F. C. Allen

REPORT NO. 2970
FINAL REPORT
ON

AROCLOR IN GASES

Job No. 171-1089 File No. 141

March 15, 1954 H. B. Richards, Jr.

RESEARCH DEPARTMENT
Phosphate Division

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MONSANTO
CHEMICALS - PLASTICS

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FINAL REPORT ON

AROCLOR IN GASES

Job No. 171-1089
File No. 141

Research Department - Phosphate Division
Anniston, Alabama.

Work Started: May 8, 1953
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CONTENTS

		Page
I.	Introduction	1
II.	Summary	1
III.	References	2
IV.	Experimental Work	2
V.	Discussion	9
VI.	Conclusions	11
VII.	Recommendations	11

Appendix

Operating Procedure for Willson Products, Incorporated Chlorinated Hydrocarbon Sampling Apparatus 13

Data Sheet - Aroclor Vapor Detection 17

Sampling Positions 12

Diagrammatic Sketch 18

Photograph of Samples

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I. INTRODUCTION:

Dow Chemical Company is interested in using Aroclor as a plasticizer in manufacture of their Saran (vinylidine chloride) plastics, but is hesitant for fear that the Aroclor vapor might produce toxic affects upon the personnel in their plant.

At the request of the Sales Department this work was undertaken to determine the Aroclor vapor concentration in the Anniston plant under usual working conditions. This data would then be used in an attempt to convince Dow that the use of Aroclor in their plant would be safe.

An outgrowth of the problem was the determination of the Aroclor vapor present in a room which had been painted with a styrene latex paint using Aroclor as a plasticizer. This work is of interest to the Plastics Division, and was undertaken with this project since the same apparatus and analytical procedures are used as in the Aroclor vapor tests in the plant.

II. SUMMARY:

An Interim Report, No. 2892, covers the work up to July, 1953. This report supplements the Interim Report.

Air sampling was continued in the plant under all normal operating conditions. The chlorine concentration in the air varied greatly giving calculated Aroclor concentrations ranging from about 0.35 mg. per cu. meter up to 5 to 8 mg. per cu. meter. The filtering and drumming operations present the most severe exposure to the Aroclor vapors. No definite average daily exposure was determined.

A small room, 14' x 13' x 14', was painted on four walls and on the ceiling with Lustrex Latex Paint, Lot No. P-0938 containing Aroclor 1248. The room has only one door and a small 12" ventilating fan with no windows that can be open. Most of the time the room was closed, especially, during the early testing periods.

A hood, 2' x 3 1/2" x 7', was also painted on three walls. The exhaust fan on the hood was not operated during the test period. Two 1000 watt electric hot plates were turned on in the hood to heat the air during the sampling operation.

Sampling of the air in the room revealed that the Aroclor vapor concentration rapidly reached a maximum and then generally decreased to a relatively safe low level after three days. In the heated laboratory hood the concentration remained in the 1.0 - 2.0 mg. per cu. meter range over a period of about one month. In a heated room it appears that the concentration of Aroclor 1248 vapors may remain high for a long period of time.

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III. REFERENCES:

Report No. 2892, Interim Report on Aroclor in Gases, June 17, 1953, H. B. Richards, Jr.

IV. EXPERIMENTAL WORK:

The apparatus and operating procedure are described in the Appendix to Report No. 2892. This description is supplemented in this report by a photograph of the test apparatus, Willson Product's Incorporated, Chlorinated Hydrocarbon Sampling Apparatus. The sampling positions in the plant are the same as described in the Interim Report No. 2892.

Analytical Procedure: Anniston Test No. 20-25-41, Nephelometric determination of Cl^- ion.

Range:

5 - 120 micrograms in 20 ml. maximum volume.

Apparatus:

1. Fisher Electrophotometer with blue filter and 25 ml. sample tube.
2. 25 ml. glass stoppered volumetric flask or graduate.
3. 10 ml. measuring pipette and 10 ml. transfer pipette.

Solutions:

1. 3N Nitric Acid - 190 ml. CP conc. HNO_3 diluted to 1,000.
2. 0.3% Silver Nitrate - 3 grams dissolved in 1,000 ml. distilled water. Store in dark bottle out of light.
3. 1% Phenolphthalein. 1 gram solid in 100 ml. CP Methanol.

Procedure:

1. Pipette required volume of water solution into 25 ml. volumetric graduate. Adjust sample size to obtain 10 - 120 micrograms of Cl in less than 22 ml. For testing scrubber solutions of Aroclor vapor tests use 10 ml.

2. Add 1 drop Phenolphthalein and neutralize with 3 N nitric acid (or dilute caustic if sample is acid).

3. Dilute to 22 ml. with distilled water.

4. Add 1 ml. HNO_3 and 1 ml. $AgNO_3$ in this order.

5. Mix by inverting Only Once and set in complete darkness for 30 Min.

6. Balance Electrophotometer with distilled water in both tubes, using blue filter and 25 ml. cylindrical sample tubes.

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7. Transfer sample to the Electrophotometer tube quickly with minimum exposure to light and read density or "A" scale. Read quickly without prolonged exposure of sample to the beam of blue light.

8. Compare the scale reading with a calibration chart made from identical determinations of standard chloride solutions.

9. Make a blank run on all reagents and subtract from reading of step (8) or plot data of standard runs to include the chlorides of the blank, in which case the reading may be taken directly from the curve. However a new curve must be made when reagents change.

10. To calculate parts per million (ppm.) of chlorides, divide answer in micrograms by volume of sample.

$$\text{ppm.} = \frac{\text{Micrograms Cl}}{\text{Volume of Sample}}$$

$$\% = \frac{\text{Micrograms Cl}}{\text{Volume X 10,000}}$$

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TABLE I

AROCLOR 1242 VAPOR CONCENTRATION IN THE PLANT

Test No.	Position No.	Concentration, mg/cu.m. Chlorine	Aroclor	Remarks
10	5	0.14	0.33	Still vented on top.
29	4	1.5	3.6	Still in operation
30	5	1.5	3.6	
31	6-S	2.2	5.3	Press not operating and cold. 1242 in No. 2 B. T. at 110° C.
45	1	0.85	2.0	Both stills operating
46	2	0.34	0.80	on 1242
47	3	0.85	2.0	Filter press replaced with Sparkler leaf filter which was cold at time of test.

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TABLE II

AROCLOR 1254 VAPOR CONCENTRATION IN THE PLANT

Test No.	Position No.	Concentration, mg.cu. m. Chlorine	Concentration, mg.cu. m. Aroclor	Remarks
24	10	0.68	1.4	By steam recorder
25	5	0.41	0.76	Still operating on 1254
27	5	0.50	0.92	
32	6-S	1.45	2.68	
33	6-N	1.04	1.93	Filtering 1254 at 110-115° C.
34	8	0.75	1.39	
35	4	0.51	0.94	
36	4	0.37	0.68	
37	5	1.30	2.40	Pumping 1254 from receiver
38	5	1.98	3.66	to B. T.
39	10	0.40	0.80	By steam recorder
40	7	0.73	1.46	
42	6-N	2.4	4.5	Filtering 1254 at 110° C.
43	8	4.3	8.0	Drumming 1254 at 150° C.
44	8	1.3	2.4	

NOTE: The analytical procedure on samples 37-40 inclusive was carried 1/2 the normal sample size.

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TABLE III

AROCLOR 1260 VAPOR CONCENTRATION IN THE PLANT

Test No.	Position No.	Concentration, mg/cu. m. Chlorine	Aroclor	Remarks
11	5	0.32	0.53	Still in operation
13	4	0.55	0.92	Still open
14	5	0.52	0.87	
15	6-S	1.5	2.5	Filtering 1260 at 125° C.
16	6-N	1.2	2.0	
18	5	0.22	0.36	Still operating
19	4	0.91	1.5	Still open
20	4	0.75	1.2	Still charged and started
21	4	0.53	0.88	Still operating
28	8	2.8	4.6	Drumming 1260 at 150° C.
22	7	1.1	2.2	By operator's desk, assuming 50% Cl.

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TABLE IV

AROCLOR 1248 VAPOR FROM LUSTREX LATEX PAINT, LOT P-0938

Test No.	Date	Concentration, mg./cu. m. Chlorine	Aroclor	Remarks	Temp. At sample Intake
53	7/20	0.32	0.67	Center, east. 30 min. after painting	32° C.
54	20	1.28	2.68	Center, 1 1/2 hrs. after painting	32
55	21	0.93	1.93	Center, south	31
56	21	0.92	1.93	Center	31
57	21	0.53	1.11	North east corner	32
58	21	0.98	2.0		46
59	21	0.52	1.1	In laboratory hood	45
60	22	0.57	1.2		50
61	22	0.30	0.62	Corner	33
62	22	0.55	1.1	Center	32
63	23	0.12	0.25	Corner	40
64	23	0.14	.29	Center	33
66	24	0.11	0.23	Corner	33
67	24	4.9	9.6	Center	84
68	28	1.14	2.4	Corner } Anomolous deviation,	33
69	28	2.5	5.2	Center } probably air contamination.	34
70	8/12	0.34	0.71	Corner	38
71	12	0.21	0.44	Center	35
72	18	1.3	2.7	In laboratory hood at 60° C.	
73	18	0.52	1.1	Corner, fan off 4 hrs. after ventilating.	37
74	18	0.57	1.2	Center	41
76	9/10	0.23	0.48	Center	37
78	10	0	0	Corner	35
79	10/5	0.18	0.37	Center	35
80	5	0.43	0.89	Corner	34

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TABLE V

BLANK AND MISCELLANEOUS DETERMINATIONS

Test No.	Total Cl^- mg.	Concentration, mg./cu. m. Chlorine	Concentration, mg./cu. m. Aroclor	Remarks
12	0.006	-----	-----	
26	0.007	-----	-----	Reagent blanks. Total Cl^- in reagents to be subtracted from vapor determinations.
41	0	-----	-----	
52	0.007	-----	-----	
65	0	-----	-----	
77	0.006	-----	-----	
17	-----	0.21	0.42	atm. blanks on 5-19 and 5-20, on aroclor tank at North end of building. Assuming 50% Cl.
23	-----	0.32	0.64	
48	-----	0.32	-----	Blanks on air in instrument room before painting. Tests made on July 17.
49	-----	0.89	-----	
50	-----	0	-----	
51	-----	0.64	-----	
75	-----	0.10	-----	Air blanks in instrument room. No heat on combustion tube. Tests made on 9-10, and 10-5, respectively.
81	-----	0	-----	

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V. DISCUSSION:

The conditions of the tests and the limitations of the apparatus are such that absolute determinations of the chlorinated hydrocarbons are not possible. Comparative determinations of the total chlorine in the air during any specific test period can be made. The chlorine detected by this apparatus can be attributed to three sources:

1. Chlorinated hydrocarbons in the air (Aroclors, or any other chlorinated hydrocarbon).

2. Chlorine in the air as free Cl_2 or as HCl.

3. Chlorine ion present in the absorption solution and wash water.

Source No. 3 can be eliminated by use of Cl^- free reagents, or by making blank determinations on the reagents used and subtracting the Cl^- from this source from the total. The latter method was used throughout these tests.

The chlorine from source No. 2 listed above insofar as these tests are concerned is indistinguishable from the chlorine from the aroclors. Furthermore, the concentration of Cl_2 and HCl in the plant area varies so greatly and rapidly that the establishment of a satisfactory "blank" to compensate for this source is impossible without a statistical evaluation. It was felt that the amount of work involved for such an evaluation would not be justified. For this reason the results from all the vapor tests include chlorine from Sources 1 and 2. Under normal plant conditions, however, chlorine from Source 2 should be relatively low and the results are therefore assumed as chlorine from Aroclor. In some instances this assumption might produce gross error in the reported results, and must therefore be kept in mind in any discussion or evaluation of the reported results.

Another assumption was made in the method of converting the chlorine concentration in the air to Aroclor concentration. The Aroclor vapor was assumed to contain the same percent of chlorine as the aroclor from which it was vaporized. For example, Aroclor 1242 vapor was assumed to contain 42% chlorine. This assumption is probably not true since the vapor would more likely be richer in the more volatile, lower chlorinated material. Use of this assumption therefore minimizes the concentration of chlorinated hydrocarbon (Aroclor) calculated from any given chlorine concentration found in the air.

Tables I, II, and III are tabulations of all of the tests in the Aroclor plant. The calculated Aroclor vapor concentration was in the 0.5-1 mg. per cu. meter range during the still operation and with the sampling intake close to the still except for the tests during Aroclor

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1242 operations which gave comparatively high results. The average of the concentrations of Aroclor 1242 vapors tested by the stills (Positions 1, 2, 4, & 5) was 2.1 mg. per cu. m., which is about 2.3 times as much as the average of 0.89 mg. per cu. m. for the tests of Aroclor 1260 vapors in the same positions. The most severe concentrations were encountered during filtering and drumming operations ranging from about 2 to 5 mg. per cu. meter usually, with one test going up as high as 8 mg. per cu. meter during drumming.

No doubt the drumming operation results is the longest continuous exposure of an operator to high Aroclor vapor concentration in the plant, but this exposure only takes place intermittantly usually with a few days between the drumming operation. The filter operation presents a severe exposure also, especially, if it is necessary to change filter papers during filtration of an Aroclor batch. This operation requires 20-30 min. work in a heavy Aroclor vapor concentration. Formerly, this operation was necessary an average of one to two times per shift if both stills were producing liquid Aroclors. One of the filter presses was replaced by a Sparkler closed leaf filter about July 1, however, reducing the necessity of dressing a hot filter press to an average of probably less than once per shift.

An average daily exposure of the operator was not determined. As may be seen in Tables I, II, & III the Aroclor vapor concentration varies quite widely. Several variable factors such as location, temperature of Aroclor, operation being carried out, boiling point of the Aroclor under test, and direction and intensity of drafts through the still room all affect the aroclor vapor concentration. Furthermore, the time spent by the operator in any one operation or location varies greatly from day to day. This situation makes establishment of an average daily exposure practically impossible. However, from general observations in the plant it appears that the operator spends no more than 4 to 6 hours a day actually in the Aroclor still room, and of this time probably no more than half is spent in localities of severe aroclor vapor concentration.

Table IV is a complete tabulation of tests on the Lustrex Latex Paint, Lot P-0938, from Springfield. The same limitations and assumptions apply to these tests as to the plant tests. The Aroclor vapor concentration in the room rose rapidly to a maximum and then decreased. This trend is best presented graphicly as in Fig. I. Two sampling positions were employed in the room, one in the approximate center of the room, and the other with the intake close to the wall in the northeast corner of the room. These positions are noted in Table IV. The Aroclor concentration in the heated laboratory hood remained in the 1-2 mg. per cu. meter at 45-80° C. throughout the test. Table V is a tabulation of the various blank determinations which were made. The value used for the reagent blank in all of these tests was 0.006 mg. Cl^-.

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VI. CONCLUSIONS:

1. No absolute determinations of Aroclor vapor concentration can be made by the method used, due to presence of unknown quantities of Cl_2 and HCl in the air. The assumptions made permit simplified evaluation of the data.

2. The apparent Aroclor concentration in the plant varies greatly from 0.5 to 1 mg. per cu. meter under mild conditions up to 5 to 8 mg. per cu. meter under the most severe conditions.

3. The filtering and drumming operations present the most severe exposure.

4. No average daily exposure may be developed from these data.

5. The Aroclor vapor concentration in the room painted with Lustrex Latex Paint was sufficiently high (greater than 1.0 mg. per cu. meter) to make the room unusable for about 3 days. After this the odor and concentration of Aroclor 1248 diminished to a safe limit.

VII. RECOMMENDATIONS:

Any recommendations on the lack of toxic effects of Aroclor in the concentrations found will have to come from the Medical Department. However, based on the present MAC of 1 mg./ cu. meter, it appears advisable to wear a mask or respirator if concentrations of Aroclor are high enough to be irritable to the nasal membrane.

Since the room painted with Lustrex Latex Paint containing Aroclor 1248 definitely has an odor of Aroclor for several days after painting, caution should be exercised in recommending Aroclor for this use.

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March 23, 1954

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SAMPLING POSITIONS

Position Number	Description
1.	No. 1 still, simulating position of operator's head during charging operations. 54" from floor on west side of still about 18" from charging hole.
2.	No. 1 receiver, at sight glass, simulating position of operator's head. 54" from floor and 12" west of sight glass.
3.	No. 1 filter press; N- northside, S- southside. 54" from floor and 15" from edge of press when on south side or 9" from edge of press when on north side. Northside location simulates operator's head position when redressing the filter press.
4.	No. 2 still, same as position 1 above.
5.	No. 2 receiver, same as position 2 above.
6.	No. 2 filter press, same as position 3 above.
7.	At operator's desk, 54" from floor and 8" beyond front edge of desk to simulate position of operator's head when writing at desk.
8.	At scales for drumming Aroclor. 54" from floor and 14" from Aroclor outlet. Opposite side of drum from operator.
9.	Blank determinations as described.
10.	Miscellaneous positions, as described.

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OPERATING PROCEDURE FOR WILLSON PRODUCTS INCORPORATED, CHLORINATED HYDROCARBON SAMPLING APPARATUS

The following outline of operating procedure for this apparatus has been found quite satisfactory, and is considerably more detailed than the operating instructions supplied with the apparatus:

1. Mount the absorption apparatus on the tripod and start heating the furnace so that it reaches a bright red heat (850°C.) within 15-20 minutes. A rheostat setting of 40 was found to be about right on the instrument used here. This same setting is maintained throughout the run.

2. Connect the suction pump to the apparatus. A needle valve for bleeding in air to regulate the rate of flow through the apparatus affords much better control than does the bleeder cock on the pump alone. The base of an ordinary laboratory gas burner containing a needle valve is satisfactory.

3. Prepare the absorption column as follows:

   a. Close all outlets and fill with distilled water, drain and repeat to wash the column.

   b. Pipette 1 cc of the absorption solution into the column and allow to drain thoroughly. Add another 1 cc of absorption solution and drain. This prevents the absorption solution's being diluted by residual water from the washing operation. Close the stopcock and attach suction tube to top of column.

4. When the combustion tube reaches the proper temperature start the pump before opening the stopcock to prevent the absorption solution from draining into the tube connecting the absorption tube to the combustion tube. Turn the stopcock to the operating position, and simultaneously start the stop watch.

5. Adjust the rate of flow by manipulation of the bleeder valves to a rate of one to two liters per minute as indicated by the flow meter. The flow meter must be previously calibrated for use under the conditions of operation. This may be done by operating the apparatus with a calibrated gas meter (such as a wet-test meter) attached to the intake of the combustion tube.

   It takes 3 to 5 minutes for the pump to reach a constant rate and the bleeder valves must be adjusted frequently during this time to maintain the proper sampling rate. The adjustment of the sampling rate must always be done with the furnace for the combustion tube on and the furnace must then remain on during the entire run. Since the furnace draws current from the same source as the pump any large changes in the load on the line

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affect the speed of the pump.

6. Continue the run for the desired period, 30 to 40 minutes for concentrations below 10 mg. of chlorinated material per cubic meter of air.

7. Close the stopcock, stop the pump, and turn off the current to the furnace.

8. Wash the absorption column by filling with distilled water to just above the beads. The beads are washed in this manner just twice and the washings containing the chloride are collected in a clean, 2-oz. bottle. The chloride in the washings is then determined by Anniston Analytical Methods. The concentration in the air may then be calculated.

   It is convenient to remove the apparatus from the tripod and to place it at a lower level to carry out the washing operations, but it may be done in place by using a step ladder if desired.

9. Repeat steps 1 through 8 as the normal operating cycle.

Reagent Blank - Although the reagents used in the absorption solution are specified as chlorine free, it is advisable to run a blank determination on the reagents. This blank is run by preparing the absorption column as in step 3 for a regular run. Then without drawing air through the apparatus the column is washed and the sample taken as in step 8. The chloride is then determined in the usual manner.

Absorption Solution: Recrystallize sodium carbonate twice from distilled water in order to obtain chloride-free sodium carbonate. With this sodium carbonate make a saturated water solution (saturated at 25°C.). To 80 cc. of the sodium carbonate solution add 20 cc. distilled water. Add one gram chloride-free arsenic trioxide to the 100 cc. carbonate solution.

Chloride-free water may be obtained by distillation from sodium hydroxide; and chloride-free arsenic trioxide, by sublimation.

Sample Calculations:

1. $MgCl_1^- = \frac{ppm. \times gm.\ washings}{10^3}$

$MgCl_1^-$ = milligrams Cl^- in washings.
ppm. = Cl^- concentration in washings.
gm. = wt. of washings in grams.

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2. $MgCl/cu.\ m = \frac{MgCl_2^- \times 10^3}{\text{liters of sample}}$

= milligrams of Cl per cubic meter in the sample.

$MgCl_2^-$ = $MgCl^-$ in washings minus $MgCl^-$ in reagent blank.

Example No. 1:

Data:

Rate of sampling, lpm.	1.5
Duration of sampling, min.	30
Total vol. of sample, liters	45
Net wt. of washings, gm.	15.25
Total Cl^- in blank, mg.	0.006
% Cl in vapor being sampled	54
Concentration of Cl^- in washings, ppm.	6.3

Calculations:

1. $MgCl_1^- = \frac{6.3 \times 15.25}{10^3}$

$= 0.096$

$MgCl_2^- = 0.096 - 0.006$

$= 0.090$

2. $MgCl/cu.\ m = \frac{0.090 \times 10^3}{45}$

$= 2$

Chlorinated hydrocarbon concentration would then be:

$$\frac{2.0}{0.54} = 3.7 \text{ mg./ cu. m.}$$

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Example No. 2:

Data: Rate of sampling, lpm. 2.0
Duration of sampling, min. 43
Total vol. of sample, liters 86
Net wt. of washings, gm. 14.91
Total Cl^- in blank, mg. 0.006
% Cl in vapor being sampled 42
Concentration of Cl^- in washings, 3.2 ppm.

Calculations:

1. $MgCl_1^-$ = $\frac{3.2 \times 14.91}{10^3}$

= 0.048

$MgCl_2$ = 0.048 - 0.006

= 0.042

2. $\frac{MgCl}{cu\ m}$ = $\frac{0.042 \times 10^3}{86}$

= 0.49

Chlorinated hydrocarbon concentration would then be:

0.49/0.42 = 1.2 mg./cu. m.

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DATA SHEET - AROCLOR VAPOR DETECTION

Run No. Date: Bar. Press., mmg. Hg.:

Location of intake:

Rate of sampling, lpm.: Duration of sampling, min.:

Ambient temp. at intake, °C.: Total vol. of sample, liters:

General Remarks:

Analysis: Net wt. of washings, gm.:

Cl^- conc. in washings, ppm.:

Total Cl^- in sample, mg.:

Total Cl^- in blank, mg.:

Cl conc. in air, mg./cu. m.:

Aroclor conc. in air, mg./cu. m.:

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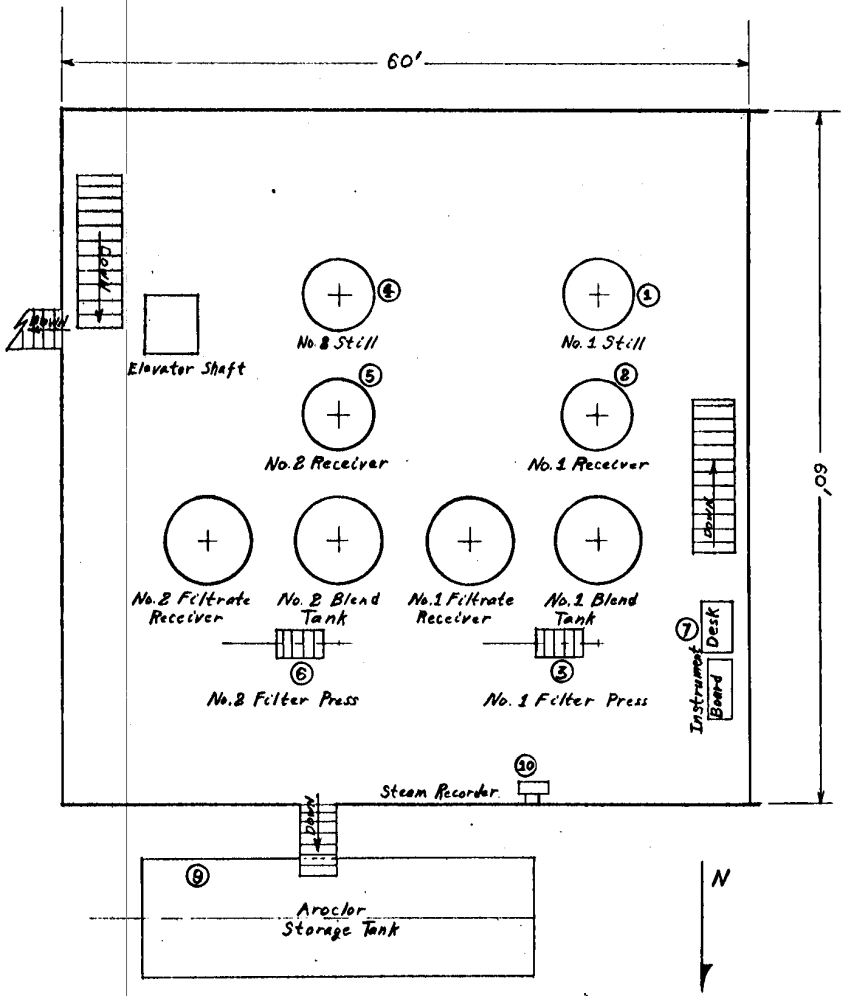
Diagrammatic Sketch
Aroclor Still Room



① Denotes Sampling Position

Scale: 3/32" = 1'-0"
H.B.R. - 6-22-53

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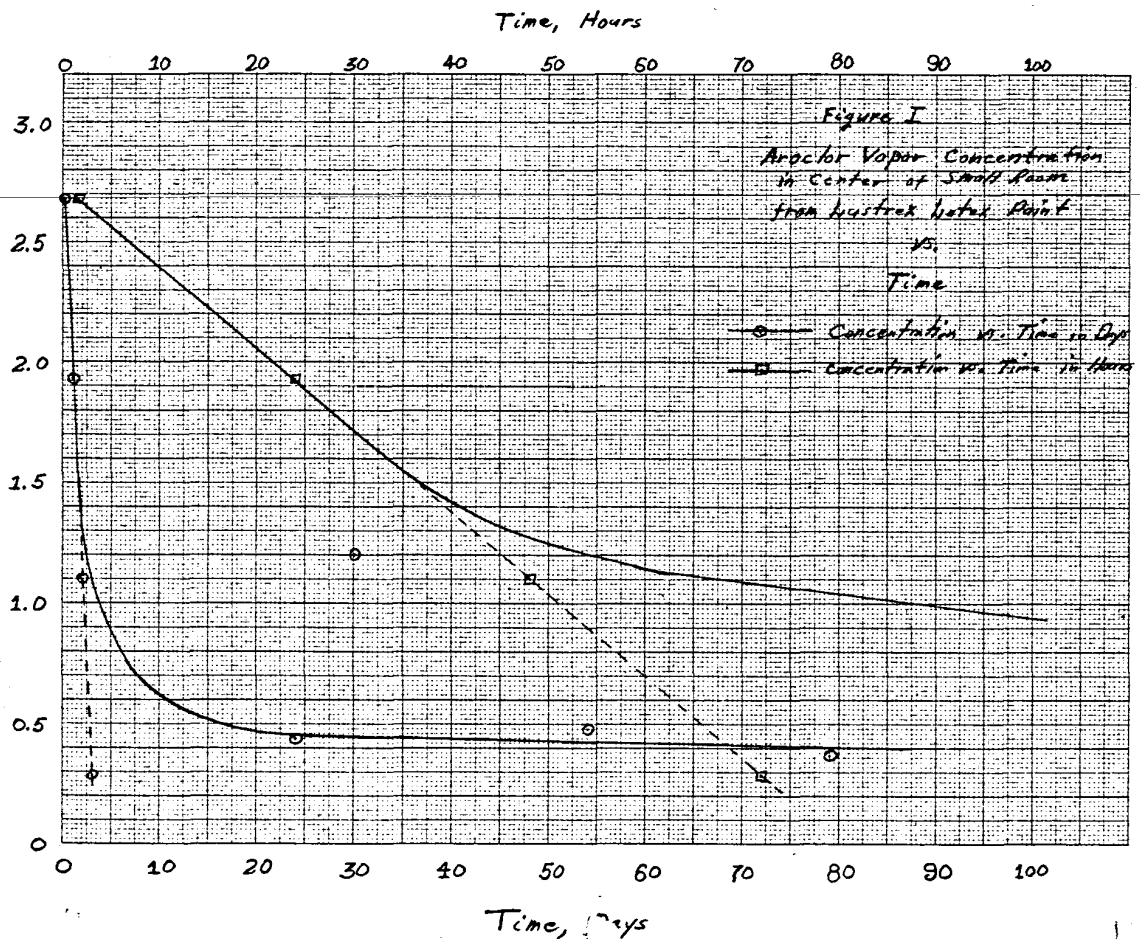

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