	52	70	33	67	27
Methyl Chloroform	MSA	53.3	250	50	207	251	21	242	17
			2000	50	56	51	-8.5	50	-12
Trichloroethylene	MSA	53.3	1000	50	83	108	30	103	21
1,1,2-Trichloroethane	MSA	53.3	1000	50	132	139	5.0	136	3.0
Carbon Tetrachloride	MSA	53.3	1000	20	85	82	-2.9	79	-6.8
				50	69	82	20	79	15
				90	66	82	25	79	20
Perchloroethylene	MSA	53.3	1000	50	129	129	—	123	-4.0

TABLE II.

(Continued)

SOLVENT VAPOR	CARTRIDGE TYPE	FLOW RATE (liters/min)	CONC. (ppm)	USE RELATIVE HUMIDITY (%)	10% BREAKTHROUGH TIME EXPERIMENTAL (min)	MECKLENBURG (min)	DEVIATION FROM OBSERVED (%)	WHEELER (min)	DEVIATION FROM OBSERVED (%)
Methyl Acetate	MSA	53.3	100	50	146	374	156	353	142
			1000		46	87	92	81.8	82
Ethyl Acetate	AO	53.3	1000	20	88	115	30	111	26
				50	92	115	25	111	22
				65	90	115	27	111	23
				90	68	115	68	111	66
Propyl Acetate	MSA	53.3	1000	50	99	110	11	106	7.5
Butyl Acetate	MSA	53.3	1000	50	97	107	10	104	7.8
Acetone	AO	53.3	100	50	245	504	106	484	198
			1000		66	94	42	91	37
Acetone	AO	53.3	1000	20	61	94	55	91	49
				50	57	94	65	91	59
				80	55	94	73	91	67
				90	53	94	77	91	72
2-Butanone	AO	53.3	1000	50	94	136	45	132	40
2-Heptanone	AO	53.3	1000	50	114	122	6.9	121	5.8
Pentane	AO	53.3	1000	50	71	86	22	84	18
Hexane	AO	53.3	100	50	565	646	14	631	13
			500		143	156	8.9	152	6.3
			2000		38	44	16	43	14
Hexane	MSA	53.3	1000	0	77	78	1.2	75.1	-1.7
				50	70	78	11	75	7.5
				65	68	78	14	75	11
				90	64	78	21	75	18
Heptane	AO	53.3	1000	50	90	106	18	104	16
Decane	AO	53.3	1000	50	82	87	7.2	87	7.2
Methylamine	AO	53.3	1000	50	18	49	174	47	161
Ethylamine	AO	53.3	1000	50	50	100	100	96	93
Diethylamine	MSA	53.3	250	50	93	117	26	110	19
			2000		21	34	64	32	55
Diethylamine	AO	53.3	1000	50	105	88	-16	85	-19

FIGURE CAPTIONS

Figure 1 — Schematic diagram of humidifier and humidity detection system.

Figure 2 — Equilibrium water adsorption capacities for two cartridge types as a function of relative humidity.

Figure 3 — Breakthrough percent as a function of time at various preconditioning humidities. MSA cartridges were tested with 1000 ppm hexane at 53.3 liters/min.

Figure 4 — Breakthrough percent as a function of time at various preconditioning humidities. AO cartridges were tested with 1000 ppm benzene at 53.3 liters/min.

Figure 5 — Weight of water adsorbed at cartridge saturation as a function of preconditioning relative humidity for seven solvent vapors. Tests were conducted at 1000 ppm and 53.3 liters/min.

Figure 6 — Breakthrough percent as a function of time at various use humidities. MSA cartridges were tested at 1000 ppm with 1-chlorobutane and preconditioned at 50% relative humidity.

Figure 7 — Weight of water adsorbed at cartridge saturation as a function of use relative humidity for seven solvent vapors. Cartridges were tested at 1000 ppm and 53.3 liters/min and preconditioned at 50% relative humidity.

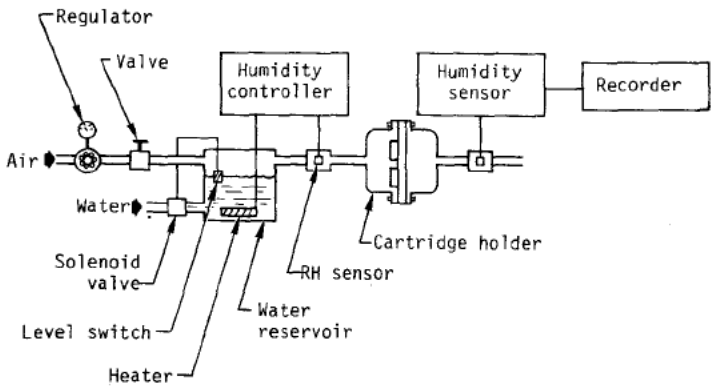
Figure 8 — Breakthrough percent as a function of time for various use humidities at (a) 50%, (b) 65%, and (c) 80% preconditioning humidities. MSA cartridges were tested with carbon tetrachloride at 1000 ppm and 53.3 liters/min.

Figure 9 — Calculated 10% breakthrough time as a function of test humidity at various preconditioning humidities. AO cartridges were tested with 1000 ppm acetone at 53.3 liters/min.

Figure 10 — Calculated 10% breakthrough time as a function of test humidity at various preconditioning humidities. MSA cartridges were tested with 1000 ppm carbon tetrachloride at 53.3 liters/min.

Figure 11 — Weight of water adsorbed at cartridge saturation as a function of preconditioning humidity at various use humidities. MSA cartridges were tested at 1000 ppm isopropanol and 53.3 liters/min.

Figure 12 — Calculated 10% breakthrough time as a function of temperature for seven solvents.



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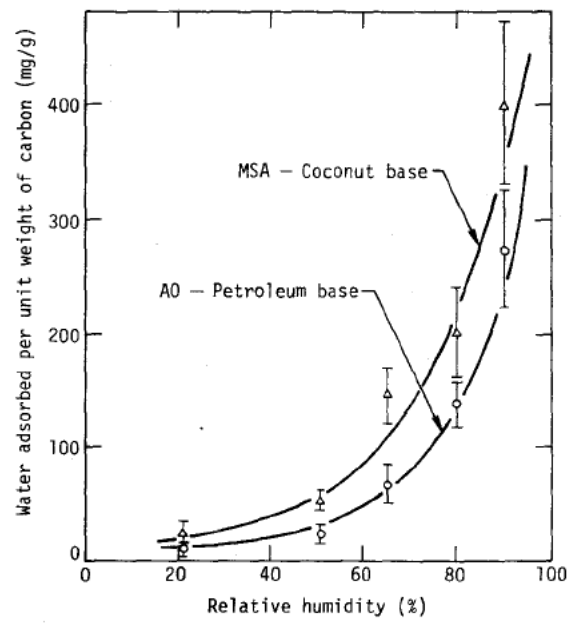

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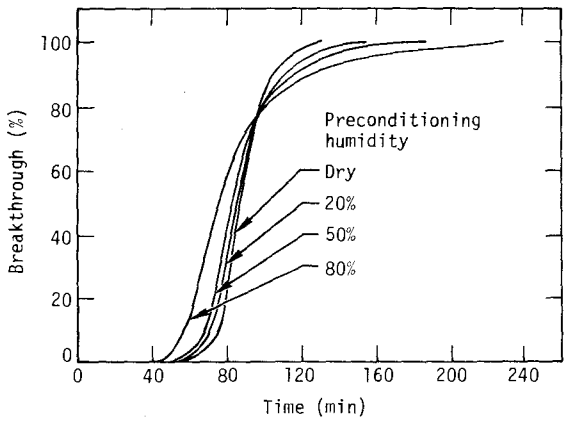

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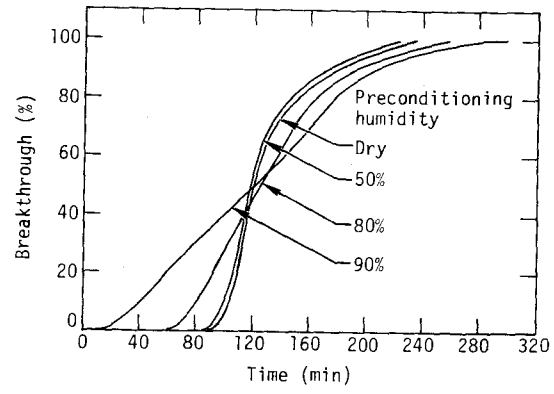

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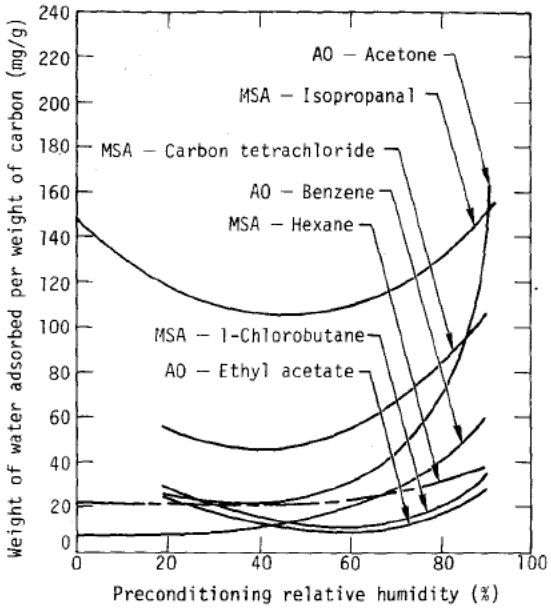

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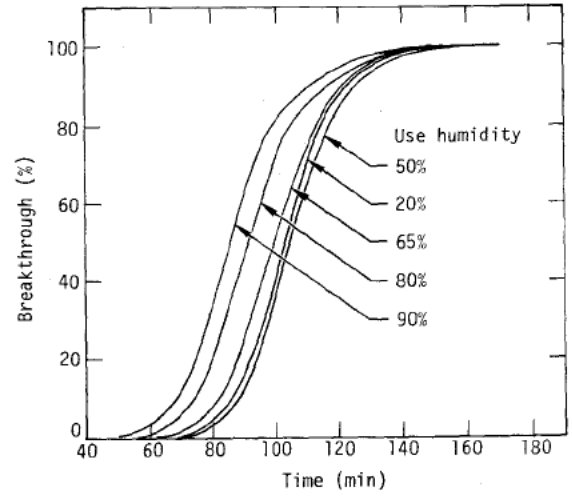

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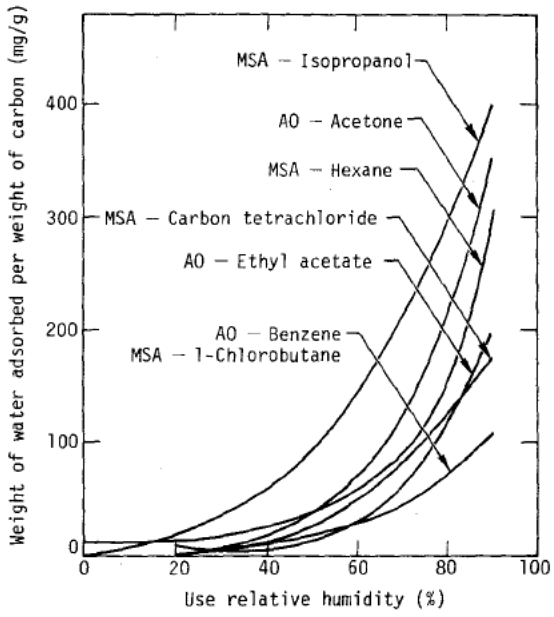
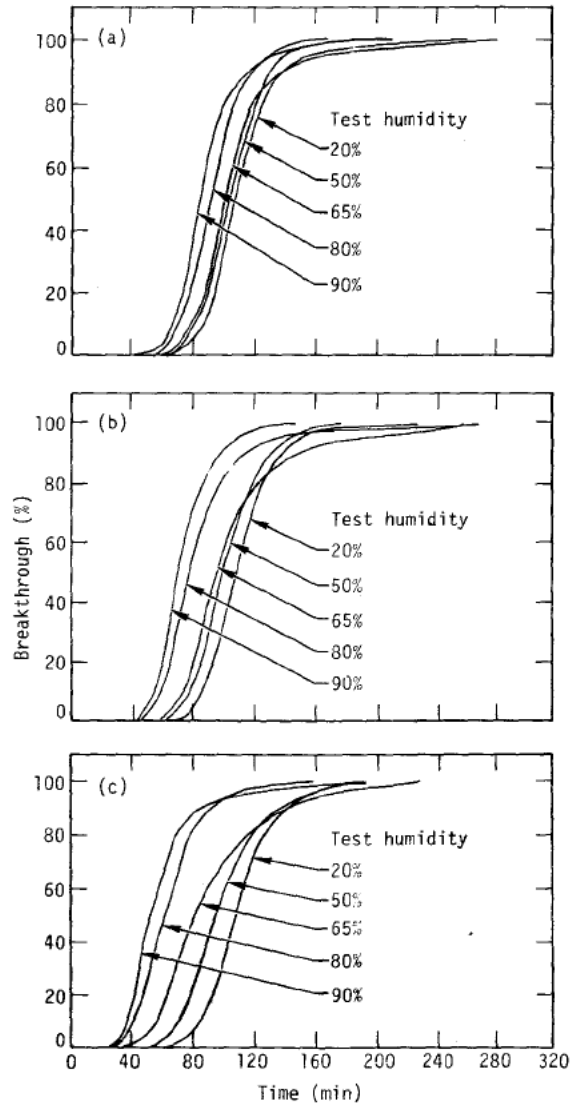
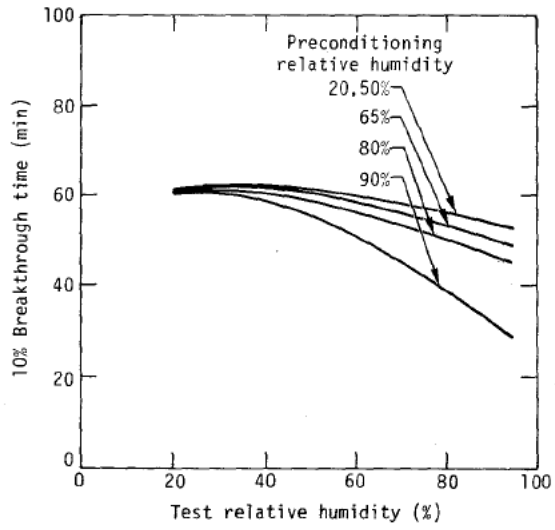


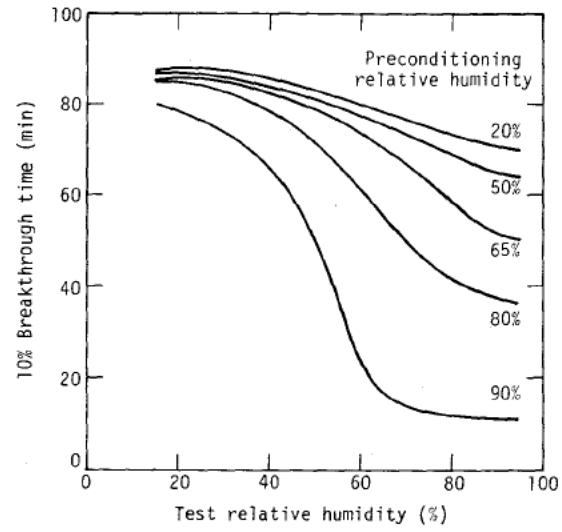
Nelson - Fig. 7



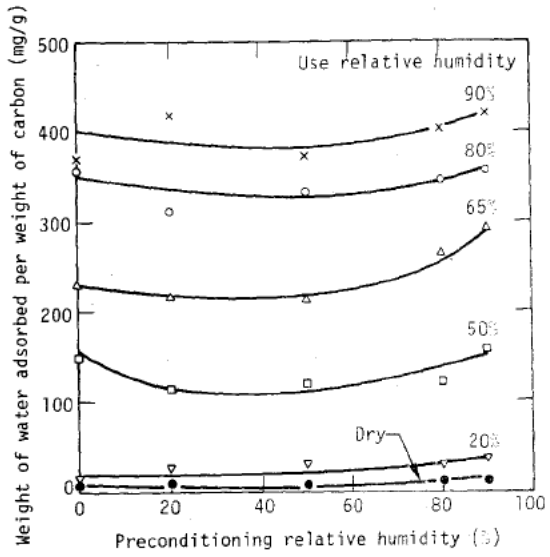
Nelson - Fig. 8



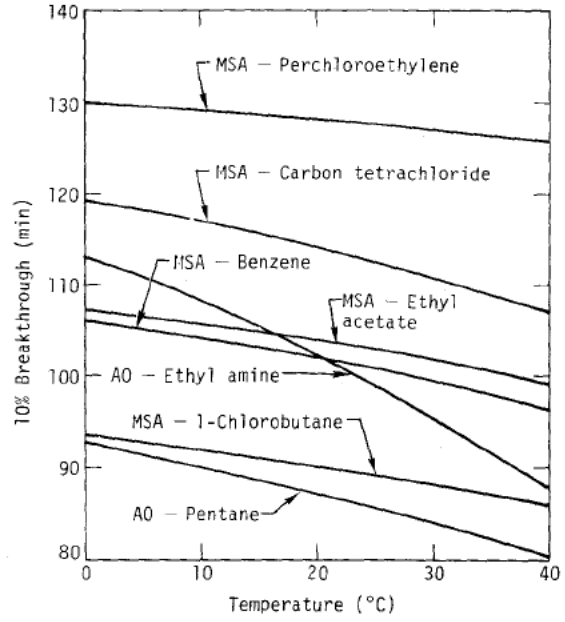
Nelson - Fig. 9



Nelson - Fig. 10



Nelson - Fig. 11



Nelson - Fig. 12

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