PROGRESS REPORT

TECHNICAL SERVICES DEPT. – W. G. KRUMMRICH PLANT

JOB NO.	REPORT NO.	DATE
91370:4286	3	10 ,27 , 7

W.G. KRUMMRICH PLANT			GENERAL OFFICE	OTHERS
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☒ P.E. Heisler	☒	☒	•☒ D.B. Hosmer/ F.J. Holzapfel	Nitro
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*☒ J.A. Gloeckner	☒	☒	•☒	*☒ E.M. Emery
*☒ R.M.McCutchan	☒	☒	•☒	*☒ J.F. Quinn
•☒	☒	☒	•☒	*☒
•☒	☒	☒	*☒	*☒

• TO RECEIVE DETAIL SECTION

TITLE: AROCLOR QUALITY - DEPARTMENT 246

PERSONNEL: W. A. Krull, E. E. Stewart (W. C. Engman)

PROBLEM: Regain pre-October, 1970, quality levels on Aroclor 1242.

SUMMARY

Since the last report, Aroclor 1242 Thermal Chemical Chloride stabilities have continued high at about 0.20 to 0.30 with the exception of the period May and June when TCC's averaged 0.18 ppm for 23 lots. Other stabilities on 1242 and all stabilities on 1254 have remained stable at normal levels.

Fifteen biphenyl tank car samples from periods of high and low TCC's were analyzed by the Technology Dept. and found to contain 13 impurities, 6 of which were identified. Fluctuations in the 6 identified impurities and some of the unidentified impurities were shown statistically to correlate at 90% or higher confidence levels to fluctuations in the TCC levels. 3 and 4 Methylbiphenyl correlated at the 99% confidence level.

Future Work:

1. Develop an analytical technique at WGK to analyze biphenyl for the impurities.
2. Collect data and attempt to verify correlations between biphenyl impurities and TCC levels in Aroclors 1242 and 1016.
3. Recommend specifications if warranted.

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DSW 329178

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BACKGROUND

Since report #2 (4/30/71), the Aroclor 1242 thermal chemical chloride (TCC) stability levels decreased significantly in May and June from earlier 1971 values. January through April the TCC's showed an average value for 39 lots of 0.28 ppm while May and June averaged 0.18 ppm for 23 lots. The lots produced since June (total of 18) have shown a return to poorer levels averaging 0.24 ppm. Other 1242 stabilities remained fairly constant.

Stability measurements from the first runs of Aroclor 1016 indicate the stability of that product to be about the same as Aroclor 1242, perhaps slightly more stable. This is based on only seven column runs.

Aroclor 1254 stabilities have been constant or have shown a slight improvement as a general trend.

Aroclor 1260 Monsanto stability has become poorer while after corrosion and hydrolysis stabilities have been fairly constant.

No lots have been outside of stability specifications. The appendix contains cumulative sum charts for all stabilities.

WORK DONE AND RESULTS

Although many attempts were made to correlate changes in TCC's to process variables, raw materials, and various other things, the only variable measured which has shown a correlation is biphenyl quality. No correlations resulted from the following areas in addition to those previously reported. Chlorine data was somewhat scarce.

1. Point Comfort chlorine non-volatile matter
2. Point Comfort chlorine water content
3. Chlorine assay
4. Aroclor department key variables
5. Bromine content of chlorine.

This report describes primarily the work done in relation to biphenyl impurities.

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2.10.13

BIPHENYL ANALYSIS

In September, 1971, the Technology Department at South Second Street (E. M. Emery, G. M. Gasser) analyzed 15 samples of biphenyl for trace impurities. The following samples were analyzed:

1. Ten Anniston samples from late 1970 and early 1971, corresponding to a period of high TCC's at WGK.

2. Three WGK reserve car samples from May and June, 1971, corresponding to a period of lower TCC's.

3. Two WGK reserve car samples from late June, 1971, corresponding to a period where TCC's returned to a higher level.

In each of these samples there were 13 impurities at various levels. A tabulation of the data appears in the Appendix along with a memo describing the technique used to quantify the impurities found. Of the 13 impurities found, the following were identified by name.

1. Napthalene
2. 2-Methylnapthalene
3. 1-Methylnapthalene
4. 2-Methylbiphenyl
5. 3-Methylbiphenyl
6. 4-Methylbiphenyl

The other 7 compounds were not identified by name.

STATISTICAL CORRELATIONS

The data was given to the WGK AR & D Section (Jim Gloeckner) to determine the statistical significance. The results were summarized in a memo which is attached in the Appendix. The data shows that the changes in impurity levels for all identified impurities and most unidentified impurities correlated to changes in TCC levels at the 90% confidence level or above.

PAST RESEARCH

In 1967 and 1968 the WGK PR & D Group undertook extensive quality related work. (Job No. 91370:3794) While investigating the various chlorination perameters, they discovered that biphenyl quality had a definite effect on the TCC level of the distilled product. In Report No's 1 and 4 they discussed the details of their biphenyl investigations. Using laboratory chlorinators they were never able to identify the impurity in the biphenyl which caused the fluctuation of TCC's.

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CONCLUSIONS:

1. One or more of the identified impurities in biphenyl may be the cause of the higher TCC's.

2. Identified biphenyl impurities may be indicators of unrecognized impurities which are the cause of higher TCC's.

3. Lab work in 1967 and 1968 may have encountered the problem of not being able to duplicate plant operations and therefore could not pin down the biphenyl impurity related to TCC's.

4. This lab work also relied on spiking the biphenyl to determine a biphenyl impurity/TCC relationship. If the identified impurities are only indicators of the presence of a trace impurity, they would have failed. The trace impurity might not have been added and no change would have been noticed.

RECOMMENDATIONS:

A program should be initiated as follows:

1. Hold all WGK reserve biphenyl samples for future analysis.

2. Develop a method at WGK to quantify all or some of the identified biphenyl impurities.

3. Collect data on biphenyl impurities and TCC levels to note any fluctuations that may occur in one or both variables.

4. If no fluctuations occur due to planned improvements in the Anniston biphenyl process (replace column trays and adding a third column), obtain Anniston cooperation to produce biphenyl at desired impurity levels. Control the WGK biphenyl and finished goods inventory to prevent masking of impurity fluctuations.

5. Using normal statistical procedures, determine which impurities if any correlate to stability changes in Aroclor 1242 or 1016 stabilities.

6. If correlations are apparent, recommend appropriate specifications for biphenyl to be sent to WGK.

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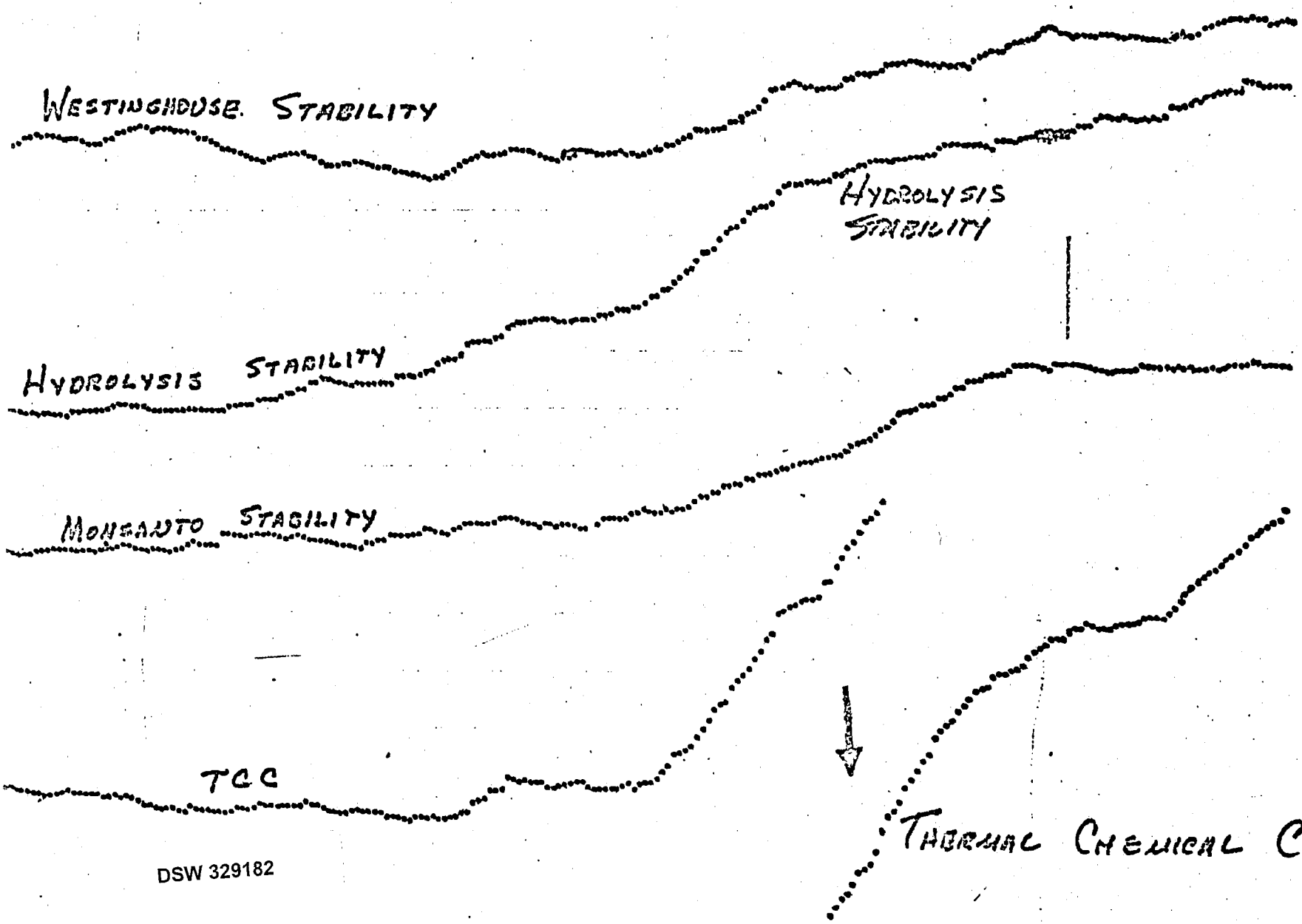
AROCLOR 1242 STABILITIES
CUMULATIVE SUM CHART



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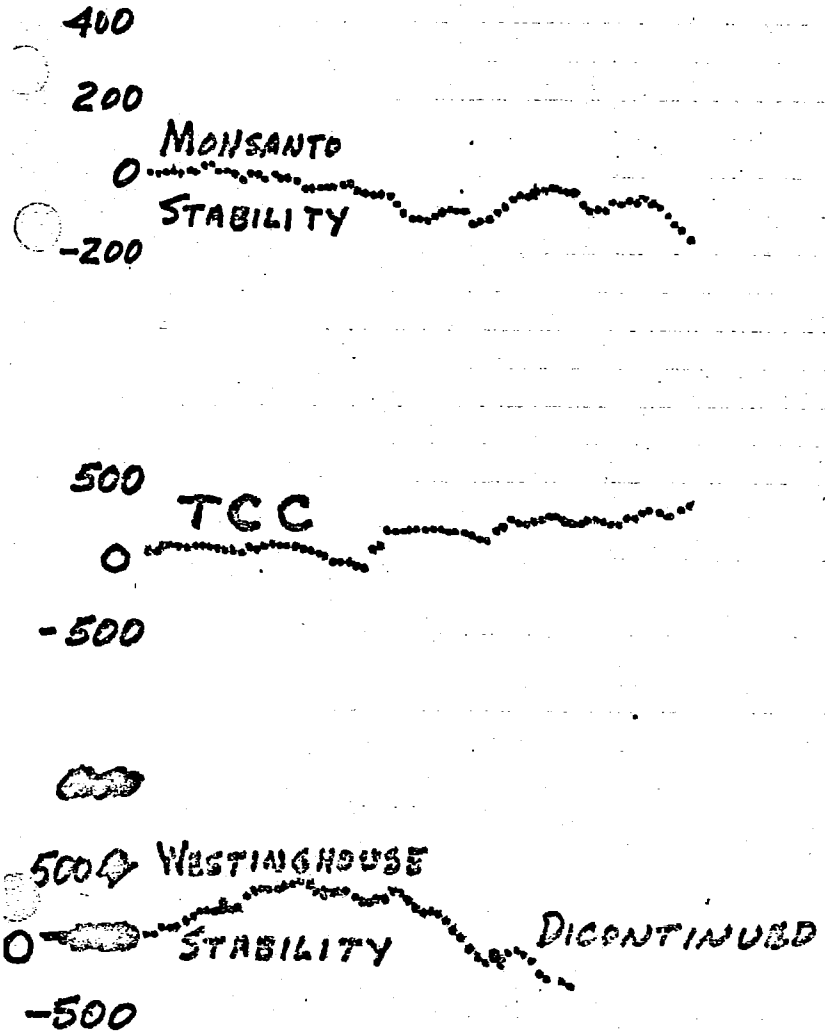
AROCLOR 125[4] — CUMULATIVE UM CHART STABILITIES

APPENDIX B (Cont'd)



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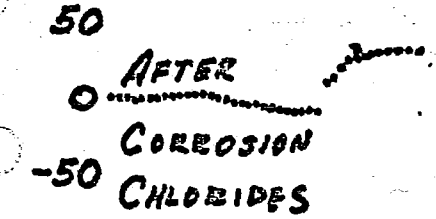
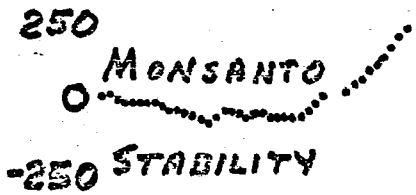
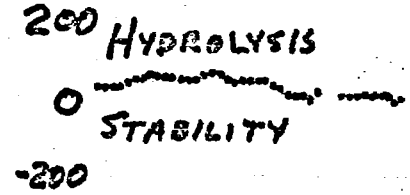
AROCLOR 1260 - CUMULATIVE SUM CHART STABILITIES

APPENDIX B (Cont'd)







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68 | 69 | 70 |

AROCLOR 1^16 CUMULATIO SUM CHART
STABILITIES

100

WESTINGHOUSE STABILITY

0

-400

HYDROLYSIS STABILITY

0

-150

HOUSAUTO STABILITY

0

-200

200

100

0

TARGARIL CHEMICAL CHLORIDES

-100

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