PROGRESS REPORT

TECHNICAL SERVICES DEPT. – W. G. KRUMMRICH PLANT

JOB NO.	REPORT NO.	DATE
91370:4286	2	4 ,30 ,71

W.G. KRUMMRICH PLANT			GENERAL OFFICE	OTHERS
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TITLE: AROCLOR QUALITY

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

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

SUMMARY Aroclor 1242 Thermal Chemical Chlorides showed a step change from 0.14 to 0.28 (Spec. 0.65, Max.) in October, 1970.

No correlation could be found between the following operating data and the step change in thermal chemical chlorides:

1. Chlorine mist eliminator pressure drop
2. Catalyst level
3. Maintenance records
4. Still pot temperature/pressure
5. Sample bottles

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Information on raw material purity, chlorinating temperatures and lime addition were not available for that time period. All of these variables except raw materials are now being monitored and are within proper control limits.

Analytical precision has been determined by J. Gloeckner and the Contro Lab showing the total test variability to be ± 0.05 ppm at two standard deviations. Sampling variability was shown to be insignificant. Reserve samples from before and after October were rerun showing the Lab was able to duplicate results.

Future Work:

1. Develop tests and data on chlorine assay, moisture, organics and sulfates.
2. Gather data on methylbiphenyl & napthalene in biphenyl.
3. Continue to try to correlate changes in variables to changes in stability.

Wayne A. Krull

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I. BACKGROUND

Aroclor 1242 has shown a step change for the worse in Thermal Chemical Chloride Stability during October, 1970. This is shown by cummulative sum charts in Report #1 and has been confirmed using statistical methods. Other stabilities measured (Monsanto, Westinghouse, Hydrolysis) seem to show a poorer level but it is not as clear cut as the TCC's

Recent TCC's have shown considerable variation. For the five lots (455 to 460) made from April 5 to April 14, 1971, the TCC's improved to an average of 0.19. The last two electrical grade lots (461, 463) averaged 0.27.

This report describes primarily the checking of department operations which was done in an attempt to discover possible causes for a change in stability.

II. WORK DONE AND RESULTS

A. All available data from the period around the stability change was surveyed for abnormal conditions. Present department operating conditions also were checked for abnormalities.

B. Operating Conditions

1. Chlorine Brink Mist Eliminators

The pressure drop across the two elements exceeded the design pressure drop at lower than design rate. The flow rate vs. pressure drop data was sent to the Brink design section of Enviro-Chem. Feedback from Enviro-Chem indicated the system should operate at a maximum of 15" W.C. pressure drop, but re-entrainment should not occur until a pressure drop of 30" W.C. is reached. At present maximum expected flow rates a pressure drop of about 21" W.C. would be expected. Extrapolation of existing data indicates flow rates of about 12,000 pounds per hour would be needed to reach 30" W.C. A plot of flow rate vs. pressure drop for the system appears as Appendix A.

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II. WORK DONE AND RESULTS

B. Operating Conditions (Continued)

2. Catalyst Level

Three times per week the last chlorinator of the series (#9) is analyzed by the control lab for ferric chloride content. A cumulative sum plot of past data indicates a change upward in the last part of October, 1970. This increase is the result of a deliberate attempt by Production to increase the level of catalyst trying to improve stabilities. Past research has shown that increasing the catalyst level in chlorination should improve the stability of Aroclor or have no effect. A plot of catalyst concentration vs. time appears as Appendix B.

3. Maintenance Records

The Master Job Reports for August, September and October, 1970, were reviewed for possible mechanical changes which could affect Aroclor stabilities. The master job report lists all mechanical repairs and construction which occurred during a particular month. No changes or repairs were noted which could affect stability.

4. Still Pot Temperature/Pressure

Pressure and temperature data from the still log sheets were plotted for the last four months of 1970. This plot shows no change during October. A change showed up in early November indicating improved control of the still pressure which should not adversely affect stability.

5. October data does not exist for chlorinator temperatures, lime charging or chlorine quality. One reserve sample of biphenyl was checked and found to be normal. Process key variables are now being recorded with the exception of raw materials. Key variables which could effect stability include the following:

II. WORK DONE AND RESULTS

B. Operating Conditions (Continued)

5. (Cont'd)

   a. Methylbiphenyl in biphenyl
   b. Sulfates and organics in chlorine
   c. Chlorination temperature
   d. % ferric chloride in #9 chlorinator (last in series)
   e. Distillation pressure
   f. Amount of lime added to distillation
   g. Still level.

   Other than raw material quality, all of the key variables related to stability are within control limits.

6. During December, 1970, production recycled sample bottles for almost all of the month. Reserve samples from the first part of the year were emptied, rinsed and reused. The TCC's for the month of December averaged almost as high (0.24) as in November and January (0.28, 0.29). This is evidence that sample bottles are not the problem.

C. Precision of TCC Measurement

Jim Gloeckner with the cooperation of the control lab has completed a statistical analysis of the normal precision of the TCC measurement in the WGK lab and a check of reserve samples from before and after October. The check of reserve samples showed the lab could duplicate results. The precision check on sampling and analysis showed the precision of the total test to be $\pm$ 0.05 ppm at two standard deviations with sampling not being significant. Samples will be sent to Anniston to include a comparison between the two control labs.

III. CONCLUSIONS

A. No documented change in Dept. 246 operations occurred during the period of the stability change.

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III. CONCLUSIONS

B. Additional chlorine mist eliminator capacity is not needed.

C. The laboratory has shown that normal variability does not explain the differences in TCC's from before and after October, 1970.

D. Analytical tests on reserve samples have shown the lab able to reproduce both high and low TCC's.

IV. FUTURE WORK

A. Analyze chlorine for assay, sulfates, organics and moisture. Develop methods where necessary.

B. Analyze biphenyl for methylbiphenyl and napthalene.

C. Correlate variable changes to stability changes.

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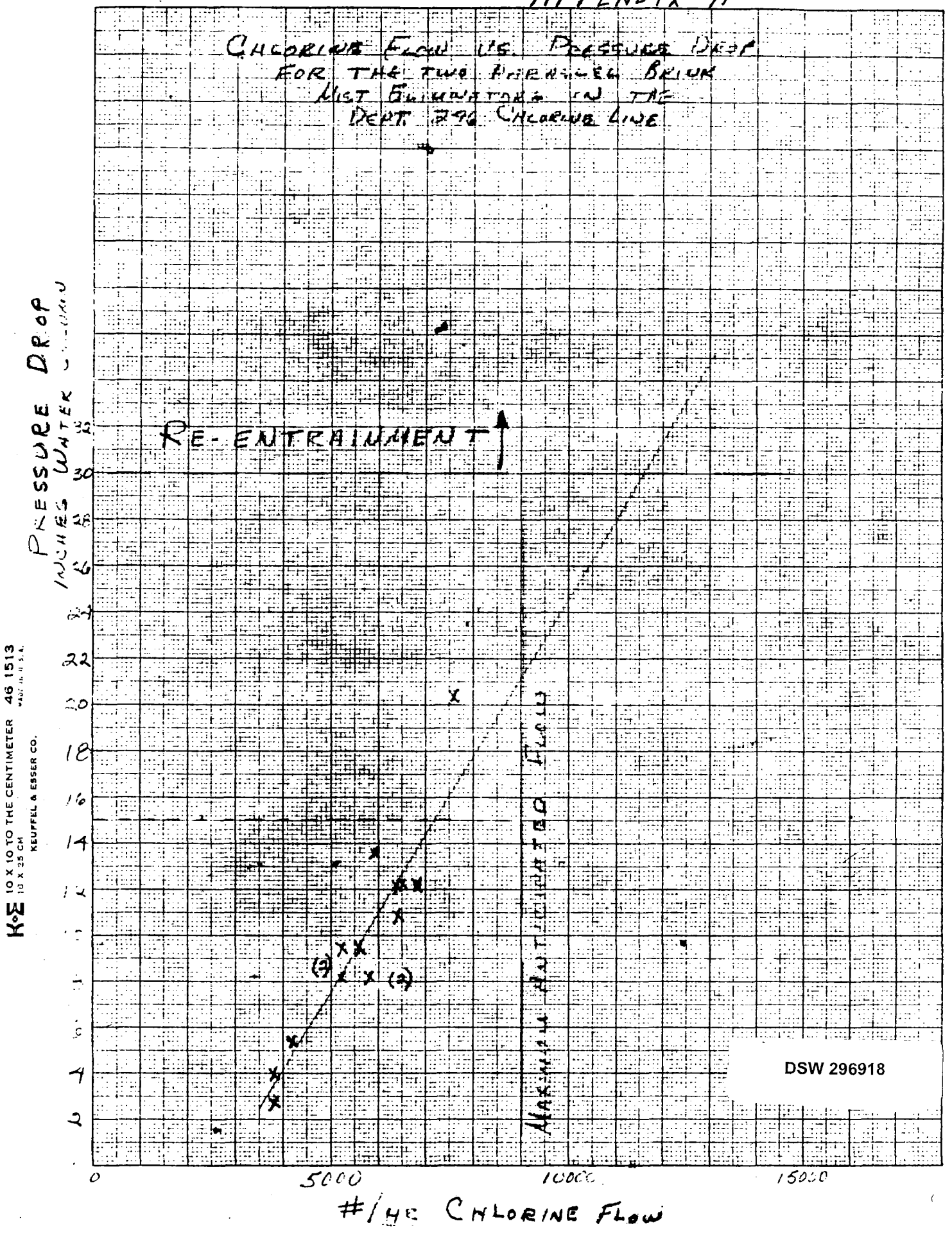
APPENDIX A



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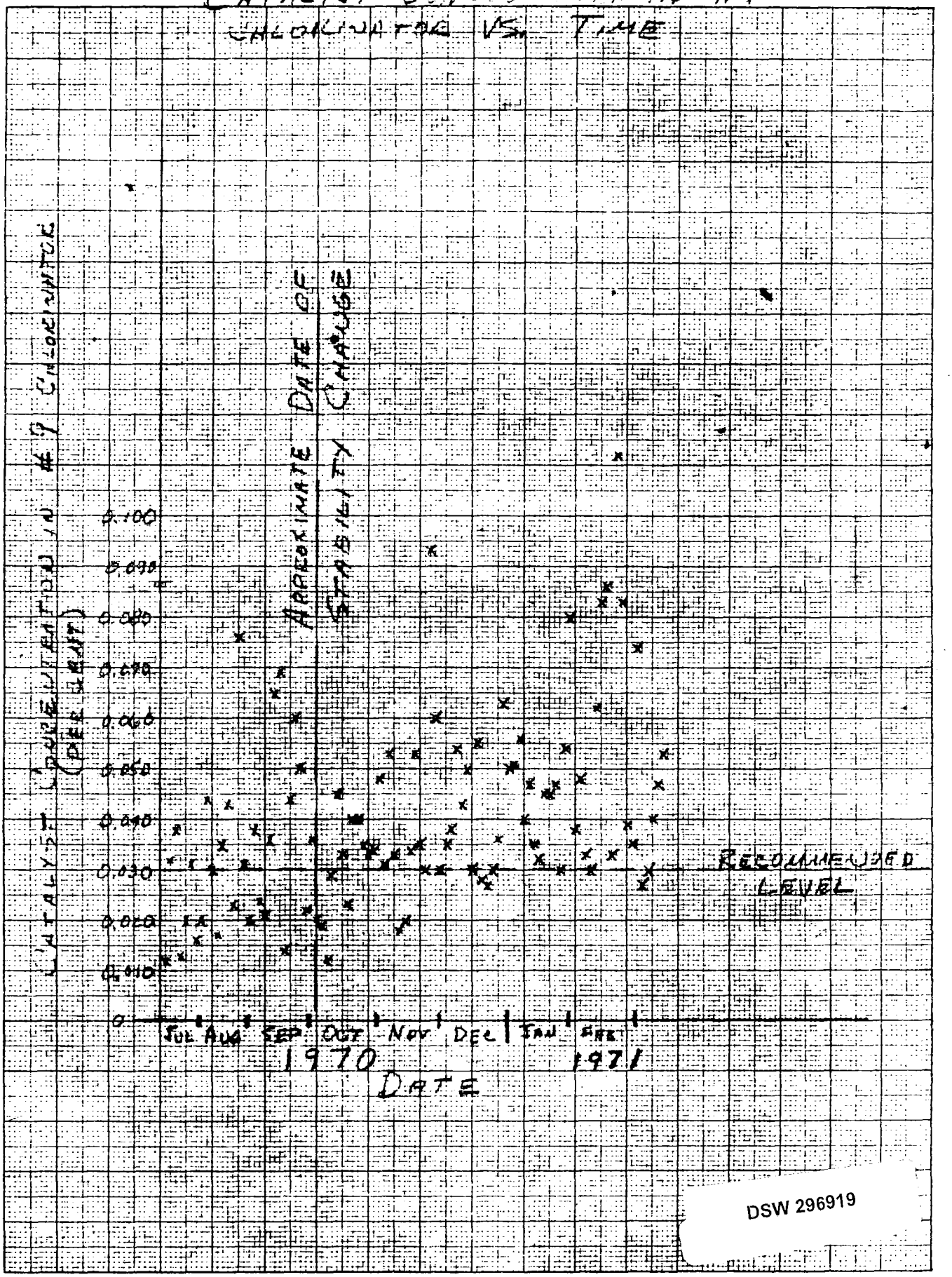
APPENDIX B

CATALYST CONCENTRATION IN H9

CHLORINATOR VS TIME



K&E 10 X 10 TO THE CENTIMETER 46 1513
10 X 25 CM.
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