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Interoffice Communication

To P. L. Fetzer

From Stephen C. Racca

Date August 15, 1980

Subject Evaluation of 3-M's OVM Dosimeters in Personnel Monitoring for Vinyl Chloride and 1,2-Dichloroethane.

Summary

Experimental laboratory work was done to evaluate the effectiveness of the 3M Organic Vapor Monitor. Proposed objectives for this study were:

1) Determination of saturation level of ethylene dichloride (EDC) and vinyl chloride (VCM).

2) Establishment of minimum and maximum detectable levels for EDC and VCM.

These objectives as well as those encountered during the course of this study were examined and conclusions drawn.

Conclusions

1) The saturation level of ethylene dichloride (EDC) is 45.16 milligrams.

2) The saturation level of vinyl chloride (VCM) is 9.85 milligrams.

3) The minimum detectable level of EDC is 0.024 ppm for an 8 hour exposure period.

4) The maximum detectable level for EDC is greater than or equal to 3121 ppm hours or 390 ppm for an 8 hour exposure period.

5) The maximum detectable level for VCM is not a constant. It is dependent upon the concentration of VCM and the environmental conditions of the exposure. This dependency is a direct consequence of 2 factors:

   1) The collection efficiency of the sorbent medium is less than 100%.

   2) VCM is held very weakly to this medium.

Recommendations

1) The plexiglas boxes used in the exposure system of Figure 3 be reconstructed of carbon steel or stainless steel. The inside dimensions of these exposure chambers should remain the same as those used for the plexiglas chambers.

2) The present personnel monitoring procedures utilizing the 3M OVM and the analysis steps outlined in Appendices I & II be continued.

Proposed Future Studies

1) Further testing of the REAL Minimonitor should be conducted. These tests should not only where this study left off but the response of the Minimonitor to organics other than vinyl chloride, such as ethylene dichloride, should also be considered.

2) Testing of other currently available dosimeters as well as new developments to find a dosimeter suitable for the monitoring of personnel exposure to both light and heavy organics should be conducted. The emphasis of these tests should be on the monitoring of ethylene dichloride and vinyl chloride.

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Introduction

The original purpose of this study was to determine the saturation level of EDC & VCM on the 3M OVM and to establish the minimum and maximum detectable levels for vinyl chloride monomer (VCM) and ethylene dichloride (EDC) using the 3M OVM and the Perkin-Elmer F-42 head space gas chromatograph. During testing and evaluation of the 3M OVM several important observations were made which necessitated deviation from original objectives in order to establish the reliability and accuracy of the 3M OVM in various environmental situations. These observations and the analytical results will be given later in this report.

The 3M Organic Vapor Monitor No. 3500 is one of several passive dosimeter monitoring systems currently available for determining time weighted average exposure to organic vapors. It is available from the Occupational Health and Safety Products Division of the 3M Company. The monitor utilizes a porous membrane to separate the wearer's environment from the charcoal pad collection medium on the interior. The purpose of this membrane is to create a convection free area on the inside of the badge while controlling the rate of transfer of organic vapors from the environment to the interior. The mode of transport across this membrane is diffusion in which gas molecules pass through pores in the membrane from an area of high concentration to an area of lower concentration. Once gas molecules reach the interior of the badge they are presumedly irreversibly adsorbed on a collection medium, in this case a charcoal pad. The 3M OVM is convenient, does not interfer with the wearer's performance, and is relatively inexpensive, although it is limited to the monitoring of those organic gases that adsorb strongly on charcoal surfaces.

During this study two other passive dosimeters were tested in order to provide comparative observations. These badges were the Protek G-BB from E. I. DuPont de Nemours & Co., and the Real Minimonitor from Reiszner Environmental & Analytical Labs., Inc. The Protek G-BB monitor works on the same principles of diffusion and adsorbtion as the 3M OVM although the Protek monitor has an additional backup section. The Real Minimonitor utilizes a Dimethyl Silicone (DMS) membrane to separate the environment from the interior. The mode of transport across this membrane is permeation where gas molecules come in contact with the membrane, are dissolved and penetrate the membrane. Once the gas molecules penetrate this membrane they are adsorbed on a colletion medium, in this case PCB 12 x 30 charcoal activated at 350°C under a flow of nitrogen. The Protek monitor affords the same merits as the 3M OVM together with the advertised advantage of a back-up section. The Real Minimonitor is approximately the same size as the 3M OVM but it contains approximately 7 times more collection medium by weight. The Real Minimonitor is more expensive than the 3M OVM but the added expense is offset by the fact that it is reusable when recharged with collection medium while both the 3M OVM and the Protek G-BB are disposable.

Experimental

The N,N-Dimethylacetamide (DMA) used for desorbtion of the collection media was certified grade supplied by Fisher Scientific Company and MCB Manufacturing Chemists, Inc. Both brands of DMA were checked for contaminants that might interfere with analysis and none were found. The standard gases that were used to generate known concentration levels for exposing calibration standards and sample badges were of certified composition and were supplied by Big Three Industries, Inc., and Airco, Inc..

The exposure chamber used to challenge exposed badges to zero organic vapor concentration atmospheres of dry nitrogen and humidified nitrogen (70% RH) at a rate of 700 ml/min was constructed of plexiglas (8½ inches long x 8½ inches wide x 2½ inches deep). All fittings used for connections in the system were ¼ inch stainless steel. The tubing used was either ¼ inch stainless steel or rubber. A water bubbler was used on the inlet to achieve a relative himidity of between 65 to 70%. The flowmeter used was a Gilmont No. 13 (0 to 12 standard liters-per minute). A U-tube with water in it was used on the outlet to insure flow exiting the system to the hood rather than through a leak in the system. The lid of the plexiglas chamber was sealed with vinyl electrical tape. An illustration of this setup can be found in Figure 1.

The exposure chamber used to expose badges to known concentration levels of organic vapors also was constructed of plexiglas (24 3/4 inches long x 2 1/2 inches wide x 1 3/8 inches deep). All fittings and lines used in the system were 1/4 inch stainless steel except for short pieces of rubber tubing used to connect the pump and the U-tube. The flowmeter used in this arrangement was also a Gilmont No. 13. The flow of standard gas from the cylinder was maintained at 3 liters per minute. The pump used to recirculate gases in order to obtain a velocity of at least 30 fpm in the exposure chamber was a Bendix Air Sampling Pump Type C115. The U-tube was used to assure flow out of system as in the previously described system. The lid of the chamber was also sealed with vinyl electrical tape. An illustration of this system is in Figure 2.

A new exposure system was designed and built during the course of this study. The reason for constructing this new apparatus will be presented later in this report. For this new exposure system 10 plexiglas chambers (4 5/8 inches long x 3 inches wide x 1 1/8 inches deep) are connected in parallel by 1/4 inch stainless steel tubing and fittings. Quarter inch ball valves were installed at the inlet and outlet of each box to allow removal of a single badge without interrupting flow to the remaining badges. The flowmeter used at the outlet of the system was a Gilmont No. 13. The flow at the outlet was maintained at 3 liters per minute. The pump used for recirculation to achieve a 30 fpm velocity in each chamber was a Lammert Oil-Less Vacuum Pump with a capacity of 40.7 liters per minute with zero head. An illustration of this setup can be found in Figure 3.

The exposures from the badges were analyzed by first placing the collection medium in a 1/4 ounce vial and capping with a rubber septum and crimp type cap. The vials were then stored in a freezer at -20°C until time for analysis. The steps outlined in Appendix II were then followed using the Perkin-Elmer F-42 Head Space Gas Chromatograph and the Spectra-Physics SP-4000 Central Processor. For the 3M OVM and the Protek G-BB 2cc N,N-Dimethylacetamide was used for desorbtion. For the Real Minimonitor 3cc DMA was required.

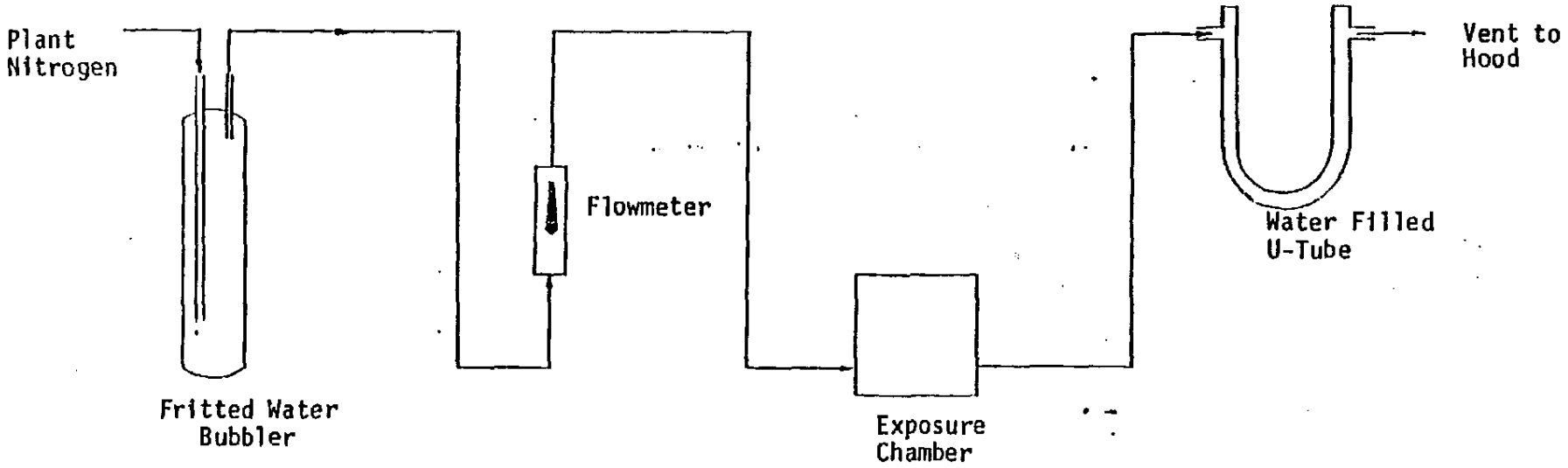

Figure 1

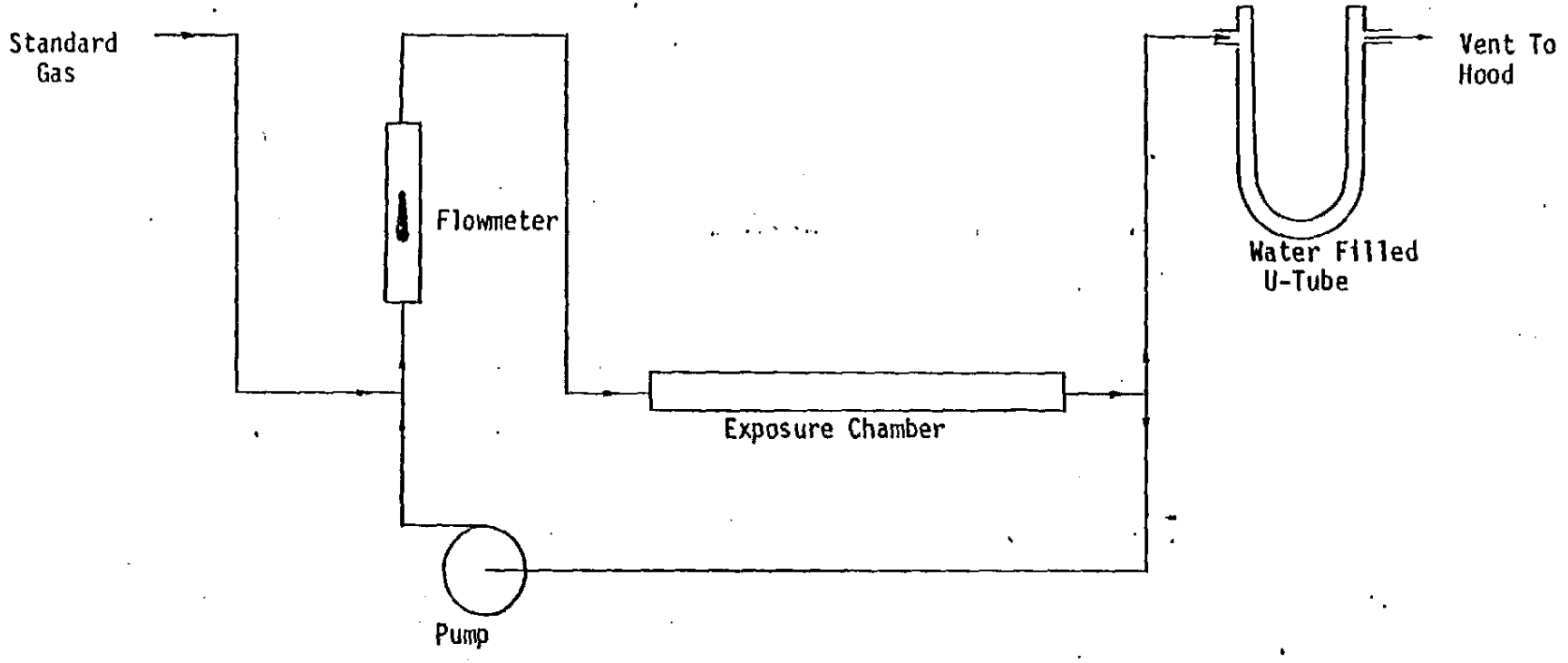

Figure 2

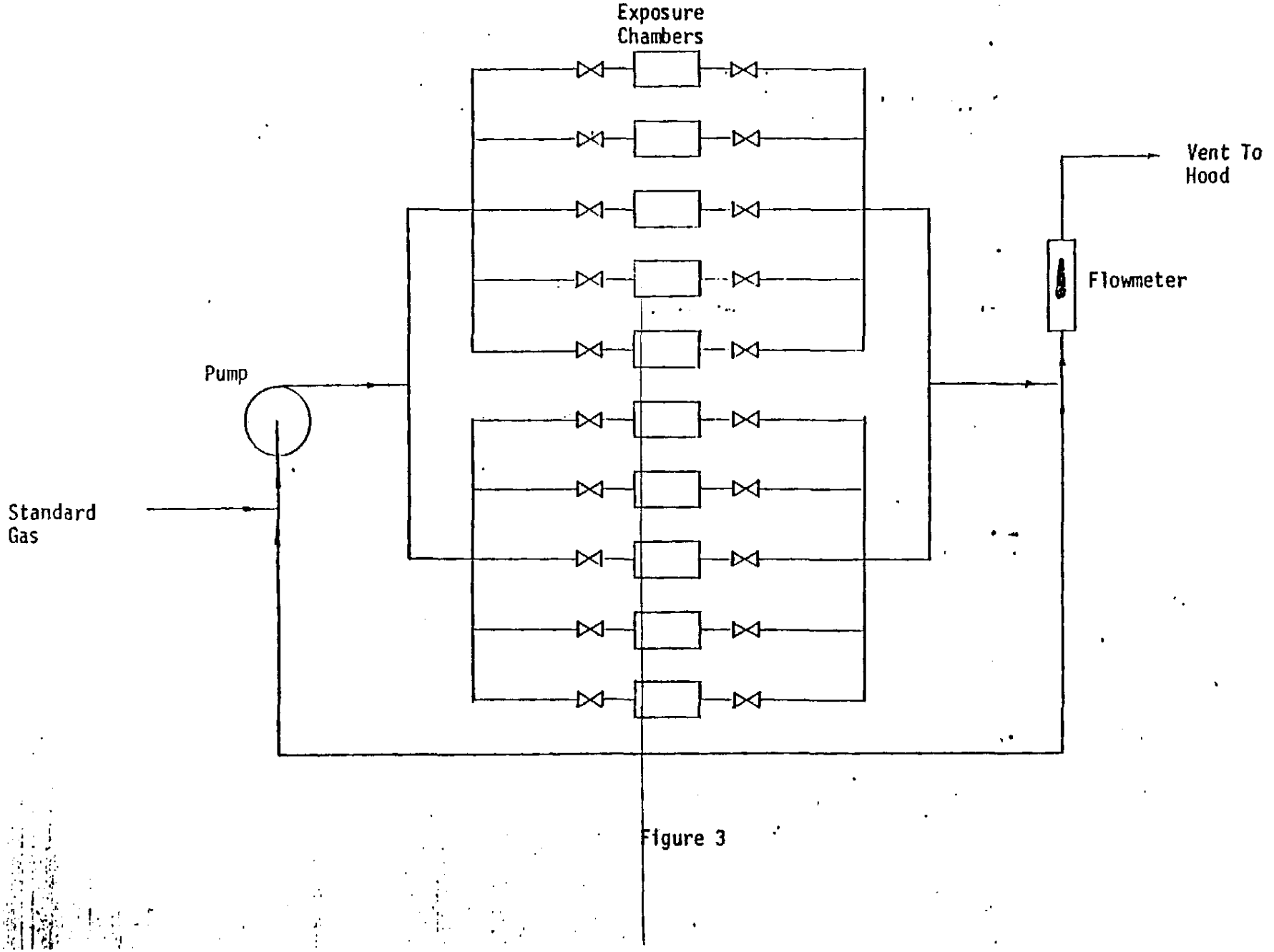

Figure 3

The calibration standards were of two types (1) Known weight standards, and (2) Known exposure standards prepared as outlined in Appendix I.

The tests conducted to compare the exposure chambers in Figures 2 and 3 were analyzed by the Perkin-Elmer Sigma 2 Gas Chromatograph, instead of Perkin-Elmer F-42, with calculations being performed by the Spectra-Physics SP-4100 Computing Integrator.

Analytical Results

1) Evaluation of Response of Perkin-Elmer F-42

The response of the Perkin-Elmer F-42 to vinyl chloride (VCM) and ethylene dichloride (EDC) was tested. These tests were necessary to determine maximum linear response to both VCM and EDC.

The test to determine response to EDC was conducted by analyzing a series of 28 samples whose weight ranged from 3 micrograms to 3,759 micrograms of EDC. The resulting data from analysis of these samples is presented in Table 1 and illustrated graphically in Figure 4. From this analysis it was determined that the response was linear up to at least 3,759 micrograms. This weight of EDC was found to be equivalent to an exposure of approximately 536 ppm-hours on the 3M OVM when analyzed using a calibration standard of 11.8 ppm-hours EDC. The test to determine response to VCM was conducted by analyzing a series of 28 samples whose weight ranged from 2.66 micrograms to 2,880 micrograms of VCM. The resulting data from analysis of these samples is presented in Table 2 and illustrated graphically in Figure 5. From this analysis it was determined that the response was linear up to 805 micrograms. This weight of VCM was found to be equivalent to an exposure of approximately 210 ppm-hours on the 3 M OVM when analyzed using a calibration standard of 13.44 ppm hours VCM.

2) Saturation of EDC and VCM on the 3M OVM

Gas spiking of the 3M OVM was conducted to determine the maximum weight of EDC and VCM which could be adsorbed on the 3M OVM badges. This was accomplished by removing the porous membrane, placing a filter paper in the badge, placing the hermetic seal on the badge, and injecting known weights of the compound of concern through the port on the hermetic seal. The port was then sealed and the badges allowed to sit overnight at ambient conditions. The following day the carbon pads were removed and placed in 1/4 ounce vials where 2 cc of DMA was added for desorbtion. Analysis was then performed to determine the amount of the compound which had been adsorbed.

To determine the saturation level of EDC on the 3M OVM a gas spike of 48.8 milligrams EDC was performed. When analyzed, using a calibration standard of 12.5 milligrams EDC injected in a 1/4 ounce vial containing an unexposed carbon pad from a 3M OVM, it was found that 45.16 milligrams of EDC had been adsorbed on the carbon pad. This level of EDC corresponds to an exposure on the 3M OVM of 6958 ppm-hours EDC. To determine the saturation level of VCM on the 3M OVM gas spikes of 19 and 11.85 milligrams VCM were performed. When analyzed, using a calibration standard of 10.3 milligrams VCM injected in a 1/4 ounce vial containing an unexposed carbon pad from a 3M OVM, it was found that 10 milligrams of VCM had been adsorbed for the 19 milligrams spike and 9.85 milligrams VCM had been adsorbed for the 11.85 milligram spike. Therefore, a conservative VCM saturation level for the 3M OVM would be 9.85 milligrams VCM corresponding to an exposure of 2570 ppm-hours on the 3M OVM.

3) Minimum Detectable EDC and VCM with the 3M OVM

The minimum detectable EDC and VCM are dependent upon the temperature of the Perkin-Elmer F-42 Carrousel and the minimum area as programmed into the SP-4000. Using carrousel temperature of 90°C and a minimum area of 200

TABLE 1

EDC Weight (micrograms)	EDC Area
3.000	447
6.200	727
12.90	1518
24.70	2954
37.50	4404
49.54	6480
61.75	7956
107.0	11365
96.60	11711
124.3	15919
145.0	18082
165.7	21832
189.8	25419
393.5	55334
586.7	81668
762.8	110065
983.6	143747
1232.	177843
1443.	208262
1670.	248512
1922.	285989
2133.	308343
2364.	345573
2613.	376931
2841.	421410
3058.	464166
3472.	509517
3759.	571349

GRAPH OF EDC AREA/100 VS MICROGRAMS EDC

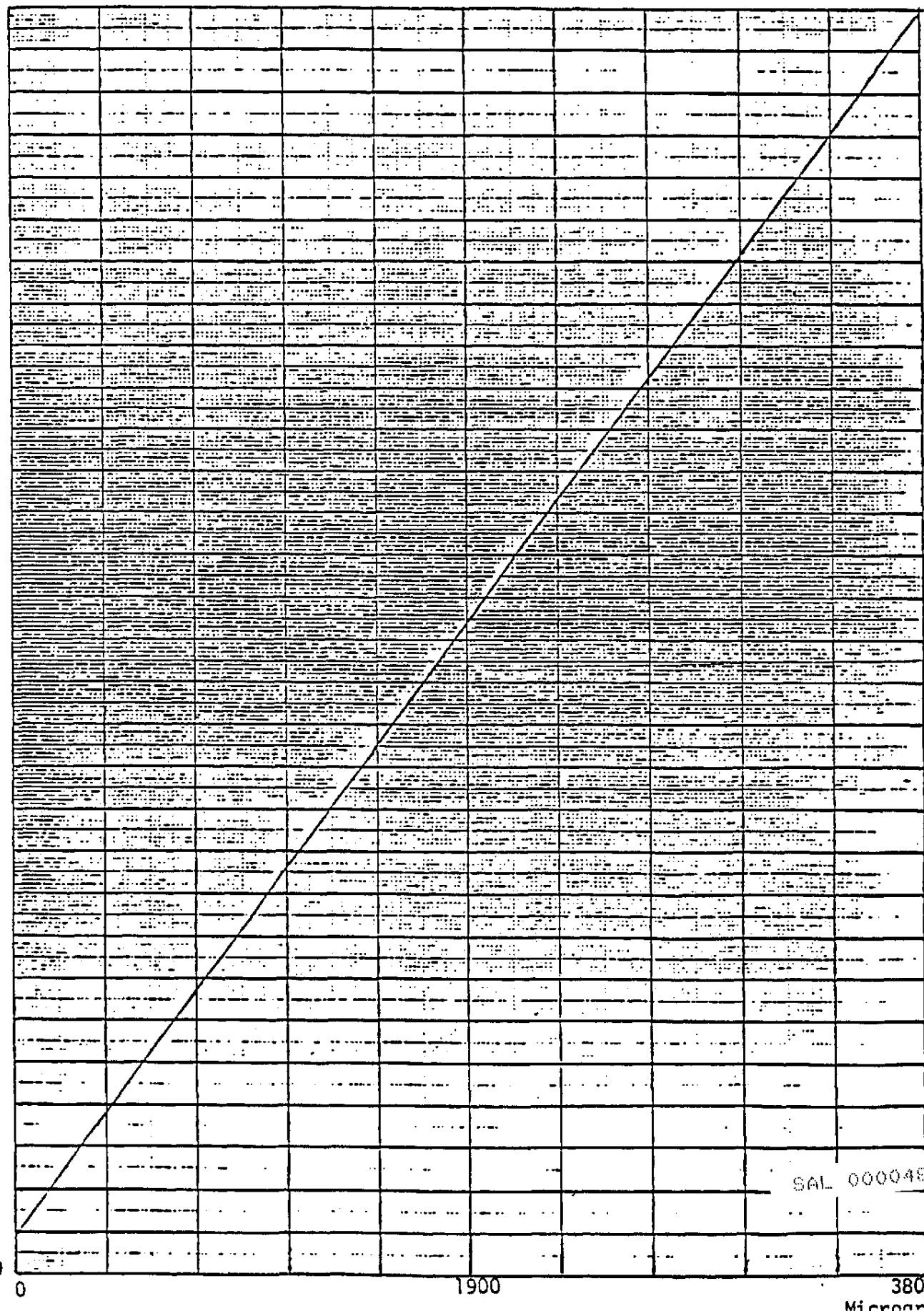

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Figure 4

TABLE 2

VCM Weight (micrograms)	VCM Area
2.663	8550
4.734	16584
9.400	31457
18.00	66191
27.00	100614
36.20	129946
45.60	165871
56.50	211490
80.60	273550
103.2	382470
123.0	492423
148.4	577713
175.3	683671
188.0	729969
337.9	1225737
476.4	1853963
651.7	2470736
769.0	2835277
972.6	3555607
1163.	3975713
1459.	4385750
1678.	4686329
1859.	4870945
2072.	4989011
2283.	5150592
2460.	5196248
2680.	5343772
2880.	5483287

GRAPH OF VCM AREA/1000 VS MICROGRAMS VCM

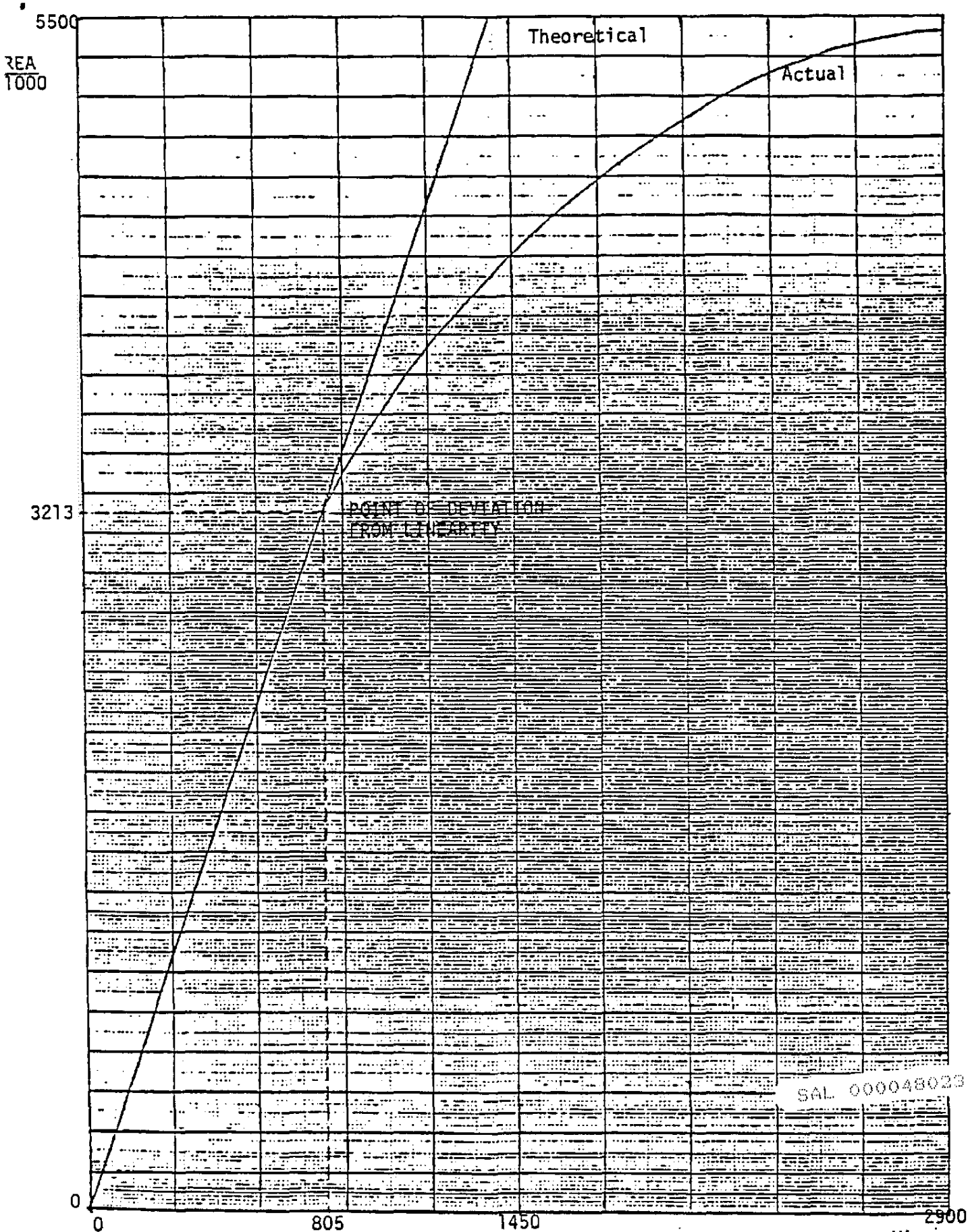


Figure 5

the minimum detectable level of EDC and VCM were determined by extrapolating from data used in section 1, Evaluation of Response of Perkin-Elmer F-42.

For EDC the minimum detectable level was found to be 1.3 micrograms or 0.19 ppm-hours. For VCM the minimum detectable level was found to be 62 nanograms or 0.016 ppm-hours.

4) Consistency Test of Exposure Chamber in Figure 2

The exposure system in Figure 2 allowed the standard gas to flow over the badges in a series arrangement. In order to determine if sufficient VCM was removed by preceeding badges to cause low readings on the following badges twelve 3M OVM badges were exposed to 4.99 ppm VCM in a nitrogen mattrix for 6 hours. The position of each badge was recorded relative to where the gas entered the chamber. The samples were then numbered from 1 to 12 with sample number 1 being closest to the inlet and sample number 12 being farthest from the inlet. Analysis was performed in the manner described earlier. The results of analysis are graphically depicted in Figure 6. Because of the decrease in VCM adsorbed by the badges as their relative position from the inlet increased it was decided to construct a new exposure system which exposed the badges in parallel as in Figure 3.

5) Irreversible Weight of EDC on the 3M OVM

The tests to determine irreversible weight of EDC were necessary in order to evaluate the effectiveness of the collection medium of the 3M OVM to adsorb EDC and then to retain that EDC. The procedure used for these tests was to gas spike badges with EDC, allow them to adsorb overnight at ambient conditions, challenge them to dry and humidified nitrogen, and then analyze them to determine how much EDC remained on the badge. The exposure chamber used to challenge the badges was the one depicted in Figure 1. The flow rate of the challenge environment was maintained at 700 milliliters per minute as read on the Gilmont flowmeter. Tests were conducted with a 20 microliter spike and a 5 microliter spike challenged to dry and/or humidified nitrogen for time intervals varying from 0 to 480 minutes. The analysis data is listed in Table 3.

6) Irreversible Weight of VCM on the 3M OVM

In order to determine a maximum exposure limit for the 3M OVM it is necessary to quantify the VCM that will remain on the collection medium (under zero concentration levels) once it has been adsorbed. The test required to accomplish this consisted of the following steps: (1) expose twelve 3M OVM badges to a known concentration level for a predetermined length of time (2) challenge these exposures to either dry or humidified nitrogen for periodic time intervals varying from 0 to 480 minutes, and (3) analyze resulting exposures to determine whether or not the level of VCM remaining on the badge after challenging had been reduced from that before challenging.

Initially twelve 3M monitors were exposed to 44.3 ppm VCM for 8 hours (354.4 ppm hour VCM). After challenging these badges and analyzing them it was clear that the irreversible weight of VCM had not been reached, therefore,

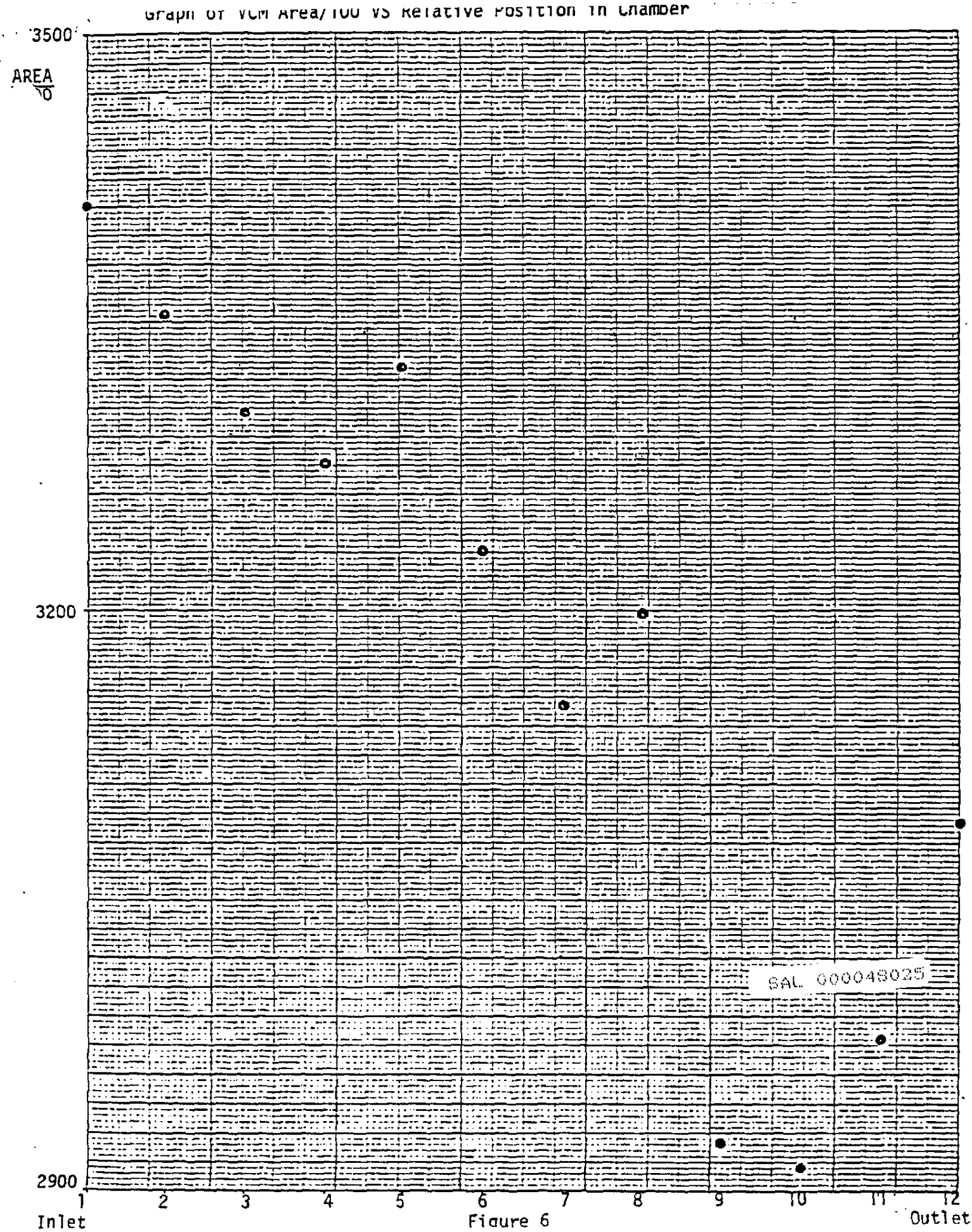

Graph of VCM Area/100 vs Relative Position in Chamber
3500
AREA
100
3200
2900
1
2
3
4
5
6
7
8
9
10
11
12
Inlet
Outlet
SAL 000048025

Figure 6

TABLE 3

Challenge Envrionment - Dry Nitrogen Time Challenged (minutes)	Spike - 20 microliters EDC Weight (milligrams)
0	22.59
38	22.87
60	22.51
120	24.52
240	23.18
480	22.29

Challenge Environment - Humidified Nitrogen Time Challenged (minutes)	Spike - 20 microliters EDC Weight (milligrams)
0	24.35
30	24.83
60	24.90
120	23.67
240	23.98
480	20.81

Challenge Environment - Dry Nitrogen Time Challenged (minutes)	Spike - 5 microliters EDC Weight (milligrams)
0	7.10
30	6.53
60	6.65
120	7.50
240	6.67
480	6.76

twelve additional monitors were exposed to 44.3 ppm VCM for 2.5 hours (110.75 ppm-hours) and challenged. Results of analysis of this second set of exposures indicated that the irreversible weight still had not been reached necessitating a third set of exposures. This set of twelve 3M badges was exposed to 44.3 ppm VCM for 1 hour (44.3 ppm-hour). When these exposures were analyzed it appeared that the level of VCM remaining had stabilized indicating that the irreversible weight for VCM had been reached.

The analytical data from analysis of these 3 sets of exposures was plotted on a graph of ppm-hours VCM versus hours challenged. The graph in Figure 7 is composed of the data accumulated for challenging with dry nitrogen and the graph in Figure 8 is for challenging with humidified nitrogen.

To negate the effect of the phenomenon demonstrated in section 4, consistency test of Exposure Chamber in Figure 2, the relative positions of each badge exposed was recorded. Then when the badges were challenged they were challenged in such an order as to cause ppm-hours VCM to increase with increasing challenge time. As seen in Figures 7 and 8 ppm-hours VCM actually decrease with increasing challenge time, therefore, the actual rate of decrease of ppm-hours VCM with increasing challenge time is actually slightly greater than that shown.

7) Irreversible Weight of VCM on the Protek G-BB

In order to compare the results of VCM retentivity of the 3M OVM to other passive dosimeters testing similar to that done in the previous section was conducted using the Protek G-BB and the Real Minimonitor. In this section the procedure and analytical results for testing the VCM retentivity of the Protek G-BB will be presented.

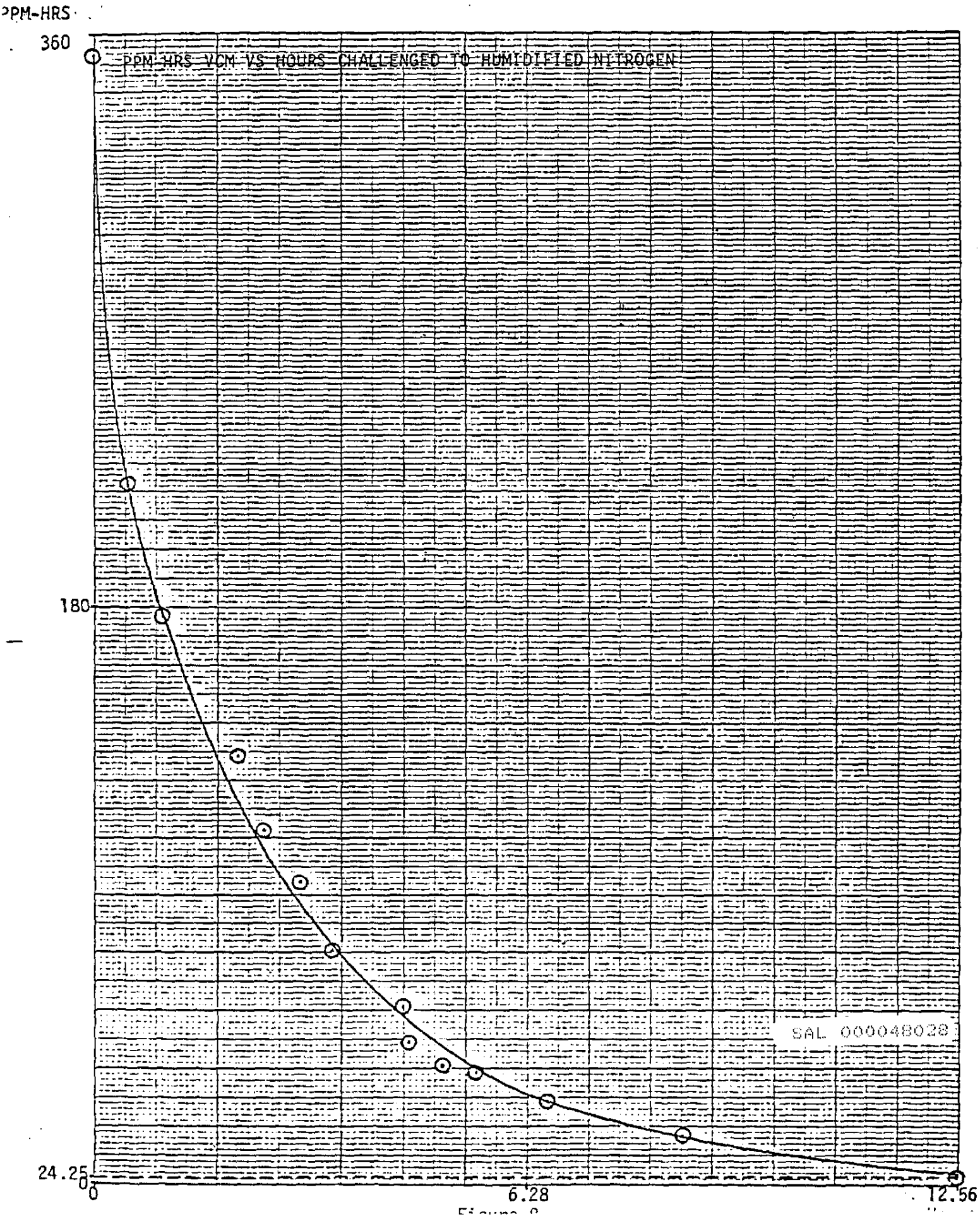
Two sets of exposures followed by challenging with dry and humidified nitrogen were conducted. The first set was exposed to 44.3 ppm VCM for 8 hours (354.4 ppm-hours VCM). The data from analysis of this set is shown in Table 4. The second set was exposed to 44.3 ppm VCM for 1 hour (44.3 ppm-hours VCM). The data from analysis of this test is shown in Table 5.

8) Irreversible Weight of VCM on the Real Minimonitor

Only one set of VCM exposure retentivity on the Real Minimonitor was conducted. In this test 5 monitors were exposed to 44.3 ppm VCM for 1 hour followed by challenging 4 monitors to humidified nitrogen for periodic time periods from 0 to 480 minutes. The fifth monitor was an unchallenged control. The results of this test are shown in Table 6.

9) Comparison of Exposure Systems in Figure 2 and 3

Tests were conducted to determine the best exposure system to use for exposure of monitors. These tests were conducted by seting up the exposure system as shown in Figure 2 and Figure 3, connecting a gas standrd of 25 ppm VCM to the inlet, and allowing gas to flow while the gas entering and exiting the plexiglas exposure box was sampled. These samples were analyzed as described earlier using a Perkin-Elmer Sigma 2 Gas Chromatograph and Spectra-Physics SP-4100 Computing Integrator. The flow rate from the standard gas


PPM-HRS
360
PPM-HRS VCM VS HOURS CHALLENGED TO HUMIDIFIED NITROGEN
180
24.25
0
6.28
12.56
SAL 000048028

PPM-Hrs VCM VS Hrs Challenged to Dry Nitrogen Gas

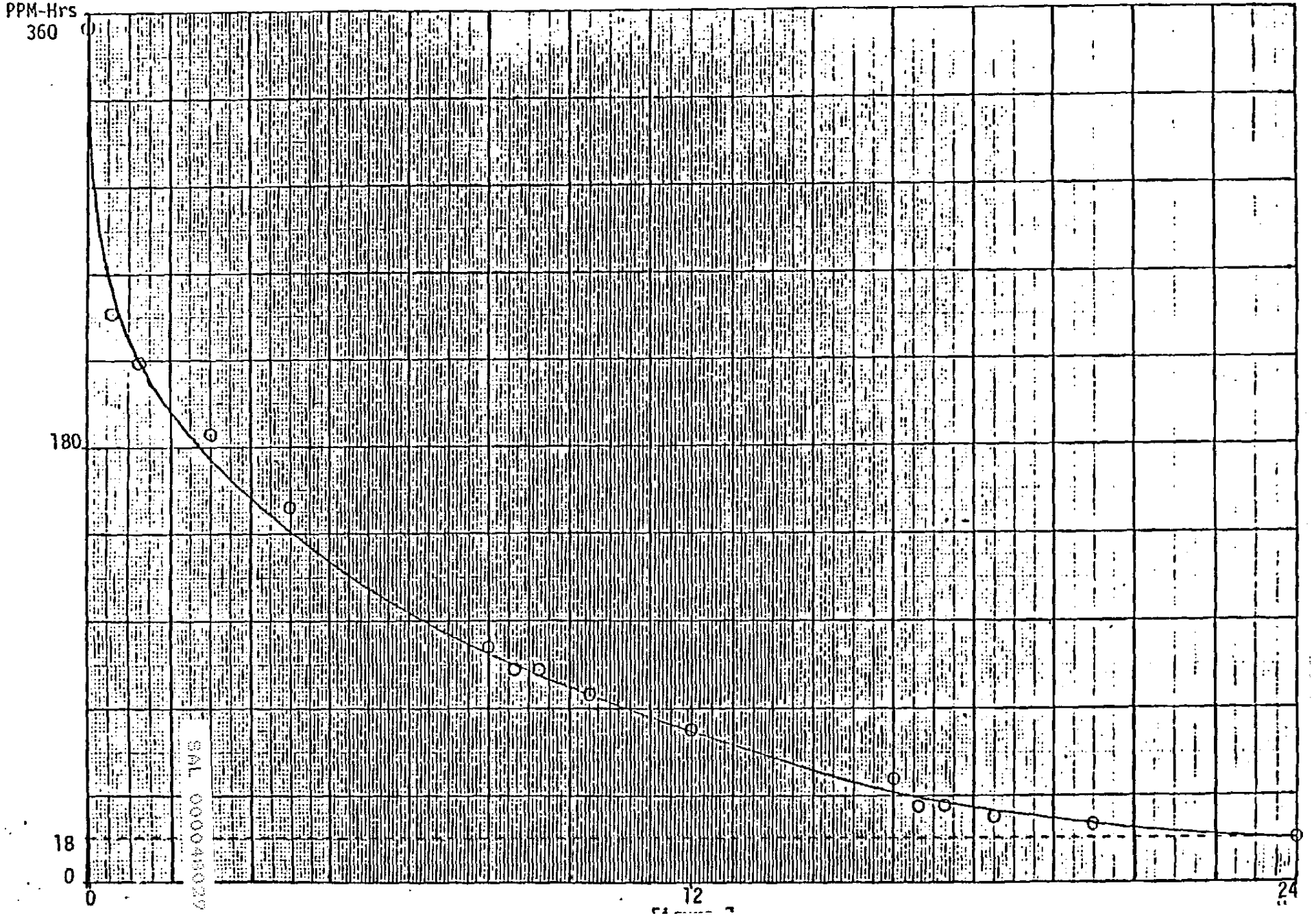


TABLE 4

Time Challenged(minutes)	Challenge Environment-Dry Nitrogen Total VCM Weight on Front and Back-Up Sections(micrograms)	Fraction of Total Weight Appearing on Back-Up Sectior
0	1764	0.359
30	1581	0.363
60	1501	0.369
120	1212	0.458
240	1366	0.482
480	1106	0.540

Time Challenged(minutes)	Challenge Environment-Humidified Nitrogen Total VCM Weight on Front and Back-Up Sections (micrograms)	Fraction of Total Weight Appearing on Back-Up Sectior
0	1764	0.359
30	1340	0.451
60	1155	0.543
120	1283	0.654
240	679	0.583
480	253	0.595

TABLE 5

Challenge Environment-Dry Nitrogen

Time Challenged(minutes)	Total VCM Weight on Front and Back-Up Sections(micrograms)	Fraction of Total Weight Appearing on Back-Up Section
0	249.1	0.0556
30	232.8	0.104
60	206.1	0.141
120	176.7	0.214
240	163.0	0.339
480	169.2	0.452

Challenge Environment-Humidified Nitrogen

Time Challenged(minutes)	Total VCM Weight on Front and Back-Up Sections (micrograms)	Fraction of Total Weight Appearing on Back-Up Section
0	249.1	0.0556
30	213.7	0.206
60	180.8	0.344
141	140.9	0.538
240	133.7	0.595
527	73.4	0.602

TABLE 6

Time Challenged (minutes)	VCM Weight (micrograms)
0	209.9
120	204.3
240	157.4
360	208.9
480	186.5

cylinder and exiting the system were maintained at 3 liters per minute both with the pump recycling and with the pump off. When the pump was used for recycling the flow rate through the recycle loop was 43.7 liters per minute. During testing there were no badges in the exposure chambers. The results of this test are shown in Table 7. The elapsed time is measured relative to the time at which flow was initiated. The fraction of VCM exiting was calculated by dividing the VCM area of the exit sample by the VCM area of the inlet sample.

TABLE 7

Elapsed Time (minutes)	With Pump Off Fraction of VCM Exiting System in Fig. 2	System in Fig. 3
1	0.674	0.871
11	0.632	0.813
21	0.894	0.850

Elapsed Time (minutes)	With Pump On Fraction of VCM Exiting System in Fig. 2	System in Fig. 3
1	0.658	0.906
11	0.494	0.912
21	0.923	0.856

Discussion

In various sections of this report reference is made to an equivalent ppm-hour exposure for the 3M OVM. It should be understood that this equivalent exposure was determined in one of two ways. Method number one for determining an equivalent exposure was to prepare a calibration standard to be analyzed with a sample containing the desired weight injected on a blank carbon pad from a 3M OVM. The calibration standard used for such an analysis was prepared as outlined in Appendix I. Use of this type of calibration standard along with injecting the desired weight onto a blank carbon pad eliminated the need for further tests and calculations to determine sampling rate and desorbtion efficiency. Method number two for determining an equivalent exposure was to extrapolate for the desired weight from data already determined by method number one.

In comparing the exposure chambers of Figures 2 and 3, in section 9 of Analytical Results, it was found that only a fractional amount of the VCM entering the plexiglas chambers was actually exiting these chambers. The reason for this phenomenon is that VCM is adsorbed by and gradually permeates through plexiglas. So what happens in the plexiglas chambers is that prior to initiating flow of a VCM containing atmosphere there is no VCM on the walls of the plexiglas but as flow is initiated and progresses eventua-ly there will be an equilibrium set up between the VCM atmosphere in the chamber, VCM adsorbtion by the plexiglas , and VCM permeation through the plexiglas to the relatively low VCM concentration on the exterior of the chambers.

In section 4 of Analytical Results it was shown that the exposure chamber in Figure 2 caused inconsistent readings of VCM adsorbed on 3M monitors. At the time that this test was performed it was felt that the series exposure of the badges caused these inconsistencies but now that the idea of plexiglass adsorbtion of and permeation by VCM has been shown it is felt that these inconsistencies could be the result of one or both of these factors. Therefore, in order to eliminate both of these factors it is felt that the plexiglas chambers used in the exposure system of Figure 3 be reconstructed of a material which will not affect any of the organic vapors of interst.

Because of the revelations of this study as outlined in this report there are two possible sources of error that could cause erroneous computations of equivalent exposures. The first source of error addressed in this report is that introduced by exposing calibration standards in plexiglas chambers resulting in the ppm-hour VCM per weight of VCM adsorbed by 3M OVM being greater than what is shou have actually been. What this means in terms of equivalent exposures given for both experimental values and for personnel exposures is that the actual exposure, in ppm-hours, is lower than that given in this report and on personnel exposure records. The second source of error addressed in this report is that introduced by the fact that VCM is readily desorbed from exposed badges by both dry and humidified nitrogen. What this means in terms of personnel exposures is that if a reading from an analyzed 3M OVM used for personnel sampling in the field is greater than the irreversible weight of VCM on the 3M OVM then all that can be said of the actual time weighted average exposure is that it was greater than or equal to the calculated exposure. It is now possible to eliminate the first

source of error by reconstructing the exposure chambers of a material which will not affect any of the organic vapors of interest. The second source of error can only be corrected by use of a monitor more suitable for VCM exposure.

It should be noted here that the only effect that the two sources of error introduced above have on the data of this report is that the equivalent VCM exposure values should actually have been lower. These sources of error will not affect any of the rates of change or slopes shown. What this means for the irreversible weights given for VCM in this report is that these values should be noticeably lowered.

Summary

During the course of this study several enlightening results were achieved.

It was determined that the 3M OVM is apparently a very effective method for monitoring for ethylene dichloride (EDC). The saturation level of EDC on the collection medium was high and the retentivity of EDC was also very good indicating that EDC was strongly adsorbed on this charcoal collection medium. The EDC was also readily desorbed for analysis by 2 cc DMA.

The effectiveness of the 3M OVM to monitor for vinyl chloride (VCM) is much less encouraging. Although the saturation level of VCM was less than that of EDC it was still within tolerable limits. The retentivity of the 3M OVM for VCM was very poor. The study showed that VCM was readily desorbed from the charcoal collection medium by both dry and humidified nitrogen indicating that VCM was weakly adsorbed. As an example of this phenomenon consider what would happen should the monitor be exposed for a short period of time, say 1½ hours, to a high level of VCM, such as 19.2 ppm. Now during the remainder of this 8 hour shift let the badge be exposed to typical atmospheric conditions in the Gulf Coast region of between 70 to 80% relative humidity. At the end of the shift we can expect to see enough VCM to give us an eight hour time weighted average of 0.303 ppm VCM while the actual 8 hour time weighted average would be 3.6 ppm VCM resulting in a 91.6% difference. This example does consider worst case conditions but it is a common occurrence for the relative humidity in the Gulf Coast region to reach a level of 70% and higher.

In testing the Protek-G-BB for VCM retentivity it was found that the challenging with both dry and humidified nitrogen of previously exposed monitors forced VCM to both migrate to the back-up section and be stripped from the badge entirely indicating that the adsorbtion medium used in the Protek G-BB was no better at adsorbing VCM than that in the 3M OVM. The Real Minimonitor showed better VCM retentivity than both the Protek G-BB and the 3M OVM. The reason the Real Minimonitor had better VCM retentivity could be the result of one or both of two factors: (1) the Minimonitor uses permeation instead of diffusion as the mode of transport across the barrier membrane and (2) the Minimonitor contained 1.35 grams of activated charcoal while the 3M OVM had only 200 milligrams and the Protek G-BB had 300 milligrams in the front section and 300 milligrams in the back-up section. Neither the Protek G-BB nor the Real Minimonitor was tested with organic vapors other than VCM, therefore, a complete comparison cannot be achieved without further testing.

In conclusion it should be emphasized that while the 3M OVM appears to provide an excellent method for monitoring for heavy organics, such as ethylene dichloride, it does not appear to be a reliable method for monitoring for lighter organics, such as VCM. It should also be noted that results from a comparative field study conducted by A. J. Hart, Appendix V, shows that the 3M OVM correlates well with the charcoal tube and other passive dosimeters indicating that the 3M OVM and the charcoal tube dosimetry behave in similar manner in field conditions.

APPENDIX I

Preparation of Calibration Standards for use in Analysis of Personnel Exposures

Step 1 Place fresh 3M Organic Vapor Monitor(s) in exposure chamber and seal chamber with vinyl electrical tape.

Step 2 Initiate flow of gas from a certified composition standard gas cylinder making sure to record time. Turn recirculation pump on immediately after initiating flow. Maintain flow out of cylinder at 3 standard liters per minute as read by a Gilmont No. 13 flowmeter.

Step 3 When sufficient time has elapsed to give a representative exposure to the calibration standard terminate flow by turning pump off and then shutting valve from cylinder. Make sure to record the time at which flow was terminated.

Step 4 Open exposure chamber by removing tape. Remove 3M monitor(s) from chamber.

Step 5 Immediately after removing 3M monitor(s) from chamber take the charcoal pad out of the monitor(s), using tweezers, and place it in a ¼ ounce vial and cap with rubber septum and crimp type cap.

Step 6 Store vials in freezer at -20°C until time for analysis.

APPENDIX II

rocedure for analysis of Personnel Dosimeter Samples
(Revised from Interoffice Communication of February 27, 1978 from H. L. Hackett to P. L. Fetzer)

Step 1
a) Remove samples and the calibration standards from freezer and allow them to warm enough to make the septums soft and pliable.
b) Turn on Oil Bath and check the Oil level.
c) Check pressure on H_2 cylinder south of lab.
d) Make sure nitrogen cooling for the column oven is on.
e) Add exactly 2 cc of NN-dimethylacetamide to each vial.

Step 2
a) Prepare a Run Sheet. An example is shown in Appendix III.
b) Place the samples and calibration standards in the PE/F-42 carrousel according to the order they are listed on the run sheet.
c) Check head-space parameter settings. Parameters are shown in Appendix IV.
d) Make sure turn table is rotated to its proper initial position which is the position just before the first calibration sample.
e) Check that the proper "status lights are on" on the oven program module and on the head-space sample programmer module.
f) IMPORTANT-Make sure proper patch panel connections are made to route GC signal to the SP-4000.

Step 3
Enter the run program into the SP-4000 as detailed in the following steps. If The head-space file is already set up it will usually be necessary only to make the entries or changes marked by an asterisk. Except for the items mentioned in the following steps, default parameters will usually be satisfactory for handling head-space samples.

Step 4
a)* Attach head-space file to channel desired. The file no. used will normally be #23 and the channel #6.
b)* Check that proper output device (OD) is used.
c)* Clear previous data in sample file by entering FSO in file center. Re-enter FS 5.
d) Enter FD1

Step 5
Roll up to component file preamble and
a)* Enter MN 5
b)* Enter CA 0
c)* Enter FP 2

Step 6
Roll up to component data file
a) Enter RT XXXX (Retention time of peak)
b) Enter NM XXXXXXXXX (Name of component, maximum of 9 spaces)
c) Enter CC XXXX (concentration of component in standard gas used for preparation of calibration standards.)
d) Enter the above three parameters (RT first) for each of the other components in the standard. When entering the parameters for EDC, enter DP = 1 (DP = 0 for all other components). This says to use the KF value for EDC on all unidentified components.

Step 7
a)* Roll down to file center and enter SF (sample file). The display will show SN 001. The data entered here applies to the first analytical sample to be run as shown on the prepared run sheet.

b)* Enter NM XXX.....X (maximum no. of characters and spaces is 27 when FS = 5. The name ofthe sample should contain: (1) name of the person sampled (not over 14 letters), (2) date person was sampled, (3) badge no. used. The following format is strongly recommended:

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c)* XF XXXX (this is the average time of exposure used in preparing the calibration standards as calculated on the prepared run sheet. XF is a multiplication factor and is the same for all analytical samples in a calibration/analysis sample group.

d)* SA XXXX (amount of time sampled by the dosimeter as calculated on the run sheet.) SA is a division factor.

e)* Roll up to SN 002 and enter the NM, XF, and SA values for the second sample. Enter these same parameters for each of the other dosimeter samples.

Step 8 Roll down into file center

a)* Enter RN 1

b)* Enter MS-1 (if running more than one sample besides the calibration run or runs)

c)* Enter IX 1

d)* Enter SN 0

e) Enter PW 005

f) Enter PP 004

g) Enter FS 5

h) Enter MA 0200

i) Enter PT 15

Step 9 Go to the function file and check the following if necessary:

a) Enter TP 1

b) Enter AP 0

c) Enter FX 1

d) Enter XD 1

e) Enter TB 0

f) Enter zero for all other parameters in the function file.

Step 10 Roll down to time file and -

a) Enter RT0001 T40

b) Enter RT0850 II1

c) Enter RT1250 T41

Step 11 Exit file and -

a)* Enter ST, check to see that a Procedure (usually PR 14) which was used for a previous set of head-space samples is not in memory. If it is, erase it by entering SE, then entering DE 14 PR. (Make sure you are not erasing a procedure used for some other purpose). Exit SE by entering XX.

b)* Enter the following "canned" procedure which controls when the calibration light is to be on during the set of samples.

Enter CP n a b c d e (use a space following the P and each entry thereafter)

where CP = CP is the code for automatic cal/anal sequencing
n = No. given the procedure (usually 14)
a = File no. (usually 23)
b = No. of calibration runs (usually 2)
c = No. of analysis samples (before re-calibration)
d = No. of runs per analysis sample (usually 1)
d = No. of cal/anal cycles (one cycle consists of the calibration samples and the analytical samples before a new calibration)

Step 11 c)* Enter PX n (n = no. of the procedure used above)

Step 12 When the previous steps have been completed and the samples have equilibrated at 90°C for at least 60 minutes, Depress the "Start" button on the head-space programmer module.

Step 13 Check to see that a "stop peg" has been inserted at the position of the last sample and that none is present at any of the previous sample positions.

Step 14 If the oil level gets too low a safety switch cuts power to the heaters but not to the pump. It may not be readily apparent that this has happened unless the temperature shown by the thermometer is checked.

APPENDIX III

ANALYSIS DATE 7/20/80

RUN SHEET FOR HEAD - SPACE SAMPLES

ANALYST SCR

Sample No.	Person Sampled	Date Person Sampled	Badge No. Used			Sample Vol (min.)	XF Average Time of Calibrations	SA (Time of Analysis samples)
Std. 153	Calibration	—	0982			30	—	—
Std. 154	Calibration	—	0983			30	—	—
1	W. Jones	7/5/80	5683			480	30	480
2	B. Smith	7/7/80	6538			500	30	520
3	A. Hebert	7/8/80	8365			400	30	400
4	T. Doe	7/8/80	6385			450	30	450
5	C. Landry	7/12/80	4530			480	30	480
6	B. Simms	7/12/80	0450			500	30	500
7	J. Guidry	7/12/80	6502			510	30	510
8	M. Pelican	7/13/80	8204			460	30	460
Std. 155	Calibration	—	1876			30	—	—
Std. 156	Calibration	—	1879			30	—	—
9	T. Chaisson	7/15/80	5643			480	30	480
10	B. Thomson	7/16/80	6345			480	30	480
11	S. LeBlanc	7/18/80	4365			510	30	510
12	H. Martin	7/18/80	3456			460	30	460
PROCEDURE ENTRY: CP			14	3 2 8 1 2				

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APPENDIX IV

PE/F-42 Parameters for Personnel Dosimeter Samples

		Programmed Runs	Isothermal Runs
Bath Temperature		90°C	90°C
Needle Temperature		150°C	150°C
Temperature Control Module			
Oven		65°C	65°C
Injector		150°C	150°C
Detector		170°C	170°C
Multi-Temperature Program Module		ON	OFF
Times	Initial	4	(With
	Middle	0	switch
	Final	8	on back
Rates	Initial - Mid	8	of
	Mid-Final	8	module)
	Cool	1	
Temperature	Middle	130°C	
	Final	130°C	
FID Amplifier Module			
Range		1	1
Attenuation		2	2
Polarity		+	+
Head Space Programmer			
F		BF	BF
Anal And Backflush Times		X1	X.01
Times Inj		5	5
A		10	17
BF		10	18
S		9	3
Gases FID Air		3.6	3.6
FID A Hydrogen		2.2	2.2
FID B Hydrogen		2.15	2.15
Carrier Top		1.8	1.8
Middle		0	0
Bottom		1.65	1.65

APPENDIX V

Data from Comparative Field Study of Passive Dosimeters with Charcoal Tubes Conducted by A. J. Hart:

	Vinyl Chloride (ppm)					1,2 Dichloroethane (ppm)				
Sample Number	Charcoal Tube	3M	duPont	Gas-badge	Mini-monitor	Charcoal Tube	3M	duPont	Gas-badge	Mini-monit
1	.1	.1		0	.1	.2	1.0		.3	1.5
2	0	0		.3	.1	.1	0		.2	.3
3	.1	.1		0	.1	.1	.2		3.7	.4
4	.2	.1		0	.1	.3	.3		.8	1.3
5	.1	.5		.4	2.2	0	.1		3.3	.1
6	.1	.1		.3	.1	.2	.2		3.4	2.0
7	0	0		.3	0	.1	.1		.9	1.3
8	0	0		0	.1	.1	0		0	.2
9	.1	.1		0	.1	.1	.2		1.0	.3
10	.2	0		0	.1	.2	0		5.3	.4
11	0	0		0	0	.2	.1		0	.3
12	0	0		.2	0	0	.1		.2	.1
13	0	1.1		.3	.9	.2	.1		3.9	.1
14	.1	.1		.9	.1	0	.4		0	.2
15	0	0		.3	0	.2	.2		4.3	.1
16	0	0		.3	0	.3	.2		.4	.3
17	0	0		0	0	.6	.4		4.0	.1
18	.5	.5		.4	.3	.4	0		3.0	.3
19	0	.1		.3	.1	.1	.1		2.3	.2
20	0	0		.2	0	.1	.2		.7	.2
21	.1	.2		0	.3	.2	.2		0	.3
22	1.2	.5		2.0	2.2	.2	.2		.3	.3
23	0	0		0	0	.1	.3		6.0	.1
24	0	.1		.5	.2	0	0		4.0	12.1
25	.1	.1		0	.1	.2	.2		.1	.2
26	.1	.1		.2	.1	.3	.4		.2	.2
27	.4	.1		0	.5	1.7	3.5		2.8	8.9
28	.1	0		0	.1	.2	.2		.2	.2
29	0	0		2.6	0	0	.1		3.4	0
30	.1	.1		1.0	.1	.5	.2		1.1	.3
31	.1	.2		0	.1	.4	.8		.7	.6
32	.3	1.2		0	.4	6.9	5.7		0	1.3
33	.3	.1		0	1.3	1.7	2.9		.2	1.2
34	0	0		0	.2	1.3	.9		.7	.4
35	.5	.1		.2	.3	.3	.9		.9	1.4
36	.1	.3		0		.4	0		.4	
37	.1	.1		0	.1	.2	.2		5.2	.1
38	0	0		0	.1	1.3	.4		.3	.4
39	0	0		0	.1	0	.2		.3	.1
40	.2	.7		2.7	.3	.1	.2		.2	.1
41	0	.1		.2	.1	.1	.3		.5	.3
42	.1	.1	.1	0	0	0	.3	.3	.4	.2
43	1.9	.9		.1	1.0	.5	.7		1.1	1.0
44	.1	0	.1	0	.1	0	0	.3	0	.1
45	0	2.1	.5	2.4	2.3	0	3.8	4.7	21.4	10.1
46	.2	.1	.2	.3	0	1.5	.5	.6	.4	.4
47	.1	.1	.1	.1	.1	.3	2.6	4.6	.3	2.5
48	.1	0		0		.6	.7		0	
49	.2	.1		.1		.6	.5		.3	
50	0	0	0	.1	.1	.7	.7	.9	.8	.8
51	.2	0	0	0	0	.3	.2	.3	.1	.1
52	.1	0	.1	0	.3	0	3.0	7.9	4.2	5.5
53	.1	.1	.2	.2	.3	.8	.9	1.0	.8	1.5
54	0	0	0	0		.3	.4	.5	.3	
55	.1	0	.1	0	.1	8.5	.5	1.0	.4	.5
56	0	0	0	.1	0	.5	.3	.4	.2	.2
57	0	0	0	.1		.2	.2	.3	0	
58	0	0	0	3.7	.1	.1	.4	.6	.3	0
59	0	0	0	0	0	.1	.2	.2	.3	.2
60	0	0	0	.4	0	.1	.4	.5	.6	.6
61	.1	.1	.1	0	.1	.1	.7	.9	.4	.9
62	.1	0		.2		0	.2		.2	

(Appendix V Cont'd)

Sample Number	Vinyl Chloride (ppm)					1,2 Dichloroethane (ppm)				
	Charcoal Tube	3M	duPont	Gas-badge	Mini-monitor	Charcoal Tube	3M	duPont	Gas-badge	Mini-moni
63	.1	0	.1	0	.1	0	.2	.2	7.8	14.6
64	.1	0	.1	0	0	.8	.6	.7	.4	.4
65	.1	0	0	0	.1	4.7	1.5	2.3	.9	1.3
66	0	0	0	0	0	.2	.2	.3	.2	.2
67	.1	0		0	.1	.6	.8		1.2	.4
68	.1	.1	.1	.3	.1	.8	.3	.4	.2	.2
69	.1	0	.1	0	0	.3	.2	.3	.2	.4
70	0	0	0	0	0	.3	.1	.2	.1	.1
71	.1	0	0	0	.3	.5	.5	.7	.4	1.1
72	.1	0	0	.6	0	1.1	.2	.7	0	.2
73	.2	0	.1			.9	.3	.2		.1
74	0	0	0		.2	.3	8.5	10.4		2.7
75	0	0	0		.1	.6	.2	.2		.2
76	0	0	0		0	.5	.5	.5		.3
77	.1	.1	.1		.1	.3	.3	.4		.1
78	0	0	0		0	.6	.3	.2		.1
79	.8	.2	.1			.5	.2	.2		
80	0	0	0			.3	.3	.4		
81	0	0	0			.4	.1	.2		
82	.1	.1	.1			.1	.2	.1		
83	.1	.2	.1			.1	.2	.1		
84	.1	.1	.1			.1	.6	.4		