PROGRESS REPORT TECHNICAL SERVICES DEPT. – W. G. KRUMMRICH PLANT	JOB NO. 91370:4286	REPORT NO. (1)	DATE 2, 10, 7

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•TO RECEIVE DETAIL SECTION

TITLE: AROCLOR QUALITY

PERSONNEL: W. A. Krull, C. Kleist (W. C. Engman)

PROBLEM: Regain pre-September, 1970, quality levels on Aroclor 1260 and 1242.

SUMMARY

Four consecutive lots of Aroclor 1260 from late September through early December, 1970, failed the after corrosion chloride test. No lots have failed specifications since Aroclor 1260 lot KM-1019 in early December.

All four stability measurements on Aroclor 1242 have indicated a decrease in stability since October, 1970, as shown by cumulative sum charts.

To date, none of the actions or checks have linked a cause with the problem. Following is a list of actions and results to this point.

1. Raw Material Checks

   a. Ferric chloride was out of spec. but not to a degree which should cause a problem.

   b. Lime was checked against Anniston lime and found to be normal grade.

   c. Biphenyl checks were made and no abnormal material was found as compared to reserve samples from May.

   d. Bromine and organics in chlorine have been checked. Although impurities are present, no stability relationship has been found.

2. Laboratory distillation of individual plant chlorinator

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SUMMARY (continued)

batches showed all chlorinators bad.

3. Laboratory chlorinations were inconclusive. Our method failed to duplicate plant results in parallel runs.

4. Laboratory redistillation of previously analyzed bad material produced specification grade Aroclor 1260. Only one trial was made.

5. Cross checks between Anniston and WGK control labs have shown variations in stability analysis between the labs on eight samples.

6. A designed experiment was prepared for improving Aroclor 1260 stability but not run as normal conditions produced good Aroclor.

Future Work

1. Continue to monitor all stabilities of all electrical grade Aroclors for trends.

2. If Aroclor 1260 does not meet specs. on the next run, instigate cross checks with Anniston, run the designed experiment, and redistill bad material in the plant.

3. Continue to improve the data bank relative to raw materials, especially chlorine and biphenyl.

4. Continue cross checking with the Anniston Lab.

5. Determine a method for checking the efficiency of the chlorine demisters.

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I. BACKGROUND

A. Aroclor 1260 Stabilities (Data in Appendix A)

1. After Corrosion Chlorides

In September, 1970, Lot KM-1016 of Aroclor 1260 failed the after corrosion chloride (A. C. Cl) test. Lots KM-1017, 1018 and 1019 subsequently failed A. C. Cl. Of these lots, only KM-1016 showed A. C. Cl drastically above the specifications (0.22 vs 0.05 max.). The other three lots showed A. C. Cl close to the specification (0.06, 0.09). Some intermediate sampling of these lots showed scattered values from normal (in specification) to very high. Resampling and reanalysis was done with no significant change in results.

2. Monsanto Stability

Monsanto stability failed on Lot KM-1016 only, although it was bordering failure at 0.61 to 0.67 (spec. 0.7 max.) on the other three lots.

3. Hydrolysis Stability

Aroclor 1260 hydrolysis stability showed no significant variance from normal.

4. Rechecking Reserve Samples

A recheck of reserve samples from KM-1008, 1009, 1010, 1011 and 1013 for A. C. Cl. was done and resulted in good checks of OF 1008, 1009 and 1010 but failures on 1011 and 1013. All of these lots were analyzed as good material during the period May through August, 1970. A recheck of Monsanto stability of Lot KM-1013 showed it was ok.

B. Aroclor 1254

Electrical Grade 1254 showed no significant change in any of the stability measurements made (Monsanto, Thermal Chemical Chlorides, Westinghouse and Hydrolysis).

C. Aroclor 1242

Aroclor 1242 showed a decrease in stability, but it appears to have occurred closer to early October or about a week later than the 1260 problem. All four of the stability measurements have shown a decrease in stability of Aroclor 1242 although none have failed specifications.

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BACKGROUND (Continued)

D. Cumulative Sum Charts (Appendix B)

Cumulative sum charts of the stabilities of Aroclors 1242, 1254 and 1260 are attached as Appendix B.

II. WORK DONE AND RESULTS

A. Lab Distillation of Plant Batches (Appendix C)

Samples of each plant chlorinator were lab distilled and all five chlorinators showed poor A. C. Cl and good Monsanto stabilities.

B. KTA Session

A KTA session was held to analyze the problem. Raw materials, sample handling and analysis or plant operations were possible problem areas. More information on all of these areas was required since no single solution was evident. In order to isolate a raw material problem, lab chlorinations were planned. Further checking of plant and laboratory operation changes was also planned.

C. Lab Chlorinations (Appendix D)

Details of the lab chlorinations can be found in WGK AR&D Report No. 1771 dated November 19, 1970. Of the trials the most significant were the last two which were run parallel to plant batches and then samples were distilled in the lab. Neither of these runs corresponded to the plant batches indicating our methods were not able to duplicate plant operation.

D. Raw Materials (Appendix E)

1. Biphenyl

A check on biphenyl showed that the biphenyl being used in November was not different than biphenyl used during May when good 1260 was produced.

2. Lime

Lime was checked against Anniston lime and found to be of equal quality. Purchasing confirmed that no change in lime source had occurred.

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WORK DONE AND RESULTS (Continued)

3. Ferric Chloride (Catalyst)

Although a sample of the catalyst failed to meet some of the specifications, it was not thought to be significantly poor to cause the problem.

4. Chlorine

a. Bromine content is the only variable analyzed regularly that is considered to affect stability. This data showed no change in bromine content.

b. Organic content in chlorine is being checked by AR&D. This method has shown carbon tetrachloride and trichloroethane in the chlorine at Dept. 246. WGK AR&D Report No. 1821 dated January 18, 1971, describes the method and preliminary results.

c. Crosschecks with other chlorine using departments revealed low assays in chlorophenol production. This problem was noticed around the first of October and seems to coincide.

d. The chlorine demisters in Dept. 246 have been trapping a milky substance. In May, 1970, a similar material was caught. This material was identified as sulfuric acid with some carbon tetrachloride present. During this period, 1242 TCC's increased and batch reactions were killed. The organic layer recently contained an unknown with a boiling point of about 300° C.

e. Point Comfort chlorine was identified as the source of the acid/organics. (See memo by Dave Hobelmann dated July 15, 1970). An attempt was made to correlate the frequency of Point Comfort car unloading with the 1260 problem. No relationships seemed to exist between the source of chlorine and Dept. 246 usage.

f. A header change made in the Chlorine Department at the end of 1970 will provide Dept. 246 with a larger percentage of Point Comfort chlorine.

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E. Redistillation

A laboratory redistillation of a sample of Aroclor 1260 with high A. C. Cl resulted in material which passed the test.

F. Plant Operations

The department was combed for changes in equipment operations.

WORK DONE AND RESULTS (Continued)

The only change found that could be connected to stability was a lowering of the biphenyl feed storage temperature (115 to 95° C.) for safety reasons. This in turn lowered the starting chlorination temperature from about 125° C. to about 110° C. Research reports and the SMP recommend starting chlorination at no lower than 125° C.

G. Designed Experiment

Since the starting chlorinator temperature was suspect, a designed experiment was drawn up to include it and two other variables, each at two levels.

Variable	Normal or Low Level	High Level
Starting Biphenyl Temp.	110° C.	125° C.
Chlorination Temp.	150° C.	190° C.
Catalyst Level	2 #/Batch	4 #/Batch

These variables are the ones which have been shown to affect the stability of Aroclor 1242 in past research. When this test was started with all variables at the normal level, good Aroclor 1260 was produced. Since no inventory was on hand, production made the decision to complete a run at normal levels to insure sales could be met. No Aroclor 1260 has been produced since this run.

H. Control Lab Crosschecks (Appendix F)

Samples have been exchanged between the WGK and Anniston control labs. Both laboratories show occasional high values for the stability tests. There seems to be a lack of consistency in the data whether on good or bad material.

III. CONCLUSIONS

A. The stabilities of Aroclors 1260 and 1242 have apparently decreased (worsened) on the most recently produced lots as shown by the cumulative sum charts. No lots have failed tests since Aroclor 1260 Lot KM-1019.

B. Laboratory chlorinator batches were of little use in pinning down causes while troubleshooting. It was impossible to duplicate the plant chlorinator with our method.

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CONCLUSIONS (Continued

C. No definite raw material can be blamed for the upset. Although no conclusive data was found, the wide variances in the quality of finished goods in a single run seem to indicate chlorine as the most suspect of the raw materials.

D. Although it was done only once, redistillation improved the stability of Aroclor 1260.

E. To this point, data received from lab crosschecks has shown inconsistencies.

IV. RECOMMENDATIONS

A. TSD continues to monitor finished goods stability to catch any differences that may be correlated to the chlorine header change or any other change.

B. If further 1260 problems are encountered with after corrosion chlorides:

1. Make crosschecks with the Anniston lab.

2. Try redistillation in the plant.

3. Complete the designed experiment.

C. More data be obtained during production runs. Bromine, sulfuric acid and organics in chlorine and methyl biphenyl and low boilers in biphenyl are examples. This will give something to compare to if upsets occur and help pinpoint causes.

D. Continued crosschecking between the control labs is needed and should be carried out to determine normal variability between labs. Samples should be handled as similar as possible beforeanalysis to reduce sample variability.

E. TSD investigate ways to check the efficiency of the chlorine demisters.

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APPENDIX A

1970 AROCLOR 1260 LOT SAMPLES

Date	Lot	Monsanto Stability (Spec. 0.7 Max) 1st Analysis (ppm)	Monsanto Stability (Spec. 0.7 Max) Reanalysis (Date)	(Spec. 0.05 Max) After Corrosion Chlorides 1st Analysis (ppm)	(Spec. 0.05 Max) After Corrosion Chlorides Reanalysis (Date)
3/18	1003	0.43		0.02	
3/30	1004	0.31		0.03	
5/3	1005	0.34		0.02	
5/8	1006	0.46		0.02	
5/13	1007	0.45	0.46 (5/14)		0.14 (5/14)
5/17	1008	0.25		0.02	
6/5	1009	0.31		0.02	
6/21	1010	0.43		0.03	
7/10	1011	0.40		0.05	
8/9	1013	0.52		0.02	0.03 (8/11)
9/10	1015			0.33	
9/22	1016	4.9	2.3 (9/23)	0.22	0.68 (9/23)
			3.6 (9/24)		0.37 (9/24)
10/2	1017	0.61	0.67 (10/4)	0.06	0.06 (10/4) 0.06 (10/5)
10/8	1018	0.67		0.09	
12/9	1020	0.63	0.59 (12/14)	0.08	0.03 (12/14)
12/23	1021	0.51		0.04	

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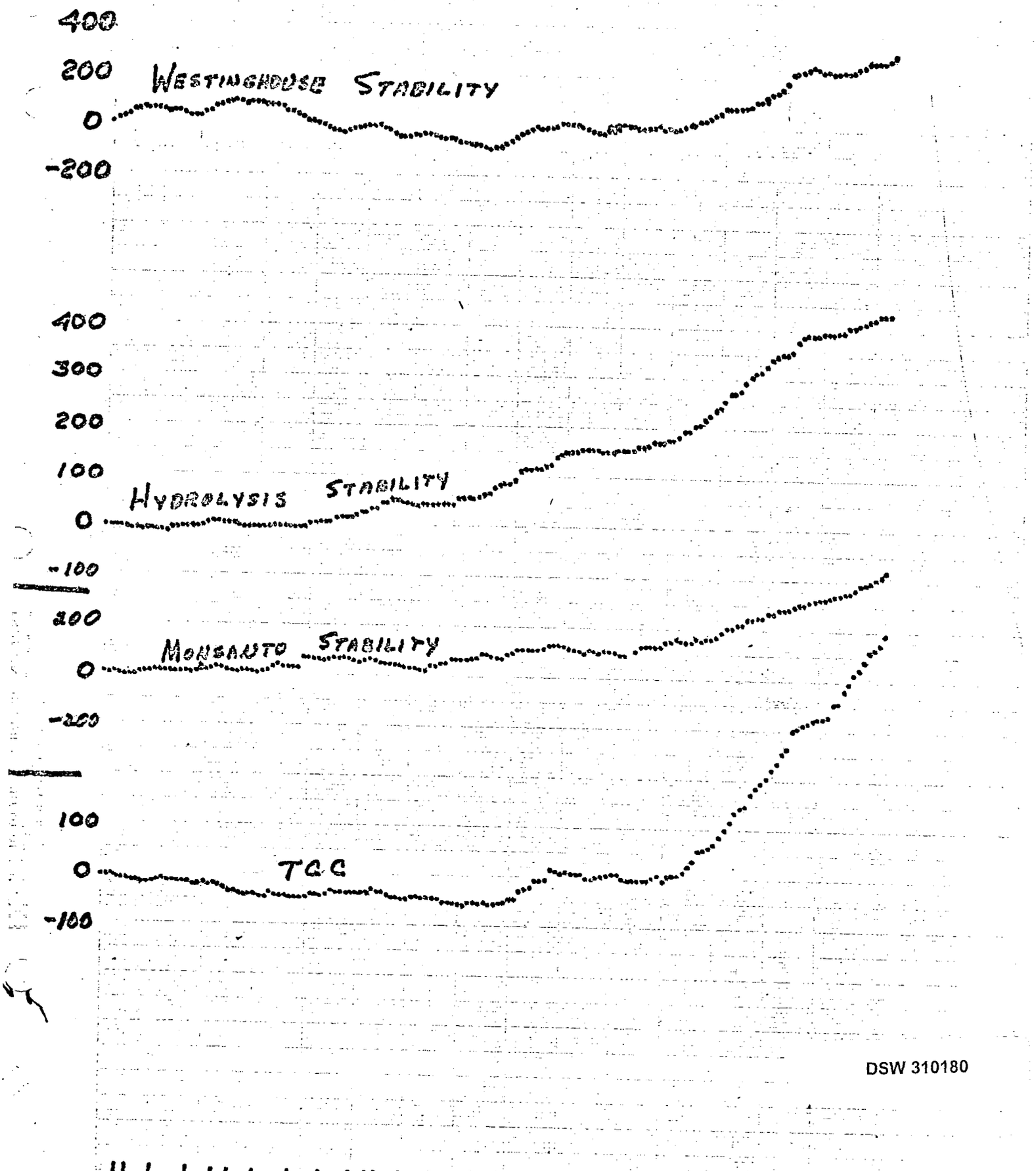
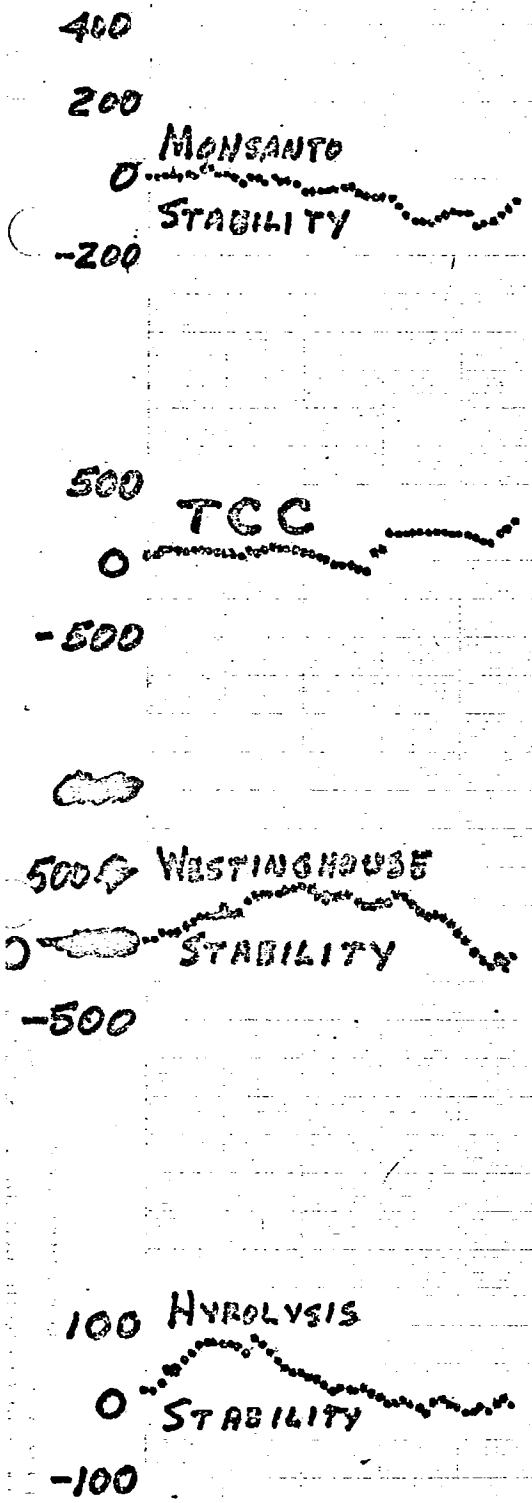
AROCLOR 1242 — CUMULATIVE SUM CHART STABILITIES

APPENDIX B



APPENDIX B (Cont'd)

AROCLOR 1254 — CUMULATIVE SUM CHART STABILITIES



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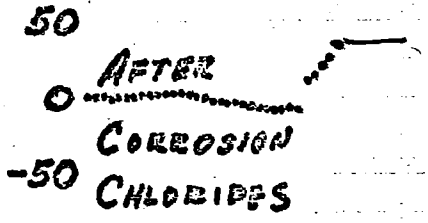
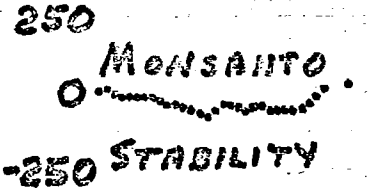
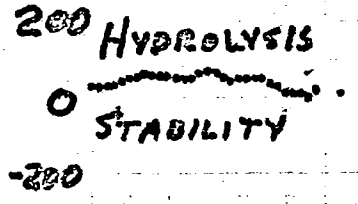
AROCLOR 1260 - CUMULATIVE SUM CHART STABILITIES

APPENDIX B (Cont'd)







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APPENDIX C

AR&D Distillation of 1160 (Part of Lot KM-1018)

Chlorinator No.	#1	#1	#2	#3	#3	#4	#5
Porocel Treatment	No	Yes	No	No	Yes	No	No
Mons. Stab.	.04	.04	.06	.38	.49	.24	.06
A. C. Cl$^-$	.19	.10	.10	.14	.08	.05	.14
A. C. Acid	.001	.001	.001	.001	.002	.001	.001
A. C. Color	20	15	15	25	20	10	15
T. C. Chlorides	--	--	--	0.84	0.75	--	--

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APPENDIX D

LABORATORY CHLORINATIONS

Date	Method	A. C. Cl^-	Mons. Stab.
Week of 10/26	1. Aroclor 1260 in lab from plant Biϕ and cylinder Cl_2	0.03	
	2. Aroclor 1260 from purchased Biϕ and cylinder Cl_2	0.04	
	3. Aroclor 1260 in field from plant crude Aroclor 1154, plant Cl_2	0.04	
11/2	1. Aroclor 1260 from plant raw materials	0.06	0.89
	This plant run parallel to plant batch #421. Lab distillation of #421 crude produced these results - 0.35		
11/4	1. Lab size batch parallel to plant batch #428 with plant materials, then lab distilled both plant and lab.		
	Lab size	0.11	0.27
	Plant #428	0.01	0.49

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APPENDIX E

BIPHENYL ANALYSIS

Sampling Date	Car No.	3,4 Methyl-Biphenyl	Low Boilers	Est. Use Date
8/6/70	NATX 21258	740 ppm	380 ppm	-
8/13/70	NATX 21265	1260	290	-
9/1	GATX 94514	670	260	9/3, 9/4
9/2	NATX 21249	710	400	9/3, 9/4
9/3	NATX 21265	700	300	9/5, 9/6, 9/7

Samples of biphenyl representing AM-160, or May production, was compared to car NATX-21249 dated 11/2/70 by GC and found to have no significant differences.

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APPENDIX E (Cont'd)

FERRIC CHLORIDE ANALYSIS

	Spec.	Actual
Assay	97.0%, Min.	98.9%
Sulfates	0.05%, Max.	0.20%
Ferrous Salts	Trace, Max.	>Trace
Solubility:		
Water	Complete to	>Sl. Turbid
Alcohol	Sl. Turbid, Max.	

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APPENDIX E (Cont'd)

BROMINE IN CHLORINE

Date	Bromine (ppm)
1-19-70	42
1-26	246
1-23	452
1-21	220
1-28	90
2-2	81
2-5	162
2-7	222
2-8	190
2-9	66
2-17	98
2-18	54
2-21	392
2-22	262
2-22	313
2-23	371
2-24	480
2-25	146
2-25	480
2-27	210
2-27	494
2-28	389
2-28	399
3-3	400
3-6	316
3-7	332
3-11	252
3-18	25
4-1	66
4-8	230
4-22	298
4-29	294
5-7	177
5-13	10
7-28	212
9-4	90
9-30	246
10-6	40
11-3	76
11-12	280
11-16	187
11-25	247
12-2	282

Date	Carbon Tetrachloride (ppm)	Trichloroethan (ppm)
	From 246 Vaporizer	
12/14/70	37	25
12/21/70	40	35
1/11/71	175	15
	From Nash Pumps	
1/12/71	5	14

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APPENDIX F

AROCLOR 1260 CONTROL LAB COMPARISON

Sampled	Test	Spec.	Typical	Anniston (Rerun)	WGK (Rerun)
KM-1019 12/3/70	A. C. Cl	0.05 Max.	0.03	0.02	0.24
	Hydrolysis	None	0.20	0.71	0.45
	Monsanto	0.7 Max.	0.40	0.58	0.80
KM-1019 Finished Product Tank 12/3/70	A. C. Cl	0.05 Max.	0.03	0.17	0.04
	Monsanto	0.7 Max.	0.40	0.15	0.64
KM-1020	A. C. Cl	0.05 Max.	0.03	0.07	0.03
	Free Cl	0.05 Max	0.01	0.07	
	Monsanto	0.7 Max.	0.40	0.37	0.59
KM-1021 12/14/70	A. C. Cl	0.05 Max	0.03	0.018	0.04
	Free Cl	0.05 Max.	0.01	0.018	
	Hydrolysis	None	0.20	0.46 (0.53)	0.09 (0.10)
	Monsanto	0.7 Max.	0.40	0.39	0.51
Am-7	A. C. Cl	0.05 Max.	0.03	0.04	0.01
	Hydrolysis	None	0.20	0.28	0.25

Aroclor 1242 Control Lab Comparison

KM-4013	Monsanto	0.4	0.10	0.03	0.15
	Hydrolysis	0.5	0.07	0.28	0.07
	TCC	0.65	0.15	0.19	0.10 (0.14)
AM-705	Monsanto	0.4	0.10	0.05	0.03
	Hydrolysis	0.5	0.07	0.07	0.07
	TCC	0.65	0.15	0.15	0.26 (0.28)
AM-137	TCC	0.65	0.15	0.16 (0.14)	0.21 (0.22)

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