

Interoffice Communication

To P. L. Fetzer ✓
 From H. L. Hackett
 Date August 12, 1975
 Subject Evaluation of Bendix Flasher for Ambient Air Analysis

SUMMARY AND CONCLUSIONS: Good results were obtained for vinyl chloride but more work would be required to demonstrate a satisfactory analysis for the higher boiling materials such as ethylene dichloride. Use of temperatures above 200°C to speed vaporization into the chromatograph column lead to cracking of ethylene dichloride and ethyl chloride with subsequent above theoretical recovery of vinyl chloride. Considerable mechanical problems with leaks were encountered especially around the tube seals.

It is recommended that the Bendix Flasher Unit be purchased for monitoring where vinyl chloride exposure is the only concern.

INSTRUMENT DESCRIPTION: The Bendix Flasher is basically a small tube furnace, temperature controlled, for vaporization of components absorbed on charcoal into the inlet carrier gas stream of a gas chromatograph. Current cost of the instrument is \$735. Several advantages are claimed for the unit over conventional solvent desorption/gas chromatography methods of analysis of ambient air samples.

1. No solvent is used which eliminates interfering peaks and the weighing and handling of hazardous liquids.
2. The entire amount of components from the air volume sampled is injected into the gas chromatograph. Thus the volume of air sampled and the amount of charcoal used can be smaller without exceeding its capacity and still give good sensitivity.

With the Bendix Flasher system ambient air is drawn through a 3 x 1/4 inch diameter tube packed with charcoal or some other material. A proprietary modified charcoal is recommended and supplied by Bendix for this purpose. When the sample has been collected it is placed in the "Flasher". A hinged sealing mechanism isolates the tube inside the hinged knives of the tube furnace between two solenoid valves. Two minutes are allowed for the tube to come to the set temperature then the inject switch is energized manually. This switches the (normally by-passed) carrier gas flow through the sample loop into the chromatograph.

DISCUSSION: Most of the current work with the Bendix Flasher involved a comparison of the relative peak areas of a standard gas mixture (50-55 ppm each of VCM, EtCl, and LDC in nitrogen) vaporized from sample tubes with the peak areas from the same volume injected directly into the chromatograph.

Three principal methods or modifications were investigated.

METHOD I - USE OF PCB AND BENDIX MODIFIED CHARCOALS - With PCB 12 x 30 mesh charcoal in the tubes and a tube furnace temperature of 180°C essentially 100 percent recoveries of vinyl chloride and ethyl chloride were obtained but only 50 percent recovery of ethylene dichloride.⁽¹⁾ As shown in Graph 1, increasing the furnace temperature to 220°C gave better EDC recovery, 73.7 percent, but some cracking leading to above theoretical recovery of vinyl chloride. Cracking (dehydrohalogenation) of both EDC and ethyl chloride was extensive at 250°C. Thus, the Bendix Flasher using PCB charcoal appears to work satisfactorily for VCM and relatively low boiling compounds such as ethyl chloride, but not for EDC.

1. Results reported were obtained using a 6 foot x $\frac{1}{2}$ " Porapak Q Column in a Bendix gas chromatograph at the Chemical Plant Laboratory.

The modified charcoal supplied and recommended by Bendix was difficult to condition (remove small amounts of absorbed interfering compounds) and did not give sharp symmetrical peaks for EDC.

METHOD II - USE OF TENAX POROUS POLYMER - Various porous polymers have been suggested to replace charcoal. TENAX (supplied by Applied Science Laboratories via L.M.V., Holland) was tried and is more or less typical of these polymers. Good EDC recoveries were obtained as long as the vaporizer temperature was not too high (ca. 220°C optimum) but results for VCM and ethyl chloride were low as shown in Table I.

METHOD III - USE OF DUAL ZONE ABSORPTION TUBES - Since the low results for VCM and ethyl chloride above might be due to incomplete retention of these components on the TENAX a dual zone tube was tried. These tubes were filled approximately half full with PCB charcoal and the rest of the way with TENAX. Both the slow introduction to, and elution from, the sample tube were at the TENAX end. The purpose of the charcoal was to trap any components not completely absorbed in the polymer. This procedure resulted in poorly resolved and tailing vinyl chloride and ethyl chloride peaks. Recovery data were erratic and inconclusive from these trials.

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Es

CC: JAD, JEM, CGD, JF, JSR

TABLE I
USE OF TENAX AND TENAX/PCB SAMPLE TUBES

TUBE PACKING	FLASHER TEMPERATURE °C	RECOVERY		
		VCM	EtCl	EDC
TENAX	180	57.7	86.1	87.7
TENAX	220	78.0	63	124.1
TENAX	250	134	79.6	54.8
TENAX/PCB ⁽¹⁾	180	80.8	101.3	89.3
TENAX/PCB ⁽¹⁾	220	125	tr.	58.4

(1) Tailing and incompletely resolved VCM and EtCl peaks.

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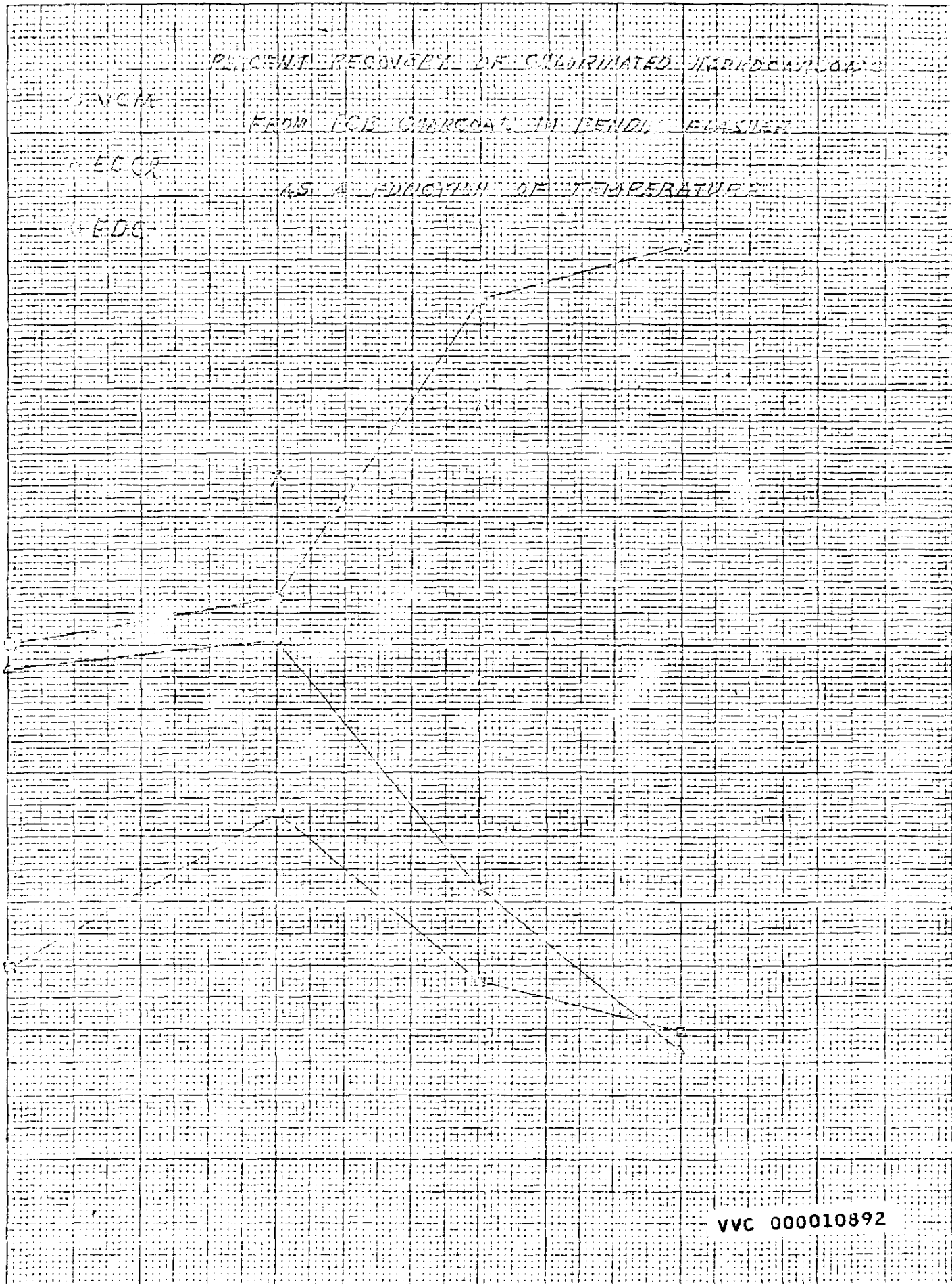
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PERCENT RECOVERY

PERCENT RECOVERY OF CHLORINATED HARDENING OIL
FROM LOB CHARCOAL IN BENDI EMASHER
AS A FUNCTION OF TEMPERATURE

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10



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BENDIX HS-10 FLASHER AND PERSONNEL MONITORING SYSTEM

A SUMMARY REPORT OF CURRENT DATA

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I. SAMPLING TECHNIQUES

A. Bendix Personnel Monitoring Collection Column (PMCC).

The Bendix PMCC modified charcoal* is the best packing material we have found for use as a collection medium for organic industrial type vapors. This packing allows long sampling times (greater than 24 hours in selected cases) for VCI and other vapors. This packing also allows the collected vapors to be quickly thermally desorbed at reasonable temperatures, e.g., 200-300°C., thereby eliminating the possibility of thermal degradation of most vapors during analysis. The percent desorption of VCI from the PMCC is greater than 98%. Equal concentrations of VCI, which were loaded onto both Bendix modified charcoal and Porapak Q, when thermally desorbed gave identical responses. Since Porapak Q is known to desorb greater than 98% of its trapped organic vapors, it is reasonable to assume the Bendix modified charcoal also desorbs greater than 98% of its VCI.

Also, the excellent linearity curve shown in Fig. 3 supports the conclusion that thermally desorption is essentially 100%.

B. Bendix PMCC and Micronair Pump

1. Test Methodology

For time-weighted averaged samples it is generally best to operate the sample pump and PMCC at low flow rates (10 cc/min or less).

In an effort to test the performance of the Bendix Micronair pump and PMCC at low flow rates (2 cc/min) this sampling system was allowed to operate in an uninterrupted manner for nearly eight (8) hours.

Flow rates were periodically checked with a bubble meter and stop watch. (Figure 1)

*Bendix modified charcoal is proprietary information. Patent pending.

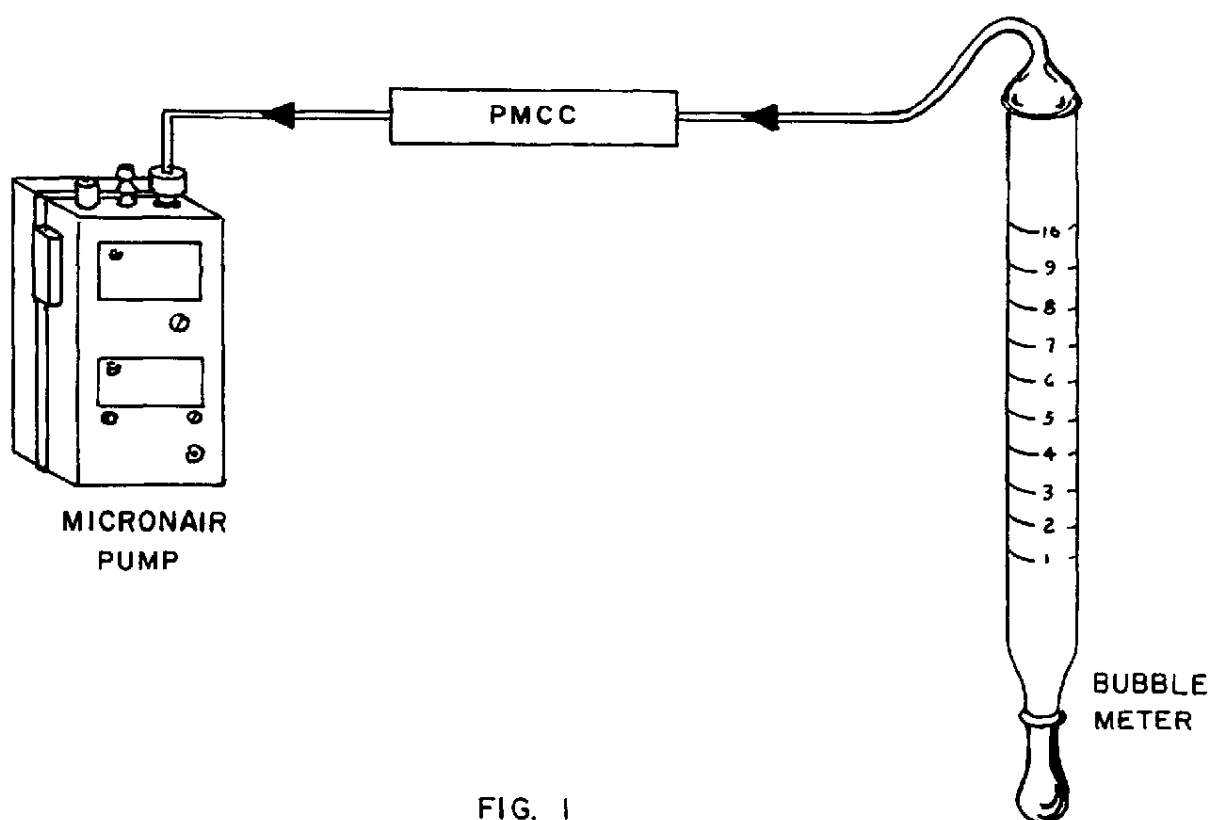


FIG. 1

2. Sampling Performance Data

Detailed sampling data are shown in Tables I and III. Summarized data are presented below.

FOUR-HOUR PERFORMANCE DATA

Average flow rate	1.79 cc/min
Average deviation from the mean	0.007 cc/min
Standard deviation (σ)	0.04
Percent average variation	1.64%

SEVEN-HOUR FORTY-MINUTE PERFORMANCE DATA

Average flow rate	1.77 cc/min
Average deviation from the mean	0.01 cc/min
Standard deviation (σ)	0.06
Percent average variation	2.88%

C. Bendix PMCC and Longer Life Pumps

While the Micronair pump data presented above is adequate for 4-8 hour personnel monitoring, it is recognized that this pump is not capable of performing for longer periods of time without recharging the battery. The Sipin pump was found to be comparable to the Bendix Micronair.

For sampling times of twenty four hours or more in a stationary mode, the line operated pumps are feasible.

II. PMCC RETENTION OF VINYL CHLORIDE DURING SAMPLING

A. Break-through Test Methodology

Sampling retention data was obtained by placing the PMCC in series with a VCI cylinder and Flame Ionization Detector (FID) at 32°C and 25°C. Special tests to determine the effect of water vapor were conducted by passing the VCI through a water bath prior to the PMCC. Flow rates through the PMCC were determined with a bubble meter. A positive change in FID response indicated VCI breakthrough had occurred. Both sample rates and sample concentrations were varied during these tests. (Figure 2)

B. Most of our breakthrough data are summarized in Tables IV and V. Three factors appear to control VCI sample breakthrough volumes. They are:

1. Sample Concentration
2. Sample Flow Rate
3. Humidity

Extreme temperature and humidity (32°C, 95% R.H.) tests with a 40-ppm VCI sample @ 5 cc/min flow rate gave no breakthrough after 55 hours.

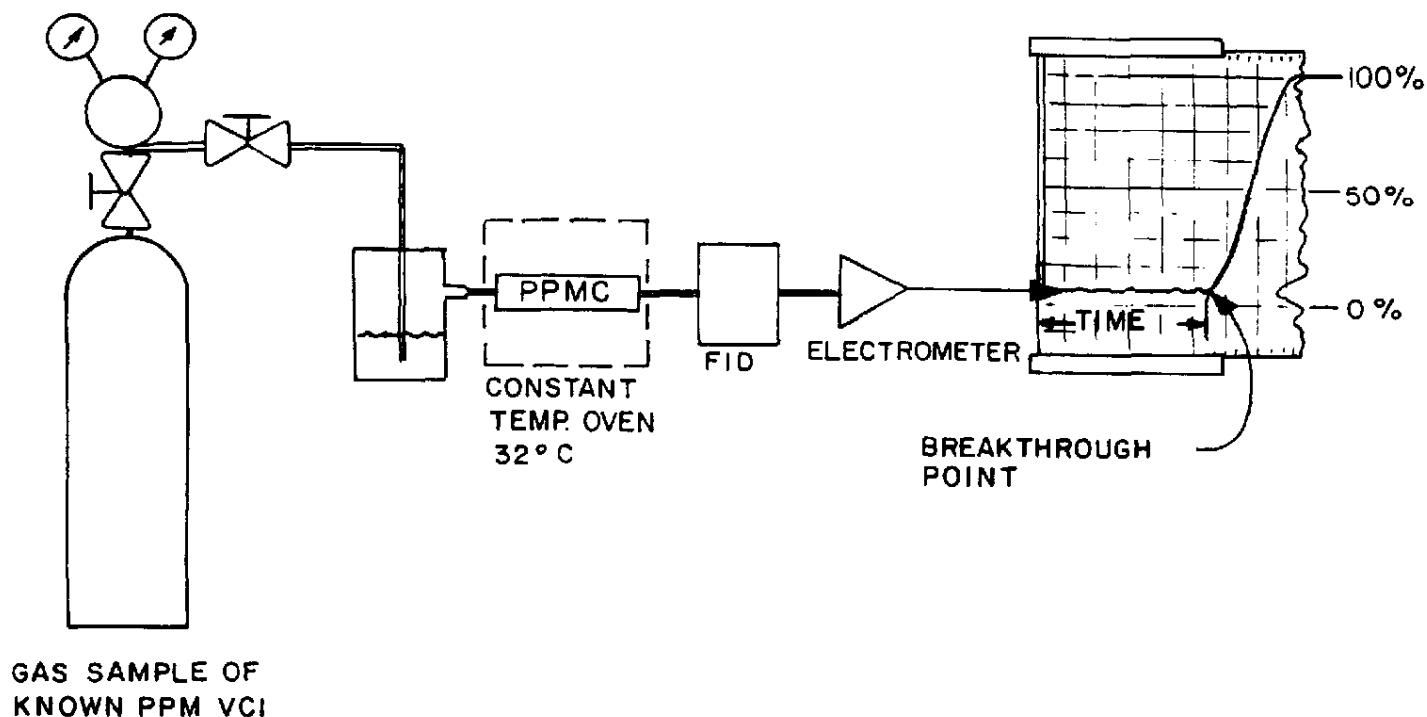


FIG. 2

III. BENDIX FLASHER ANALYTICAL TECHNIQUE FOR VCL

A. Reproducibility of PMCC-to-PMCC

1. Test Methodology

For this test thirty-six (36) individual PMCC tubes were loaded with 150 cc of an uncertified sample of 10 PPM Vinyl Chloride over a period of 4 hours. The PMCC's were analyzed in batches of twelve (12) during a 48-hour period with the following equipment and parameters:

EQUIPMENT:

Bendix 2500 G.C.

350 VDC FID

4 ft. X 1/8 in. Porapak Q 60/80
Column @ 100°C

Electrometer 2×10^{-9} afs

0-10mv Recorder and Autolab 6300 Integrator

Bendix Flasher

Temp. 250°C

Heat Time 2 min

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2. Reproducibility Data of VCI PMCC-to-PMCC

STAT	BATCH JP-716A	BATCH JP-716B	BATCH JP-716C	BATCH JP-716A-B-C
Ave. Count*	17600	19200	18400	18400
% Ave. Var.	3.5%	2.1%	1.6%	2.4%
% Max. Var.	9.7%	5.7%	4.3%	9.2%
95% Conf'd (2σ)	16	10.2	8.4	11.6
Std. Dev. (σ)	7.9	5.1	4.2	5.8

*FID Detector Response by Integrator Count $\times 10^{-2}$

B. Linearity with the Bendix Flasher

1. Test Methodology

In a typical test a PMCC was loaded with different volumes of a 10-PPM Vinyl Chloride sample from a premixed gas cylinder of VCI. Sample volumes varied from 50 cc to 10,000 cc. Sample rates varied from 50 cc/min to 1,000 cc/min. The samples were thermally desorbed at 250°C with the Flasher into a 6 ft X 1/8 in. Porapak Q 60/80 mesh analytical column with a 100°C isothermal oven temperature. Sample response to a flame ionization detector was determined with an Autolab 6300 digital integrator and 0-10mv strip chart recorder.

2. Linearity Test Data

The linearity curve shown in Figure 3 was obtained by plotting integrator-detector response versus VCI sample volume. Good linearity was observed over the entire sample range. More data are needed for the low parts-per-billion (PPB) range both for linearity as well as sensitivity studies.

These data are contingent upon arrival of low VCI permeation wafers from Metronics, Inc.

The good linearity plot also indicates that essentially all the Vinyl Chloride is thermally desorbed from the PMCC tubes. This opinion has been fortified by observing that similar tubes packed with porous polymers and loaded with equal volumes (100 cc) of 10-PPM VCI gave the same detector response as the Bendix modified charcoal.

C. Sensitivity with the Bendix Flasher and FID Detector

1. Test Data

Test data indicates excellent sensitivity at the 1-10 PPM range. For example, a 150 cc sample of 10 PPM Vinyl Chloride gives Ca. 50% full scale response on a 0-10mv chart recorder at an electrometer setting of 2×10^{-9} afs. The same sample gives an integrator count of approximately 18000 counts on a autolab digital integrator (cite III.A.2.). Theory predicts excellent sensitivity in the low ppb range which is discussed below.

2. Theory and Projected FID Sensitivity

The combination of the PMCC sampling technique and Flasher thermal desorption technique offers a potential analytical method capacity for monitoring 1 PPB or less of Vinyl Chloride. The ability of the PMCC packing to retain VCI and other organic vapors from large volumes ($>10^4$) of ambient air offers an excellent means for concentrating these vapors. Furthermore, since the Flasher thermal desorption technique requires no solvent desorption, and consequently no sample dilution, the entire concentrated sample is available for analysis. For example, a 10-liter sample of 1 PPB VCI could provide $0.25 \mu\text{g}$

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(250 ng) to the detector which is well within the sensitivity limits of a FID detector. Compared with the carbon disulfide method, the Flasher method affords approximately 5000 times more sample per analysis.

IV. VINYL CHLORIDE SAMPLE DIE-OUT ON STORED PMCC TUBES

In an effort to determine the degree of sample die-out versus time, we loaded several PMCC's with equal volumes of a freshly mixed 5.3 PPM VCI and zero air in a cylinder at 100 psig. Individual samples were then analyzed after storage for 15 days. A calibration sample was run on each tube immediately following its analysis. Percent recovery of the stored VCI ranged from 85% - 98%. Average percent recovery (14 values) was 91% (Table VI).

V. BENDIX PMCC-FLASHER TECHNIQUE FOR OTHER INDUSTRIAL VAPORS

The PMCC-Flasher system has been observed to perform well for the other OSHA specified industrial vapors listed in Table VII. Good peak shape has been observed for all the compounds listed. Typical chromatograms are shown in Figures 4 and 5.


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PMCC MICRONAIR PUMP DATA

TABLE I
Low Pump Rate Data

<u>Time</u>	<u>Sec/1 cc</u>	<u>cc/min</u>	<u>cc/min</u>	<u>cc/hr</u>	<u>cc/4-hr</u>
10:05 AM	33.5, 33.2, 33.0	1.79, 1.81, 1.82	1.81	108.6	434.4
10:45	34.5, 33.0, 32.0	1.74, 1.82, 1.87	1.81	108.6	434.4
12:15 PM	33.0, 33.5, 33.5	1.82, 1.79, 1.79	1.80	108.0	432.0
1:07	34.5, 35.0, 33.0	1.74, 1.71, 1.82	1.76	105.6	422.4
2:05	33.6, 33.6, 33.2	1.78, 1.78, 1.81	1.79	107.4	429.6
2:55	35.0, 34.0, 36.0	1.71, 1.76, 1.67	1.71	102.6	410.4
4:30	33.6, 33.0, 34.0	1.78, 1.82, 1.76	1.79	107.4	429.6
5:45	35.5, 36.5, 36.6	1.69, 1.64, 1.64	1.66	99.6	398.4

STAT ON PMCC MICRONAIR PUMP DATA

TABLE II

STAT on 4.0 hr (Initial) Performance (cc/min Data)

<u>Time</u>	<u>cc/min</u>	<u>Δ</u>	<u>Δ^2</u>
10:05 AM	1.79, 1.81, 1.82	.00, .02, .03	.0000, .0004, .0009
10:45	1.74, 1.82, 1.87	.05, .03, .08	.0025, .0009, .0064
12:15 PM	1.82, 1.79, 1.79	.03, .00, .00	.0009, .0000, .0000
1:07	1.74, 1.71, 1.82	.05, .08, .03	.0025, .0064, .0009
2:05	1.78, 1.78, 1.81	.01, .01, .02	.0001, .0001, .0004
	$h = 15$	$h = 15$	$h = 15$
	$\sum h = 26.89$	$\sum \Delta = 0.44$	$\sum \Delta^2 = 0.0224$

$$\text{AVE} = \sum h/h = 26.89/15 = 1.79 = \text{Average}$$

$$\text{a.d.} = \frac{\sum \Delta}{h} = \frac{0.44}{15} = 0.029 = \text{Ave. Deviation}$$

$$\text{A.D.} = \frac{\text{a.d.}}{\sqrt{h}} = \frac{0.029}{\sqrt{15}} = 0.007 = \text{Ave. Dev. from the mean}$$

$$\sigma = \frac{\sum \Delta^2}{h-1} = \frac{0.0224}{14} = 0.04 = \text{Std. Dev.}$$

$$2\sigma = 2(.04) = 0.08 = 95\% \text{ Confidence Level}$$

$$\% \text{VAR}_{\text{AVE}} = \frac{\sum \Delta^2}{(\sum h)^2} (100) = \frac{0.0224}{(26.89)^2} (100) = 1.64\% = \% \text{AVE. Variance}$$

STATISTICS ON PMCC MICRONAIR PUMP DATA

TABLE III

STAT on 7 hr-40 min Performance (cc/min data)

Time	cc/min	Δ	Δ^2
10:05 AM	1.79, 1.81, 1.82	.01, .04, .05	.0004, .0016, .0025
10:45	1.74, 1.82, 1.87	.03, .05, .10	.0009, .0025, .0100
12:15 PM	1.82, 1.79, 1.79	.05, .02, .02	.0025, .0004, .0004
1:07	1.74, 1.71, 1.82	.03, .06, .05	.0009, .0036, .0025
2:05	1.78, 1.78, 1.81	.01, .01, .04	.0001, .0001, .0016
2:55	1.71, 1.76, 1.67	.06, .01, .10	.0036, .0001, .0100
4:20	1.78, 1.82, 1.76	.01, .05, .01	.0001, .0025, .0001
5:45	1.69, 1.64, 1.64	.08, .13, .13	.0064, .0169, .0169

$$\Sigma = 42.36, \quad n = 24$$

$$\text{AVE} = 1.77$$

$$\Sigma \Delta = 1.22$$

$$\Sigma \Delta^2 = 0.0839$$

$$\text{a.d.} = \frac{\Sigma \Delta}{n} = \frac{1.22}{24} = 0.051 = \text{Ave. Deviation}$$

$$\text{A.D.} = \frac{\text{a.d.}}{\sqrt{n}} = \frac{.051}{\sqrt{24}} = 0.010 = \text{Ave. Dev. from the Mean}$$

$$\sigma = \frac{\Sigma \Delta^2}{n-1} = \frac{.0839}{23} = 0.00365 = \text{Std. Dev.} \quad \left. \vphantom{\frac{\Sigma \Delta^2}{n-1}} \right\} 2\sigma = 0.12 = 95\% \text{ Confidence Le}$$

$$\% \text{ VAR}_{\text{AVE}} = \frac{\Sigma \Delta}{\Sigma n} (100) = \frac{1.22}{42.36} (100) = 2.88\%$$

TABLE IV
PMCC Vinyl Chloride Breakthrough Data

<u>Packing</u>	<u>PPM VCI</u>	<u>Flow Rate cc/min</u>	<u>Sample Time</u>	<u>Sample Volume, Liters</u>	<u>Breakthrough Time</u>	<u>Breakthrough Volume</u>
Bendix Modified Charcoal	50	60	4.0 hr +	14.4 <i>ℓ</i>	> 4 hr.	> 14.4 <i>ℓ</i>
	10	1000	0.5 hr +	30 <i>ℓ</i>	> 0.5 hr	> 30 <i>ℓ</i>
	100	100	1.5 hr	9.0 <i>ℓ</i>	1.5 hr	9.0 <i>ℓ</i>
	1000	100	0.55 hr	3.2 <i>ℓ</i>	0.55 hr	3.2 <i>ℓ</i>
	10,000	1000	2 min	2.0 <i>ℓ</i>	> 2.0 min	> 2.0 <i>ℓ</i>
	10,000	1000	4 min	4.0 <i>ℓ</i>	< 4.0 min	< 4.0 <i>ℓ</i>
	50*	100	118 min	11.8 <i>ℓ</i>	118 min	11.8 <i>ℓ</i>
	100*	100	29 min	2.9 <i>ℓ</i>	29 min	2.9 <i>ℓ</i>
	100	100	80 min	8.0 <i>ℓ</i>	80 min	8.0 <i>ℓ</i>
	5.3**	5	33.0 hr	9.9 <i>ℓ</i>	> 33 hr	> 9.9 <i>ℓ</i>

*Water saturated VCI samples (95% Rel. Hum.)

**5.3 PPM Certified Precision Gas, Inc. Sample in zero air

TABLE V
Extreme Ambient Sampling Conditions

Sample 41.8 PPM VCI (Standardized Sample)

PMCC Packing Bendix Modified Charcoal

Temperature 90°F (32°C)

Rel. Humidity 95% R.H.

<u>Flow Rate cc/min</u>	<u>Sample Time</u>	<u>Sample Volume liters</u>	<u>Breakthrough Time</u>	<u>Breakthrough Volume</u>
5 cc/min	55 hr	16.5 <i>ℓ</i>	> 55 hr	> 16.5 <i>ℓ</i>
10 cc/min	15.5 hr	9.3 <i>ℓ</i>	15.5 hr	9.3 <i>ℓ</i>
25 cc/min	6.5 hr	9.7 <i>ℓ</i>	6.5 hr	9.7 <i>ℓ</i>

TABLE VI
VINYL CHLORIDE RECOVERY FROM STORED PMCC'S

Sample 300 cc of 5.3 PPM Vinyl Chloride
 Date Sampled 11-26-74
 Date Analyzed 12-13-74

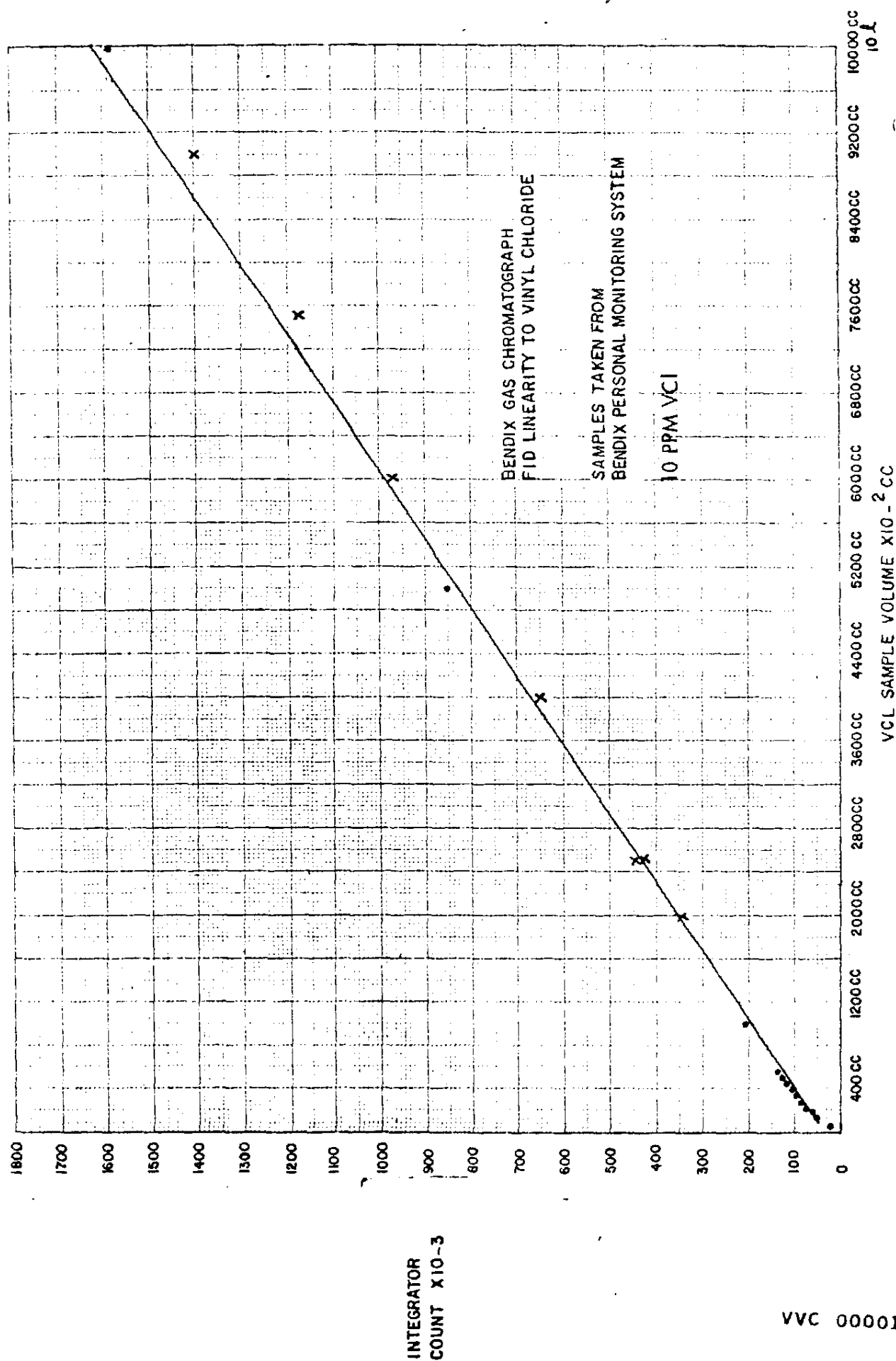
<u>PMCC</u>	<u>PERCENT RECOVERY OF VINYL CHLORIDE</u>
PMCC #1	85.6%
PMCC #2	87.0
PMCC #3	95.0
PMCC #4	95.5
PMCC #5	98.0
PMCC #6	94.0
PMCC #7	92.0

Average Percent Recovery 91.0%

TABLE VII
OSHA Specified Vapors

1. Benzene
2. Carbon tetrachloride
3. Chloroform
4. Dioxane
5. Ethylene dichloride
6. Methyl chloroform
7. Methyl ethyl Ketone
8. Styrene
9. Tetrachloroethylene
10. Toluene
11. Trichloroethylene
12. 1,1,2-Trichloroethane
13. Xylene

FIGURE 3



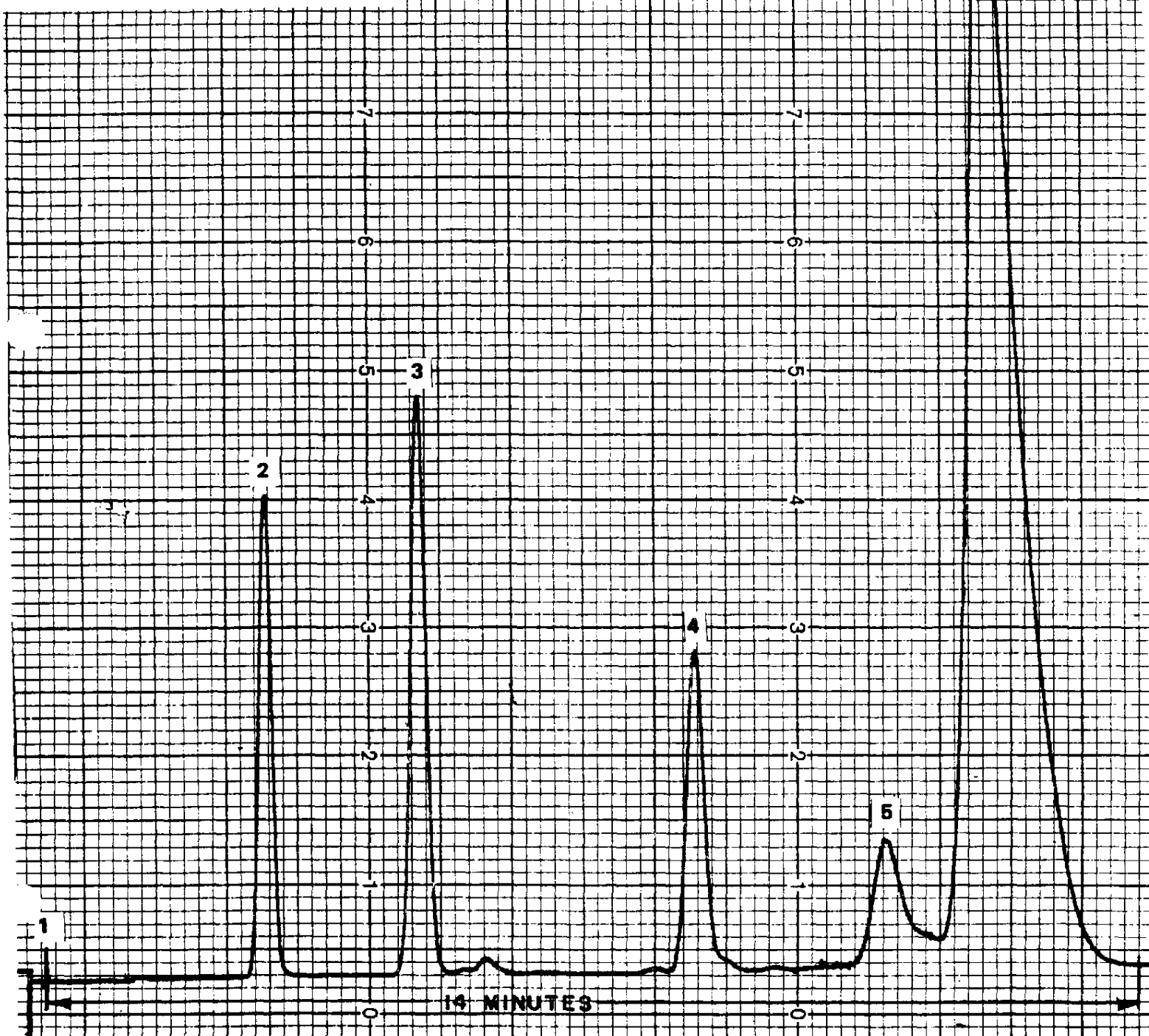
LINEARITY CURVE

FIGURE 4

PERSONNEL MONITORING COLLECTION

IMN (PMCC):
ANALYTICAL COLUMN:
8 FT X 1/8 INCH SS PPN 80/100
FID DETECTOR
N₂ CARRIER
ELECTROMETER @ 2 X 10⁻⁹ afs
LEGEND:

1. PMCC INSERT (FLASHER @ 200°C) TEMP PROGRAM
- ANALYTICAL COLUMN 40-150°C @ 100°/MIN
2. 1,2-DICHLOROETHANE (CLCH₂CH₂CL)
3. CHLOROFORM (CHCL₃)
4. 1,1,2-TRICHLOROETHANE (CL₂CH-CH₂-CL)
5. 1,1,2,2-TETRACHLOROETHANE (CL₂CHCHCL₂)
6. BENZENE (C₆H₆)



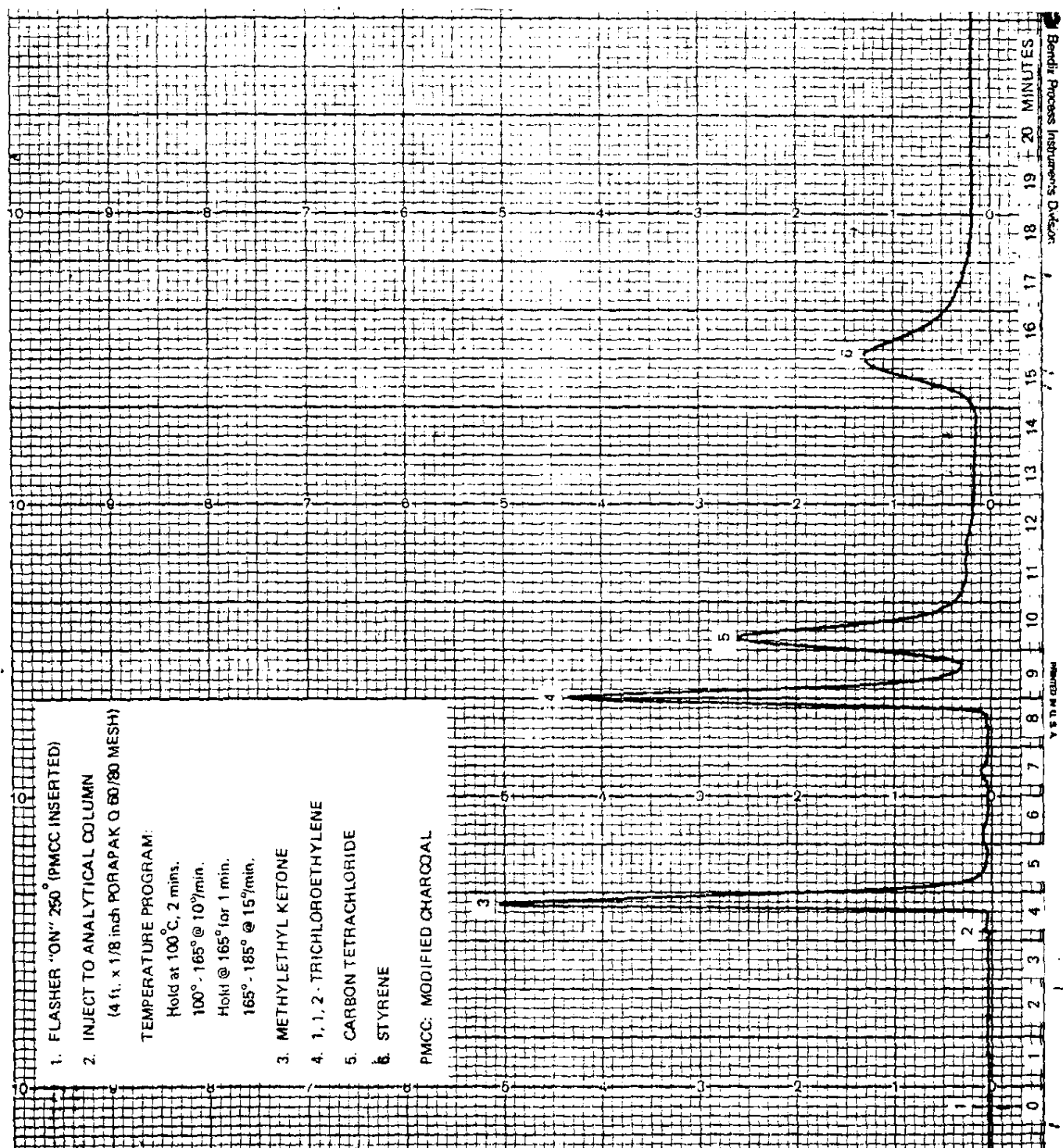


FIGURE 5

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