

St. Louis Research Department

Mr. D.B. Hosmer

ANNISTON

☐	Mr. J.H. Lam
☐	Mr. R.S. Nobus
☐	Mr. T.M. Patrick, Jr.
☐	Mr. R.L. Hedworth
☐	Mr. H.L. Merten
☐	Mr. T.W. Dalton

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AROCLOR RESEARCH PROGRAM

J.H. Lam	-M.O.-3
R.S. Nobus	-R.B.-3
T.M. Patrick, Jr.	-Res. 2
R.L. Hedworth	-Anniston
H.L. Merten	-Res. 1
T.W. Dalton	-W.G.K.

An active program has been underway since May to study the variables in the Aroclor process especially with the new stability test as a tool. This has meant repeating many of the experiments that have been made in years past in order to reevaluate the stability in terms of the newer analytical procedure.

Our program has been laid out with emphasis on the phases of the present process where deviations in the plant can conceivably occur. I am attaching a copy of the outlined program that Dr. Merten has suggested after a careful review of the former Swann and Monsanto reports and also your program suggestions of April 1, 1957.

Our conclusions to date have been reported in our progress reports and in several letters to your plant. These may be summarized as follows:

- (1) With all process conditions being met, the rate of chlorine feed can be increased to 1500 pounds per hour for 4000 lbs. batch with no deleterious effect on the stability of the Aroclor 1242.
- (2) The addition of air up to 50% by volume does not make an unstable Aroclor 1242 when operating under standard conditions and a normal chlorine rate of 660 pounds of chlorine per hour for 4000 pound charge of biphenyl.
- (3) The effect of temperatures lower than the specified 120°C. in the process may be detrimental to the stability of the Aroclor 1242. This effect varies with the amount of catalyst present.
- (4) Chlorination at temperatures of 120 to 160°C. have no adverse effect on the stability of the Aroclor 1242. Higher temperatures should not affect stability but other operating problems may result.
- (5) The aeration step is not critical in the production of stable Aroclor 1242. The quantity of air nor the temperature of the Aroclor between 100° and 150°C. appears to affect the stability of stable Aroclor. In cases where Aroclor has stability chlorides over 1 ppm, the aeration step is an aid in improving the stability.
- (6) The plant distillation step shows no tendency to isolate an unstable fraction.

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Other features of the program are being investigated now. The quality of the biphenyl is currently under study with the idea of finding how trace impurities may affect the stability.

The following data will give you a resume' of the various tests we have made. All tests are based on the standard process of chlorinating with chlorine gas at a feed rate of 660 pounds per hour based on a 4000 pound charge. Ferric chloride (0.03% based on biphenyl charge) is well mixed with the biphenyl and the temperature is maintained at 120-150°C. throughout the chlorination. After the chlorination, the crude Aroclor is blown with air at 100°C. and distilled from 0.25% Ca(OH)₂. At a pressure of 30 mm Hg., the distillation is cut when the column temperature reaches 235-240°C. The distillate is agitated for 4 hours with 0.2% dried attapulugus clay while the temperature is maintained at 70-80°C. Stability chlorides are run by the Monsanto test method based on 16 hours heating at 210°C.

Any variation from the above procedure is shown in the tables.

TABLE I

AIR-CHLORINE MIXTURES (LABORATORY)

<u>Air-Chlorine Volume Ratio</u>	<u>Distillation Yields</u>	<u>Monsanto Stability test, ppm chlorides</u>
0:100	98.6%	0.16
10:90	98.0%	0.14
20:80	98.5%	0.15
50:50	93.0%	0.13

TABLE II

CHLORINE FEED RATES

<u>Chlorine feed #1 hr.</u>	<u>Distillation Yields</u>	<u>Monsanto Stability test, ppm chlorides</u>
660 (lab.)	98.6%	0.16
1530 (lab.)	97.4%	0.12
700 (Anniston plant)	99.4%	0.49
800 " "	98.5%	0.31
850 " "	93.8%	0.22

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TABLE IIITEMPERATURE STUDIES (0.03% FeCl₃)

<u>Temperature, °C.</u>	<u>Yields</u>	<u>Monsanto Stability, ppm Cl</u>
100°, 8 hr.	98.5%	0.20
110°, 8 hr.	98.5%	0.12
120°, 8 hr.	98.5%	0.21
140°, 8 hr.	97.2%	0.13
90°, 2 hr.; 110°, 6 hr.	98.8%	1.22
95°, 0.5 hr.; 130°, 7.5 hr.	---	0.15
95°, 2 hr.; 130°, 6 hr.	98.0%	0.16
95°, 3 hr.; 120°, 5 hr.	98.0%	0.14

TABLE IVCATALYST CONCENTRATION AT 120°C.

<u>% FeCl₃</u>	<u>Yields</u>	<u>Monsanto Stability, ppm Cl</u>
0.01	98.1	0.89
0.008	97.7	8.87
0.005	chlorine passing through-slow chlorina- tion batch discarded	

TABLE VAERATION STEP - PLANT SAMPLES - W.G.K.

<u>Sample</u>	<u>Time of Aeration</u>	<u>Temperature</u>	<u>Rate of air, ml./min.</u>	<u>Stability test, ppm Cl</u>
1	None	---	----	0.96
1	0.5 hr.	100°C.	1330	1.62
1	1	100	1330	1.40
1	4	100	1330	1.10
1	4	150	1330	0.76
2	None	---	----	0.49
2	1	150	1700	0.46
2	4	150	1380	0.45

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TABLE VI
PLANT DISTILLATION TEST AT ANNISTON

<u>Aroclor 1242</u> <u>Batch No.</u>	<u>Run No.</u> <u>in Series</u>	<u>Monsanto Stability,</u> <u>ppm Chloride</u>
801	1	0.15
805	3	0.15
309	5	0.17
314	7	0.18
317	9	0.17
319	10	0.28
321	1	0.10
839	10	0.20

The conclusions above are based on these tests on the present process. At present, Dr. Merten is spending some time in the Anniston and Krumrich Plants to see if he can observe any operational details that may be a contributing factor to the manufacture of unstable Aroclor.

A. M. Ellenburg

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attachments

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