Evaluation of Carbon Tetrachloride Replacement Agents For Use in Testing Non-Powered Organic Vapor Front-Mounted/Back-Mounted Canisters

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Abstract

Carbon tetrachloride has been used for years to certify front-mounted/back-mounted (FM/BM) canisters as part of 30 CFR Part 11. Due to toxicity and availability issues, a substitute organic vapor test agent is needed. Evaluations of four potential substitute agents (ethyl acetate, pentane, n-hexane, and heptane) were performed and results are discussed. Screening tests were employed to identify agent/challenge concentrations which would be equivalent to the present 30 CFR Part 11 carbon tetrachloride criteria. Side-by-side correlation testing was done between carbon tetrachloride and pentane. It was found that pentane testing of "as received" canisters at 10,000 ppm, 80% RH, 64 lpm, and 25°C with a 50-minute breakthrough time at 5 ppm breakthrough concentration was comparable to the present 30 CFR Part 11 carbon tetrachloride criteria for FM/BM canisters. However, it should be noted that this study only looked at NIOSH-certified canisters. No work was done on sorbents which would not meet 30 CFR Part 11 performance criteria. Such studies need to be performed to ascertain if pentane can adequately screen sorbents of lesser capacity.

Introduction

The identification of a suitable substitute for carbon tetrachloride for evaluating non-powered organic vapor front-mounted and back-mounted (FM/BM) canister respirators is essential. Due to the Environmental Protection Agency's (EPA) requirement that 40 CFR 82 will be required to conform to the requirements of Title VI of the Clean Air Act Amendments of 1990 (PL 101-549), carbon tetrachloride supplies are limited and may ultimately be phased out. Also, carbon tetrachloride has been cited by the National Institute for Occupational Safety and Health (NIOSH) as a potential occupational carcinogen.[1] Carbon tetrachloride is currently mandated in Title 30 Code of Federal Regulations, Part 11 (30 CFR Part 11)[2] as the test agent for FM/BM organic vapor canisters. Both a substitute agent for carbon tetrachloride and test conditions which have adsorption characteristics similar to carbon tetrachloride need to be identified. Carbon tetrachloride has long been recognized as a standard used by the carbon industry to measure adsorption capacity. Further, it is particularly suited for respirator sorbent testing since it is water-immiscible, and a prominent diminished adsorption effect is seen at high water-vapor concentration.

The National Institute for Occupational Safety and Health (NIOSH) reported on cartridge[3] and chin-style[4] canister breakthrough evaluations against four potential replacement agents (ethyl acetate, pentane, n-hexane, and heptane). Initial testing with both cartridges and chin-style canisters identified agents/challenge concentrations which gave breakthrough characteristics similar to those for carbon tetrachloride in 30 CFR Part 11.[2] Breakthrough time was the sole critical criterion. The replacement agent for testing cartridges was pentane at a challenge concentration of 550 parts per million (ppm), 64 liters per minute (lpm), 80% Relative Humidity (RH), "as received" cartridges, 40-minute breakthrough time at 5 ppm breakthrough concentration, and 25°C. This condition was based on a comparison with the most critical cartridge tests presently in 30 CFR Part 11,[2] which had the following parameters: carbon tetrachloride at a challenge concentration of 1000 ppm, 50% RH, 64 lpm, "as received" cartridges, 50-minute breakthrough time, 5-ppm breakthrough concentration, and 25°C for NIOSH-certified cartridges.

A later study[4] determined breakthrough-time data for certified chin-style canisters. The major differences between cartridges and chin-style canisters are that chin-style canisters contain 2.5 to 3.0 times more sorbent, and that the 30 CFR Part 11[2] test requirements are different. The most critical 30 CFR Part 11 test was for preconditioned canisters at 85% RH, which was not the case with cartridges. The chin-style canister data showed that, based on breakthrough time as the critical criterion, 4000 ppm pentane testing of "as received" canisters at 80% RH was a suitable replacement for the 5000 ppm carbon tetrachloride, 85% RH, preconditioned-canister testing of organic vapor chin-style canisters. Also, side-by-side testing results gave similar breakthrough times for "as received" chin-style canisters tested at 50% RH and 80% RH, and 64 lpm and 25°C. A testing regimen using only "as received" chin-style canisters at 25% RH, 50% RH, and 80% RH (25°C) at a flowrate of 64 lpm was proposed and correlation testing done.

This report presents the breakthrough time data obtained for FM/BM canisters. These canisters contain approximately 10 to 11 times the amount of sorbent contained in cartridges.

Background

The FM/BM canisters are tested under 30 CFR Part 11[2] Subparts I and M. The specific performance criteria are given in 11.102-1 through 11.102-5 and 11.183-1 through 11.183-7. These FM/BM canisters are tested against a carbon tetrachloride challenge concentration of 20,000 ppm. The 30 CFR Part 11 test requirements for FM/BM canisters are summarized in Table I. It should be noted that the challenge concentrations for the replacement agents probably will have upper-use concentration restrictions due to the lower-flammability limit (LFL) for the various candidate agents. The LFLs* for those agents are significantly lower than 20,000 ppm. Table I indicates that, presently, FM/BM canisters are tested both "as received" from the applicant and after preconditioning at either 25% or 85% RH. Varying flowrates (32 or 64 lpm) are employed, but the minimum service life is 12 minutes at the various test conditions. The rationale for preconditioning the canisters at 25% RH or 85% RH, followed by testing at 50% RH at a reduced flowrate, has been questioned.[5] Further, testing "as received" canisters over a wide range of relative humidities at the higher flowrate (64 lpm) appears more representative of use-type conditions.[6] Such use-type condition testing would constitute more realistic performance standard for evaluating organic vapor and gas (OV) respirator canisters.

Ethyl acetate, pentane, n-hexane, and heptane were selected and screened based on their physical characteristics and toxicity,[1] as well as reported charcoal adsorption characteristics.[6-9] The FM/BM canisters were tested "as received" from the manufacturer at either 50% RH or 80% RH and at 25±2°C. These tests were conducted at various challenge concentrations of the control, carbon tetrachloride, and the above four possible replacement agents. These studies were to identify agent/challenge

*Warning: These hydrocarbons are extremely flammable, and sparks and open flames must be avoided. Further, the LFLs for these agents are as follows: ethyl acetate, 22,000 ppm; pentane, 15,000 ppm; n-hexane, 11,000 ppm, and heptane, 10,500 ppm.

concentrations which would give FM/BM-canister-breakthrough times similar to the 20,000 ppm carbon tetrachloride tests mandated in 30 CFR Part 11.[1]

Subsequently, side-by-side correlation testing was performed solely with pentane to compare it with the carbon tetrachloride control. This testing was performed at various carbon tetrachloride and pentane concentrations, due to the LFL of 15,000 ppm for pentane. The comparison testing was to confirm that the test agent(s)/challenge-concentration conditions gave cartridge breakthrough times similar to carbon tetrachloride at the most severe test condition. Further, the comparison testing determined which test condition was the most critical-equilibrated canisters, as per 30 CFR Part 11, or "as received" canisters run at a higher test RH (80%).

Experimental Method

The test configuration for the initial tests on single canisters is shown in Figure 1 and has been described previously.[4] Individual FM/BM canisters were tested "as received" from the manufacturer at a flowrate of 64 lpm through the canister. The test humidities were 50% RH and 80% RH, and the temperature was 25±2.0°C. Various challenge concentrations of carbon tetrachloride (control) and potential replacement agents (ethyl acetate, pentane, n-hexane, and heptane) were tested.

Breakthrough times were determined as the single critical criterion for determining equivalency with carbon tetrachloride.

Subsequent side-by-side testing was done as per the certification testing procedure employing the certification test equipment or the system shown in Figure 1. These tests directly compared the replacement agent with the control, carbon tetrachloride, at 50% RH and 80% RH on "as received" canisters at 25°C. The FM/BM canisters manufactured by one company were used throughout this study. The differences between the certification test system and the one shown in Figure 1 are: (1) the certification system pushes air through the FM/BM canisters rather than being pulled through with a vacuum source (Figure 1), (2) the certification methodology determines the challenge concentration gravimetrically rather than instantaneously over the entire test with an upstream IR detector, (3) the certification methodology determines the breakthrough time at 5 ppm, not continuously as a function of time (Figure 1), and (4) a teflon needle rests on heated glass beads in order to generate the pentane challenge.

The detector system was a Miran 1A (Foxboro) general purpose infrared (IR) gas analyzer equipped with a variable-path-length gas cell. The analytical wavelength and minimum detectable limits, as specified by the manufacturer, with a 20-meter cell for the test agents are as follows: carbon tetrachloride 12.6 micrometers (μm) and 1.1 ppm, ethyl acetate 8.3 μm and 0.1 ppm, pentane 3.4 μm and 0.2 ppm, n-hexane 3.4 μm and 0.2 ppm, and heptane 3.4 μm and 0.1 ppm.

The test agents evaluated were as follows: (1) carbon tetrachloride, Fisher Scientific, Pittsburgh, PA, certified American Chemical Society (ACS) spectranalyzed, or by Aldrich Chemical Company, Milwaukee, WI, ACS reagent 99%; (2) ethyl acetate, Fisher Scientific, Pittsburgh, PA, certified ACS spectranalyzed; (3) n-pentane, Fisher Scientific, Pittsburgh, PA, HPLC grade; (4) n-heptane, Fisher Scientific, Pittsburgh, PA, HPLC grade, and (5) n-hexane was either Fisher Scientific, Pittsburgh, PA, certified ACS 99 Mol % pure or J.T. Baker Chemical Company, Phillipsburg, NJ, HPLC 97%. House air which was passed through a dryer/sorbent system to remove contaminants was used.

Results and Discussions

As indicated, one manufacturer's FM/BM canisters were used throughout this study. The breakthrough data for these canisters against carbon tetrachloride (control) are presented in Table II. Carbon tetrachloride concentrations from 5,000 to 20,000 ppm were employed, and tests were conducted at 50% RH and 80% RH. A plot of this data is presented in Figure 2. Similar to the case with chin-style canisters, the effect of RH on the breakthrough times is minimal for "as received" canisters. This is due to the high challenge concentration (high ratio of contaminant to water) which severely reduces the competitive effect of the water vapor and the significant larger sorbent weight of the bed.

Ethyl acetate breakthrough data is presented in Table III. Challenge concentrations of 1,000 to 10,000 ppm and humidities of 50% RH and 80% RH were used to test the "as received" canisters. The 5,000- and 10,000-ppm data for carbon tetrachloride and ethyl acetate are of the same order. However, ethyl acetate was eliminated from consideration as a replacement agent due to its significantly different solubility characteristics from carbon tetrachloride (ethyl acetate is water soluble and carbon tetrachloride is insoluble). Even though ethyl acetate was not run above 10,000 ppm, it could have been run at a concentration up to approximately 15,000 ppm as the LFL is 22,000 ppm.

The pentane breakthrough data is given in Table IV. The challenge concentrations used in the testing were between 5,000 and 12,000 ppm. Figure 3 is a plot of the data presented in Table IV. It is noted that the 50% RH and 80% RH data is basically the same, indicating that the RH effect is minimal. The carbon tetrachloride and pentane data are both plotted in Figure 4. Figure 4 reveals that the breakthrough characteristics of carbon tetrachloride and pentane are basically identical in the 10,000 ppm range. A look at side-by-side correlation between pentane and carbon tetrachloride will be discussed later.

Table V presents the initial n-hexane data. The challenge concentration varied from 2000 to 9000 ppm. Higher concentrations were not employed since n-hexane's LFL is 11,000 ppm. Similarly, heptane has a LFL of 10,500 ppm and only two experimental tests were done using heptane. The heptane data is as follows: challenge concentration 7,500 ppm, charcoal weight 742.9 grams, 80% RH, 25°C, breakthrough time at 5 ppm was 97.6 minutes; challenge concentration 9000 ppm, charcoal weight 739.1 grams, 80% RH, 25°C, breakthrough time at 5 ppm was 94.0 minutes.

Pentane was the only agent considered for further correlation studies. Ethyl acetate was eliminated due to its water solubility which could introduce another potential variable. Both n-hexane and heptane had LFLs which limited their challenge concentration test range. Thus, pentane was the agent that was used in subsequent testing.

The correlation data for the "as received" FM/BM canisters at 50% and 80% relative humidities for pentane and the carbon tetrachloride controls are presented in Table VI. Data for carbon tetrachloride at challenge concentrations of 10,000 and 20,000 ppm are presented. A carbon tetrachloride challenge concentration of 20,000 ppm is required per 30 CFR Part 11. Pentane data was generated up to 10,000 ppm because pentane's LFL is 15,000 ppm. Evaluation of the data in Table VI reveals the following points:

1. The FM/BM canister's breakthrough times for carbon tetrachloride exceeds the required 12-minute criterion in 30 CFR Part 11 at the most severe test condition, which was found to be 85% RH preconditioned canisters tested at 20,000 ppm, 32 lpm, and 25°C.

2. Test data were obtained for one supplier's FM/BM canisters and thus, the amount of data upon which to base substitute test criteria is extremely limited. An attempt was made to set criteria for the substitute agent pentane.

3. A testing regimen employing "as received" canisters is possible and should be more indicative of use-type conditions.

The most severe 30 CFR Part 11 test was on 85% RH preconditioned canisters which were subsequently tested against carbon tetrachloride at 20,000 ppm, 50% RH, 32 lpm, and 25°C. A breakthrough time of greater than 12 minutes at a 5 ppm breakthrough concentration is required to meet 30 CFR Part 11 requirements. Pentane replacement conditions which would be comparable to the above carbon tetrachloride conditions were sought. The pentane condition identified was a pentane challenge concentration of 10,000 ppm, test "as received" canisters at 80% RH, 64 lpm, 25°C, and a breakthrough time of at least 50 minutes is required at a 5 ppm breakthrough concentration. This was determined comparing the carbon tetrachloride preconditioned 85% RH, 32 lpm, 25°C date (Table VI) with the pentane "as received" 80% RH, 64 lpm, 25°C date (Table VI). Due to the limited number of data points, a nonparametric method of estimation was employed. All controls were paired with all challenge agents and direct estimates were obtained based on the 12-minute breakthrough time for carbon tetrachloride. The 50-minute breakthrough time is the median of all pair-wise estimates.

Conclusions

The FM/BM canisters' behavior is similar to that of the chin-style canisters in that the most severe 30 CFR Part 11 test condition is the 85% RH preconditioned canisters tested at 20,000 ppm carbon tetrachloride, 50% RH, 32 lpm, 25°C and a 12-minute minimum breakthrough time at a 5 ppm breakthrough concentration. Also, the effect of test relative humidity on "as received" canisters was minimal as was the case for chin-style canisters.

Since pentane was found to be a suitable agent for both OV cartridge and chin-style canister testing, it is logical to use pentane for testing FM/BM canisters also. Pentane was the least toxic of the saturated hydrocarbon replacement agents studied. Pentane was the hydrocarbon with the highest LFL (15,000 ppm) which limited the range of challenge concentrations studied. A comparison between the existing carbon tetrachloride certification testing at 20,000 ppm and pentane at 10,000 ppm was performed. The data shows that the breakthrough times for "as received" canisters tested at 80% RH, 64 lpm, 25°C, and a 50-minute breakthrough time at a 5-ppm breakthrough concentration was comparable to the carbon tetrachloride criteria in 30 CFR Part 11. It must be noted that only NIOSH-certified canisters were investigated in this study and this is a severe limitation of this study. No testing of sorbents which would not meet 30 CFR Part 11 performance criteria were done. Studies on sorbent of lesser capacity and poorer relative humidity resistance need to be performed in order to ascertain if pentane can adequately screen charcoals with highly diverse characteristics.

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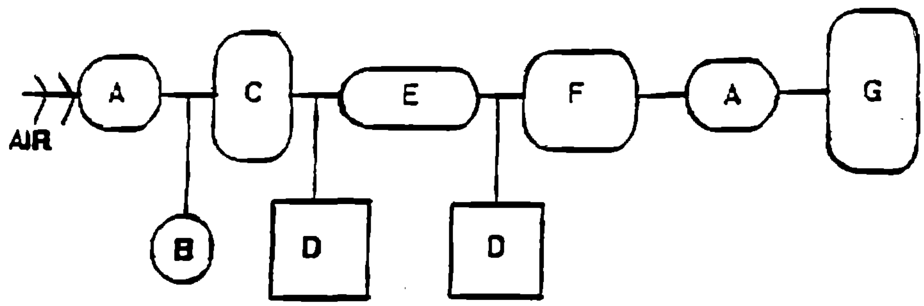
Figure Captions

1. Canister Test System
2. Carbon Tetrachloride Breakthrough Data
3. Pentane Breakthrough Data
4. Carbon Tetrachloride vs Pentane Breakthrough Data at 80% Relative Humidity

Figure 1



A – FLOW CONTROL MECHANISM
B – VAPOR GENERATOR
C – BUFFER RESERVOIR TANK
D – MIRAN 1A GAS ANALYZER
E – CARTRIDGE CELL
F – SORBENT SCRUBBER
G – VACUUM SOURCE

Figure 2

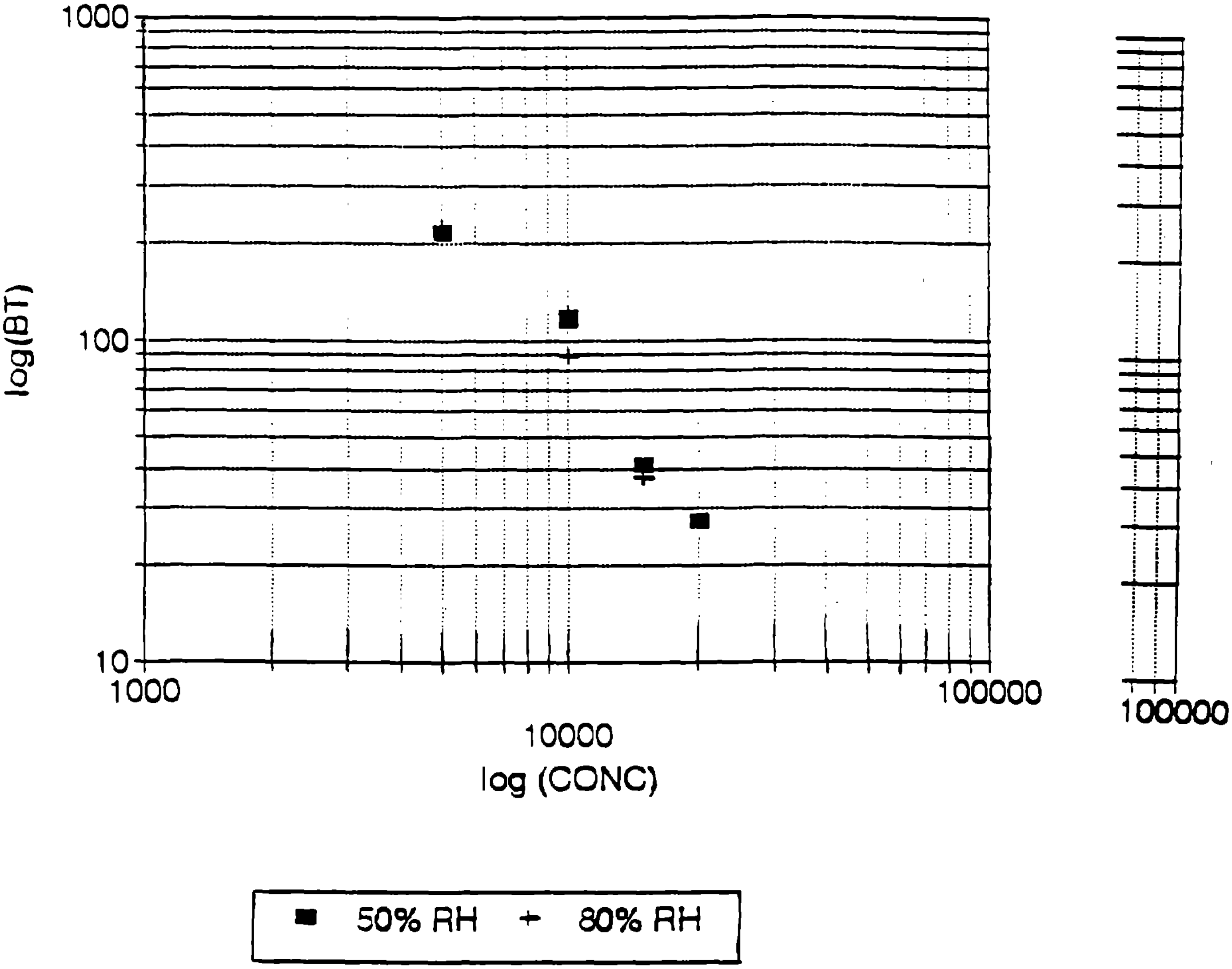

1000
100
10
log(BT)
1000
10000
100000
log (CONC)
100000
50% RH
80% RH

Figure 3

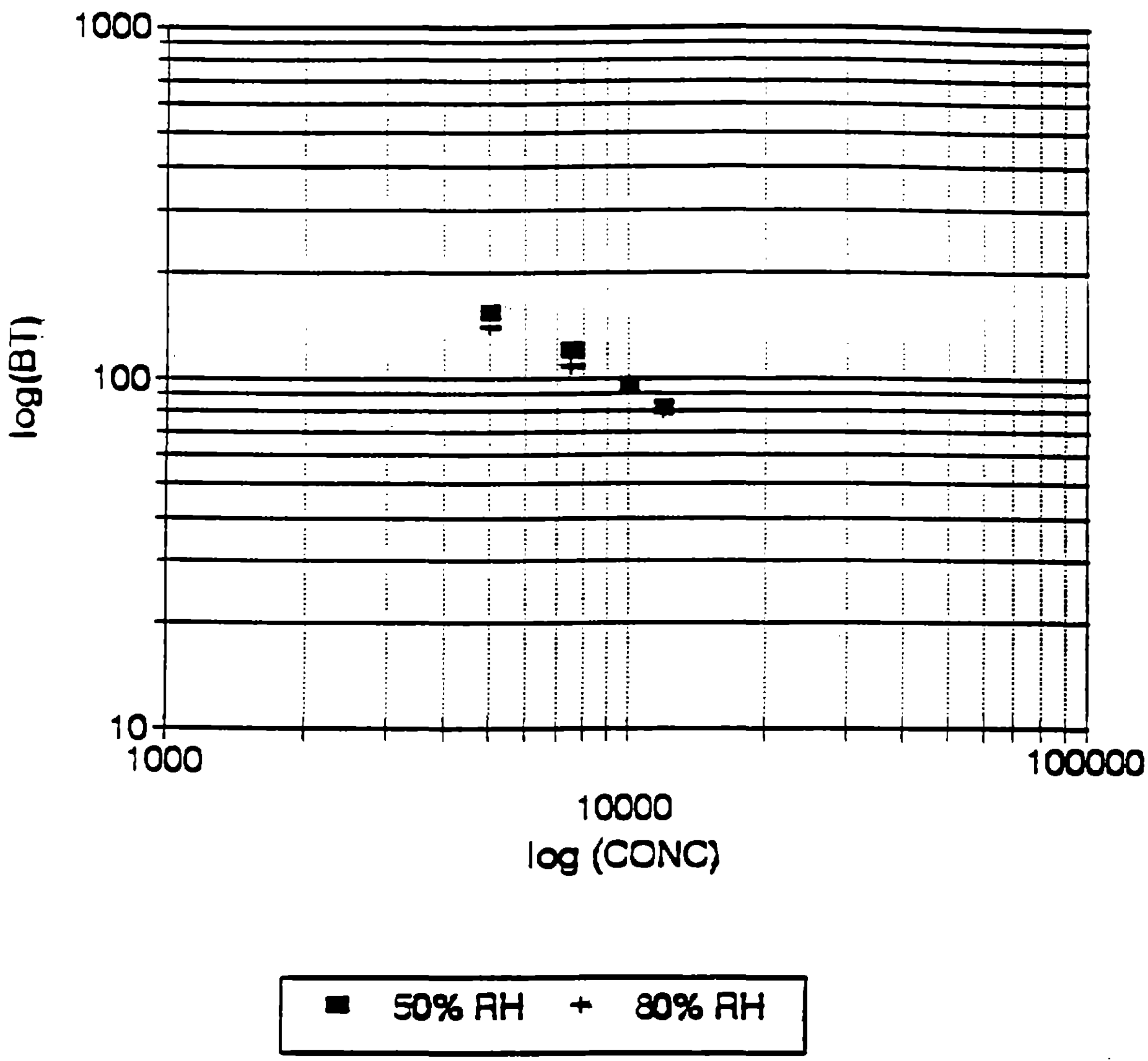


Figure 4

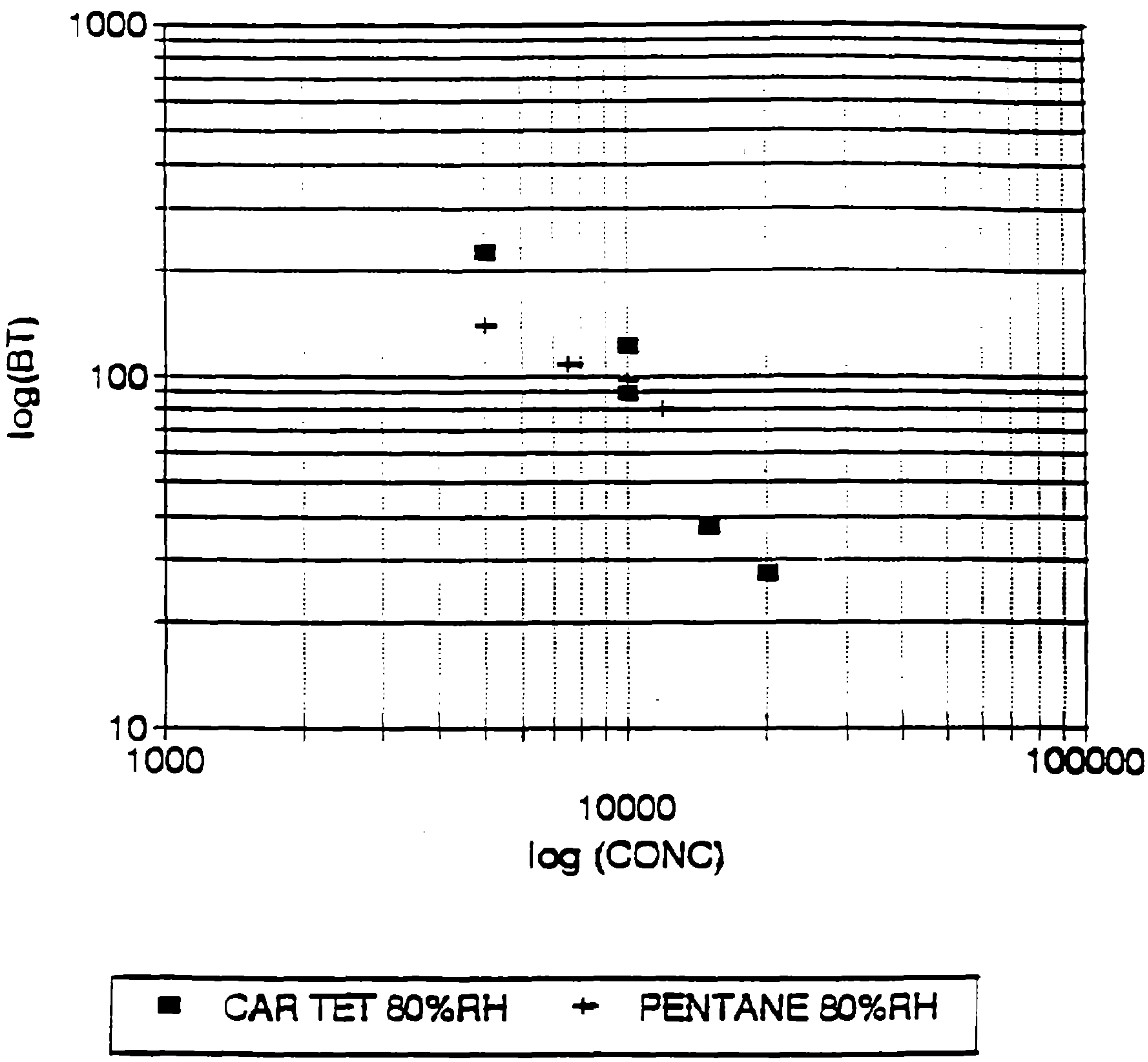

1000
100
10
log(BT)
1000
10000
100000
log (CONC)
CAR TET 80%RH
PENTANE 80%RH

TABLE I

30 CFR PART 11 ORGANIC VAPOR TESTS FOR FM/BM CANISTERS AGAINST CARBON TETRACHLORIDE

CANISTER TYPE	TEST CONDITION	CHALLENGE CONCETRATION (PPM)	TEST FLOWRATE LPM	TEST RELATIVE HUMIDITY (%)	# OF TESTS	MAXIMUM ALLOWABLE PENETRATION (PPM)	MINIMUM SERVICE LIFE(*) (MIN)
FM/BM(**)	As Received	20,000	64	50	3	5	12
FM/BM	Equilibrated 25% RH	20,000	32	50	2	5	12
FM/BM	Equilibrated 85% RH	20,000	32	50	2	5	12

(*) Minimum life will be determined at the indicated penetration
(**) Front-mounted/Back-mounted

TABLE II

SUMMARY OF FM/BM CANISTER BREAKTHROUGH TIME DATA AGAINST VARIOUS CARBON TETRACHLORIDE CHALLENGE CONCENTRATIONS

CHALLENGE CONCENTRATION PPM	TYPE	LOT	CHARCOAL WT (gm)	%RH	t_b CORRECTED TO CHALLENGE CONCENTRATION		
					5PPM	10PPM	15PPM
5,000	FM/BM*	A	753.4	50	214.7	---	---
5,000	FM/BM*	A	750.4	80	225.3	---	---
10,000	FM/BM*	A	750.3	50	118.2	---	---
10,000	FM/BM	B	753.0	50	115.8	120.7	122.5
10,000	FM/BM*	A	748.5	80	122.1	---	---
10,000	FM/BM	B	818.5	80	88.9	94.1	95.4
15,000	FM/BM	B	817.6	50	40.9	42.0	42.7
15,000	FM/BM	B	817.8	80	37.7	39.2	39.9
20,000	FM/BM	B	808.3	50	27.5	27.9	28.4
20,000	FM/BM	B	824.8	80	27.7	28.4	29.0

*CERTIFICATION TESTING SYSTEM

TABLE III
SUMMARY OF FM/BM CANISTER BREAKTHROUGH TIME DATA AGAINST VARIOUS ETHYLACETATE CHALLENGE CONCENTRATIONS

CHALLENGE CONCENTRATION PPM	TYPE	LOT	CHARCOAL WT (gm)	%RH	t_b CORRECTED TO CHALLENGE CONCENTRATION		
					5PPM	10PPM	15PPM
1,000	FM/BM	B	737.2	50	671.7	690.3	697.3
1,000	FM/BM	B	738.3	80	463.5	486.8	504.5
2,000	FM/BM	A	731.5	50	378.2	394.5	402.5
2,000	FM/BM	A	739.6	80	290.7	313.9	323.4
5,000	FM/BM	B	736.7	50	183.8	189.2	192.5
5,000	FM/BM	B	745.7	80	175.2	181.9	187.0
7,500	FM/BM	B	737.0	50	118.3	123.8	126.2
7,500	FM/BM	B	731.4	80	103.7	107.7	110.3
10,000	FM/BM	A	727.0	50	88.3	91.0	92.0
10,000	FM/BM	A	730.2	80	76.3	82.1	86.5

TABLE IV
SUMMARY OF FM/BM CANISTER BREAKTHROUGH TIME DATA AGAINST VARIOUS PENTANE CHALLENGE CONCENTRATIONS

CHALLENGE CONCENTRATION PPM	TYPE*	CHARCOAL WT (gm)	%RH	t_b CORRECTED TO CHALLENGE CONCENTRATION		
				5PPM	10PPM	15PPM
5,000	FM/BM	730.9	50	153.3	157.7	160.6
5,000	FM/BM	757.4	80	139.4	145.2	148.6
7,500	FM/BM	731.6	50	119.9	122.5	124.4
7,500	FM/BM	729.7	80	107.5	110.4	112.5
10,000	FM/BM	726.9	50	95.9	98.0	99.6
10,000	FM/BM	755.8	80	97.0	100.3	102.6
12,000	FM/BM	739.2	50	82.9	86.0	87.7
12,000	FM/BM	730.2	80	80.0	81.9	83.2

*Canisters from lot A

TABLE V
SUMMARY OF FM/BM CANISTER BREAKTHROUGH TIME DATA AGAINST VARIOUS n-HEXANE CHALLENGE CONCENTRATIONS

CHALLENGE CONCENTRATION PPM	TYPE**	CHARCOAL WT (gm)	%RH	t_b CORRECTED TO CHALLENGE CONCENTRATION		
				5PPM	10PPM	15PPM
2,000	FM/BM	742.2	50	289.7	300.9	310.2
2,000	FM/BM	739.4	80	271.0	285.6	300.1
5,000	FM/BM	769.4	50	164.9	167.6	168.9
5,000	FM/BM	760.5	80	157.0	161.9	164.8
7,000	FM/BM	743.5	50	121.3	124.8	126.9
7,000	FM/BM	749.7	80	111.9	119.0	121.7
9,000*	FM/BM	752.4	50	95.7	99.8	101.4
9,000*	FM/BM	743.7	50	85.8	88.9	90.4
9,000*	FM/BM	754.6	80	105.0	111.4	113.7

*Canisters got very warm and observed baseline shift.
**Canisters from lot A

TABLE VI

ORGANIC VAPOR FM/BM CANISTER BREAKTHROUGH TIME CORRELATION DATA

TEST SUBSTANCE/CONDITION	CHALLENGE CONCENTRATION PPM	% RELATIVE HUMIDITY	RUN	SORBENT WEIGHT (gm)	B_T 5 PPM MIN
Carbon Tetrachloride, "as received," 64 lpm, 25°C	10,000	50	1	743.0	125.9
			2	748.6	97.5
			3	728.0	101.0
			4	723.8	120.6
		80	1	748.3	99.4
Carbon Tetrachloride, preconditioned 85%RH, 32 lpm, 25°C	10,000	50	1	731.7	172.8
Carbon Tetrachloride, "as received," 64 lpm, 25°C	20,000	50	1	733.0	37.8
			2	744.1	39.4
			3	741.8	52.1
			4	732.8	52.3
Carbon Tetrachloride, preconditioned 85%RH, 32 lpm, 25°C	20,000	50	1	729.8	16.8
			2	731.2	23.0
Pentane, "as received," 64 lpm, 25°C	10,000	50	1	725.7	80.1
			2	716.7	73.5
			3	748.2	96.7
		80	1	742.1	75.7
			2	748.7	72.0
			3	753.2	73.4
			4	746.3	94.4
			5	746.5	82.1
Pentane, preconditioned 85% RH, 32 lpm, 25°C	10,000	50	1	746.2	122.1
			2	743.4	128.3