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UCRL-73820
PREPRINT

CONF-720532--1

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RESPIRATOR CARTRIDGE EFFICIENCY STUDIES:
IV. EFFECTS OF STEADY-STATE AND PULSATING FLOW

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March 22, 1972

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This paper was prepared for submittal to American Industrial Hygiene Association Journal and for presentation at the American Industrial Hygiene Association Conference in San Francisco, California, on May 14-19, 1972

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RESPIRATOR CARTRIDGE EFFICIENCY STUDIES:

IV. EFFECTS OF STEADY-STATE AND PULSATING FLOW^{*}

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ABSTRACT

The service life of organic-vapor respirator cartridges has been evaluated for steady-state and pulsating flow rates; a mechanical breathing simulator was used to evaluate the pulsating flow rates. Two types of cartridges have been tested at work rates of 0, 208, 415, 622, 830, and 1107 kg-m/min and their equivalent respective steady-state flow rates of 14.0, 20.6, 29.8, 36.9, 53.3, and 71.4 liters/min. Cartridge breakthrough characteristics were recorded with both types of flow for several solvents at 1000 ppm and 0, 50, and 80% relative humidity. No significant differences were observed between the steady-state and pulsating flows, even at the highest work rate and humidity conditions. The effective service life appears to be inversely proportional to the flow rate at a given concentration.

^{*}Work performed under the auspices of the U. S. Atomic Energy Commission.

INTRODUCTION

Historically,¹⁻³ the best suggested method of assessing the true service life of organic-vapor cartridges has been to test them with a pulsating breathing simulator. Since the maximum inspiratory flow is about three times the average flow, it has been maintained that steady-state testing would not yield true performance. This would be true even if the total volume of contaminated air drawn through the cartridge were equivalent for both types of test conditions.

There is, however, some speculation that the equilibrium between the solvent vapor and the charcoal is so rapid that little or no difference would be observed between the two types of flow. If this were the case, charcoal cartridges could then be tested without the expense and time of constructing and using a mechanical breathing simulator. Indeed, any laboratory could obtain reliable cartridge-efficiency data, using the greatly simplified steady-state flow conditions.

The purpose of this investigation was to determine if the breathing simulator were necessary to ascertain the performance of organic-vapor respirator cartridges. In addition, it was desired to determine how the flow or work rate affected the service life of cartridges.

PROCEDURE

Preparation of Test Atmospheres

The system used to produce and monitor the known concentrations of solvent vapor in air was described by Nelson and Hodgkins.⁴ However, an

important modification to the humidifier has been installed. The original humidifier produced steam that was combined with the moving air stream by metering water through a 20-gauge needle in contact with a small cartridge heater, to produce the desired humidity. After prolonged use, however, the needle tended to plug, and the resulting back pressure produced a continually decreasing humidity. The flow rate of the incoming water had to be constantly monitored and adjusted by hand.

A schematic of the new humidifier is shown in Figure 1. Laboratory compressed air now passes directly over a water reservoir, through the overflow trap, and past the humidity and flow sensors. The humidity is automatically controlled by water-temperature adjustments made by a small cartridge-heater that is activated by an electronic hygrometer. The required humidity is obtained by setting a pointer on the meter face to the desired humidity reading. If the actual humidity, measured by the LiBr-LiCl hygrosensor, is lower than the desired humidity, a relay that supplies power to the heater is tripped. The heater is deactivated when the desired humidity is approached. A distilled water inlet is provided for easy filling of the reservoir, and the overflow trap protects the humidity and gas-flow sensors. This system controls the humidity from 10 to 90% at $\pm 3\%$, with no mixing or insulation of the reservoir required.

Breathing Simulator

The pulsating-flow tests were accomplished by using the mechanical breathing simulator described in Reference 5. Work rates of 0, 208, 415, 622, 830, and 1107 kg-m/min were used; these correspond to steady-state flows

of 14.0, 20.6, 29.8, 36.7, 53.3, and 71.4 liters/min, respectively.

During these pulsating-flow tests, enough of the humidified test-gas mixture was generated to exceed the maximum inspiratory flow rate demanded by the breathing simulator. Any excess gas passes through a bypass to a hood. During the steady-state tests, however, the bypass is capped off, and the entire test-gas mixture proceeds through the cartridge of interest. The total volume passing through the cartridge for both the steady-state or pulsating flow is the same.

Cartridge Specifications

Two types of organic vapor cartridges were tested, and their physical characteristics are summarized in Table I. Both exhibited the relatively high surface area required for respiratory protective applications. These were chosen, however, because the two mesh sizes and slightly differing physical dimensions would yield a balanced cross section of most of the approved organic vapor cartridges that were commercially available.

Originally, we attempted to construct our own "standard" reference cartridge. Although the weight and volume of the charcoal could be closely reproduced, some difficulty was experienced in closing the outer case tightly enough to prevent channeling. This approach was abandoned after several case designs were tried, and it was decided to use commercially available cartridges.

Cartridge Preconditioning and Testing

The cartridges were first dried in a vacuum oven for at least 24 hr at 110°C to determine the dry weight of the charcoal. The amount of water

lost was generally 2 to 3% by weight. After being weighed, the cartridges were transferred and stored in a preconditioning cabinet for at least 2 days at 22°C and 50% RH (relative humidity). During this equilibration period, the cartridges generally experience a 4 to 6% weight increase in moisture. The effects of other methods of preconditioning will be discussed in a subsequent paper.

Although two cartridges are used in the face piece, only one cartridge is subjected to the test atmosphere of known composition. This is done primarily to determine what differences, if any, exist between cartridges originating from the same batch and manufacturer.

The preconditioned cartridge is screwed into a polyethylene adapter, which, in turn, is held in place by a holder, as shown in Reference 4. Each cartridge type has its own special adapter to accommodate its unique rolled threads.

Gas-tight integrity between the adapter and the holder is maintained by using O-ring seals, and the whole assembly is held together with four wing nuts. The cartridge gasket, normally supplied with the respirator face piece, is used as a seal between the cartridge and adapter. The entire cartridge-holder assembly is mated to the test-gas generator with a metal-to-glass 35/25 ball-and-socket connection.

The downstream concentration is continuously monitored until breakthrough is complete and the charcoal is totally saturated. The cartridge is then removed from the holder assembly and immediately weighed to determine the total weight of solvent vapor and moisture adsorbed. The cartridge casing is then pried open, the charcoal removed, and the tare weight determined. The maximum packing density of the charcoal is obtained

by pouring the charcoal slowly (for 1 min) into a vibrating, 150-ml graduated cylinder. The charcoal is then transferred and saved in a sealed jar in case further analysis is necessary.

Data Processing

The downstream concentration is monitored by an FID (flame ionization detector), as shown in Reference 5. FID output, in millivolts, is directly proportional to the concentration and yields a linear response on the recorder scale output. These data are processed through the Laboratory computer facilities by the scheme shown in Figure 2. The voltage readings at 5-min intervals, the run identification name and number, the molecular weight, the total weight absorbed, the average flow rate, and the assault concentration are input data (Part A). Voltage-time data are then normalized by letting the minimum ($V_{\min}$) and maximum ($V_{\max}$) output voltages equal zero and 100% breakthrough, respectively. The intermediate points are then reassigned proportional values, based on this scale (Part B).

Generally, more than one cartridge is run for a given set of test conditions. After individual curve normalization, the data are averaged together and graphically displayed by the CRT (cathode ray tube) output (Part C). Standard deviation error bars are shown, to give a rapid visual inspection of the curve spread. The average and range of breakthrough and the relative standard deviation for each 5-min interval are printed out.

$$W_{\text{solv}} = \left(\frac{CMQ}{24.1 \times 10^6} \right) (t_{\text{ave}}), \quad (1)$$

where C is the concentration (ppm), M is the molecular weight (g/mole), Q is the average flow rate (liters/min), and 24.1 is the molar volume at 21°C and 1 atm pressure. The t_{ave} is the average time the cartridge is exposed to the assault concentration. This is determined by establishing a line midway between the first nonzero and the 100% breakthrough points (Part D). This line is moved by small increments until the areas A and B agree to within 0.01%. The intersection of this line with the x-axis yields the t_{ave} . This operation is merely a method of integrating over the curve to obtain a theoretical weight absorbed, based on the known experimental parameters. The theoretical weight of water absorbed is found by

$$W_{H_2O} = W_{total} - W_{solv} \quad (2)$$

where W_{total} is the total weight gain of the cartridge, determined gravimetrically.

Finally, a summary plot that groups the averaged curves (Part D) is provided. This generally shows graphically the effect of a single variable such as flow rate, concentration, solvent type, or humidity on cartridge performance. A table is also provided that shows the times at which selected breakthroughs occur. In the future, time and FID voltage data outputs will be fed directly into a minicomputer, thereby eliminating the need for data cards.

RESULTS AND DISCUSSION

The cartridge adsorption data for the breakthrough curves are shown in Table II. All weights, volumes, and breakthrough times are the average

values generated from the run numbers shown in the left-hand column. The actual breakthrough times for two cartridges used in parallel would be about twice the values shown in Table II.

Cartridge Variation

Before any breakthrough variations between steady-state and pulsating flow could be established, the variation of a group of cartridges had to be established. Such differences arise from irregularities in the solvent-permeation apparatus ($\pm 2\%$) as well as inconsistencies in weight, volume, granule size, and surface area of the charcoal.

Twelve each of the two types of cartridges were subjected to a test concentration of 1000 ppm of toluene at 50% RH. Six cartridges of each type were subjected to a steady-state flow of 53.3 liters/min. The remaining cartridges were tested with the breathing machine at 830 kg-m/min, which also had an average flow rate of 53.3 liters/min. The results are shown in Figure 3; the maximum relative standard deviations are indicated by the error bars.

Note that the standard deviation for six trials is greater than any difference observed between the averaged curves for steady-state and pulsating flow. In other words, neither type of cartridge exhibits any significant difference in service life at the two air-flow patterns tested.

Effect of Flow Rate

Additional breakthrough curves for the steady-state and pulsating flows are shown in Figures 4 and 5 for both cartridges at the six standard

work rates. Once again, no significant difference was observed for the two types of flow patterns. Selected data in Figure 4 is replotted in Figure 6, showing the reciprocal flow rate vs the time at three typical breakthrough percents. A least-squares 1-degree (straight line) fit shows clearly that 1) the time to achieve a desired breakthrough percent is essentially the same for both the steady-state and pulsating flow rates, and 2) the service life of the cartridge, as expected, is inversely proportional to the flow rate.

Effect of Solvent Type

Six other solvents were randomly selected as contaminant test gases, and the results are compiled in Figure 7. The test conditions were again 1000 ppm, 50% RH, and 53.3 liters/min. Additional tests not shown were carried out with an ethyl acetate (type 1) cartridge (1000 ppm; 50% RH at 20.6, 29.8, 36.7, and 71.7 liters/min); a 2 butanone (type 2) cartridge (2000 ppm; 50% RH at 20.6, 29.8, 36.7, 53.3, and 71.4 liters/min); and a methyl chloroform (type 1) cartridge (1000 ppm; 50% RH at 29.8, 36.7, and 71.4 liters/min). Each test was performed in duplicate; but, again, the breakthrough curves for the two flow types were virtually identical.

Effect of Humidity

Relative humidity greatly affects the useful service life, as shown in Figure 8. The test conditions were 1000 ppm toluene with a type 1 cartridge. The initial breakthrough is almost 30% earlier for the 80% RH test than for the test carried out in dry air. However, the steady-state

and pulsating flow patterns at each humidity are not statistically different. A more extensive study concerning the effects of humidity will be subsequently undertaken.

CONCLUSIONS

The following conclusions are based on the results of this study:

1) no significant difference was observed between the steady-state and pulsating flow patterns, even at the highest work rate and humidity conditions; 2) increasing the humidity will shorten the effective service life of cartridges; and 3) cartridge service life is inversely proportional to flow rate for a given test vapor, concentration, and relative humidity.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the assistance of Robert S. Flint, who developed the data-reduction computer program, and Robert D. Taylor and Bernell J. Baquette for their skillful engineering support.

We also wish to thank Carl L. Lindeken, Joe Lipera, J. Louis Kovach, and Edwin J. Kloos, whose counsel and advice have immeasurably benefited our testing program.

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Table I
Characteristics of the Organic Vapor Cartridges Tested

	Type 1	Type 2
Charcoal weight, dry (g)	28	29
Charcoal volume at maximum packing density (ml)	72	80
Density at maximum packing (g/ml)	0.39	0.36
Moisture content, weight percent		
As received	3.0	2.4
Stored at 50% relative humidity	5.4	4.5
Particle size, U. S. sieve series (mesh)	8-16	12-20
Cartridge diameter (cm)	6.3	7.0
Bed depth (cm)	2.3	2.1
Cross section of adsorbent bed (cm ²)	31.2	38.5
Surface area (m ² /g)	1500-1625	1500-1625
Pore volume (ml/g)	0.9-1.0	0.9-1.0
Void volume (ml/g) ^a	0.42	0.38
External surface (cm ² /g) ^a	45	77
Charcoal-type, coconut-base carbon ^b	G212	G215
Pressure drop (in., H ₂ O)		
40 liters/min	0.9	1.0
80 liters/min	2.1	2.3
120 liters/min	3.9	3.9

^aJ. Louis Kovach, private communication.

^bNorth American Carbon specification number. 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. Atomic Energy Commission to the exclusion of others that may be suitable.

TABLE II

Explosive Cartridge Breakthrough Rate Summary (All cartridges were tested at 1000 psi and 500 Hz except where specified)

Run No.	Solvent	Work rate (g-m/min)a	Air flow (liters/min)	Cartridge type	Air breakthrough ^b time					Wt solvent and water adsorbed at 1000 psi (g)	Wt water adsorbed, calc. from Eq. 2 (g)	
					Channel Vol. (g)	Channel vol. (ml)	t ₁₂ (min)	t ₁₀₀ (min)	t ₅₀₀ (min)			t ₉₉₀ (min)
121-126	Toluene	830	53.3	2	32.7	79.0	36	66	77	125	16.4	0
127-132	Toluene	830	53.3	1	29.5	70.5	43	58	71	109	15.1	0.5
133-138	Toluene	88	53.3	1	29.5	70.5	43	57	69	100	15.2	0.8
139-144	Toluene	88	53.3	1	29.0	70.5	47	61	72	93	16.6	0.5
145-150	Toluene	88	53.3	2	33.1	70.5	53	68	75	110	16.4	0.8
146, 192,												
267	Toluene	88	14.0	2	29.5	79.5	222	236	291	415	16.3	0.1
147, 148	Toluene	88	71.4	1	29.4	69.5	30	41	50	90	14.2	0.6
149, 150	Toluene	88	71.4	2	29.7	76.5	37	45	52	83	15.7	0.8
151, 189,												
269, 270	Toluene	88	14.0	1	28.0	73.0	195	225	264	372	15.3	1.0
152, 153	Toluene	1187	71.4	1	29.5	69.5	30	39	50	84	16.6	0.7
154, 155	Toluene	1187	71.4	2	32.5	79.0	35	42	51	86	15.8	0.4
156, 193	Toluene	0	14.0	2	33.5	79.5	202	230	266	410	16.6	1.6
157	Toluene	88	36.7	2	28.7	77.0	83	96	114	177	15.1	-1.6
159, 185,												
253, 254	Toluene	0	14.0	1	28.6	72.0	101	216	250	379	15.1	1.0
160	Toluene	622	36.7	2	33.2	80.0	91	102	116	177	16.9	-0.2
161	Toluene	622	36.7	1	29.1	70.5	62	76	93	127	14.8	1.7
162	Toluene	88	36.7	1	28.1	70.0	69	82	96	137	16.8	1.2
163,												
230-232	Toluene	88	29.0	1	27.6	71.5	89	105	122	160	14.0	0.6
165, 171	Toluene	280	20.6	1	28.3	71.0	125	153	170	252	15.1	0.8
166,												
240-242	Toluene	415	29.0	1	28.0	72.0	81	101	110	164	14.0	1.2
167,												
237-239	Toluene	415	29.0	2	31.0	79.5	100	115	134	205	16.3	1.0
168	Toluene	280	20.6	2	33.4	81.5	150	170	211	397	16.7	-0.9
169, 194												
237-239	Toluene	96	29.0	2	31.3	80.5	105	118	135	207	16.6	0.7
175	Toluene	88	20.6	2	31.2	80.0	152	185	221	306	16.1	-0.3
172, 173	Toluene	830	53.3	1	29.6	71.0	42	60	71	96	14.4	0.1
176, 177	Toluene	88	53.3	1	29.5	72.0	36	46	60	117	17.3	4.3
178, 182	Toluene	830	53.3	1	28.5	72.5	30	43	57	100	17.9	4.8
181	Toluene	88	20.6	1	28.2	72.0	121	144	160	202	15.1	1.4
192, 196	Diethyl sebacate	88	53.3	1	28.4	72.0	33	41	49	86	17.9	2.8
197, 198	Diethyl sebacate	830	53.3	1	28.5	72.0	32	41	52	90	17.7	1.2
199, 200	Trichloroethylene	430	53.3	2	32.7	83.0	47	50	70	119	21.9	0.8
201, 202	Trichloroethylene	88	53.3	2	33.0	81.5	47	54	65	125	22.4	2.1
203, 209	Cyclohexane	88	53.3	2	30.7	81.0	34	41	60	86	10.5	1.1
210, 211	Cyclohexane	430	53.3	2	32.0	83.0	29	37	47	85	10.6	1.1
214, 215	Methyl chloroform	88	53.3	1	27.2	72.0	29	40	52	86	10.5	0.9
217, 218	Benzyl alcohol	430	53.3	1	37.2	72.0	56	71	80	143	15.2	0.5
219-220	Benzyl alcohol	88	53.3	1	37.0	71.5	61	72	80	136	15.6	0.6
221, 222	1-Pentanol	50	53.3	2	30.7	80.0	30	45	53	96	11.0	2.1
223, 224	1-Pentanol	430	53.3	2	28.0	79.5	37	46	55	96	11.5	2.2
225, 226	Methyl chloroform	430	53.3	1	28.4	72.0	33	36	40	71	10.4	2.2

^a88 = Steady state^bdry air^c500 Hz

FIGURE CAPTIONS

- Fig. 1. Schematic diagram of humidity-control system.
- Fig. 2. Data processing scheme.
- Fig. 3. Breakthrough curves for types 1 and 2 cartridges. Each curve was generated from an average of six trials, and error bars indicate the standard deviation for each curve. The trial run numbers for each type of flow are shown in parentheses and can be related to Table II if full cartridge data are required.
- Fig. 4. Breakthrough curves for type 1 cartridges at six flow rates.
- Fig. 5. Breakthrough curves for type 2 cartridges at six flow rates.
- Fig. 6. Reciprocal flow rate vs time for breakthroughs of 5, 50, and 95%. A least-squares fit of a straight line shows little difference between the steady-state and pulsating type flow.
- Fig. 7. Breakthrough curves for six solvents at 1000 ppm, 50% RH, and 53.3 liters/min. The flow type exhibits no significant difference. Steady-state flow is shown as solid lines; pulsating flow is shown as dashed lines.
- Fig. 8. Effect of cartridge efficiency as a function of relative humidity. These type 1 cartridges were tested at 1000 ppm at 53.3 liters/min. Increasing the humidity decreases the service life, but little variation is noted between the steady-state and pulsating flow at either the 80% RH or the dry condition. Steady-state runs 176 and 177 and pulsating-flow runs 178 and 182 were made at 80% RH; steady-state run 135A and pulsating-flow runs 172 and 173 were made in dry air.

Nelson - Fig. 1

A **B** **C** **D** **E**

Nelson - Fig. 3

Nelson - Fig. 4

Nelson - Fig. 5

Nelson - Fig. 6

Nelson - Fig. 7

Nelson - Fig. 8