PROGRESS REPORT TECHNICAL SERVICES DEPT. – W. G. KRUMMRICH PLANT	JOB NO. 91370:3794	REPORT NO. 6	DATE 1, 2, 69

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TITLE: Aroclor Process Studies - Plant Demonstration of Iodine Co-Catalyst

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PROBLEM: Determine the effect of the ferric chloride and iodine catalyst system on the stability of plant produced Aroclor 1242.

SUMMARY

Four plant tests of iodine co-catalyst were conducted in the batch chlorinators of Department 246. The stability of the Aroclor as measured by the Thermal Chemical Chloride Test (TCC Test), was not improved during any test. Five different sets of conditions were used in attempts to demonstrate the benefit of iodine co-catalyst in the plant. They included 0.02% and 0.05% iodine based on biphenyl, 175°C and 195°C chlorination temperature, off gas demister treated with crude and complete absence of the off gas demister.

Future Work: Chlorine quality will be thoroughly investigated as the major variable which has prevented agreement between laboratory and plant results in these as well as past plant tests.

Acknowledgements: These tests could not have been conducted without the efforts of Paul Verna, Bob McCutchan, Harry Ray, Virgil Brawly, Ron Kuster, Joe Lawrence, and Joel Blanchard.

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DETAILS

BACKGROUND

The use of the ferric chloride-iodine catalyst system is the only action which has consistantly met the Divisional Stability Goal of less than 0.1 ppm Thermal Chemical Chlorides (TCC) in the laboratory. It is the purpose of this report to summarize the results of the plant demonstrations of iodine co-catalyst.

RESULTS AND DISCUSSION

The first plant test was conducted in three successive batches of two different batch chlorinators. The first and third batches in each chlorinator served as baseline while the second batch contained 0.05% iodine. The chlorination temperature was 175°C. Due to the fact that the test occured during an Aroclor 1248 production run, both 1142 and 1148 samples were obtained and the TCC results are shown in Table I. The 0.07 ppm TCC result for the Aroclor 1242 post test baseline is evidently within the normal batch variations.

The second test was more complete, but unfortunately the conclusion was the same. This test involved six consecutive batches in each of three different chlorinators. The first, fifth, and sixth batches were used as baselines and the second, third, and fourth contained 0.05% iodine. The chlorination temperature was 195°C and the results are shown on Table II. The average of all the baseline batches is 0.36 ppm and the average of the test batches is 0.25 ppm. This difference is not significant at the 95% confidence level.

While the first two plant tests were in progress, the effect of a glass wool simulated demister was being studied in the laboratory. The results of this work, which are summarized in Progress Report 91370:3794, No. 5, indicated that the simulated demister actually caused the iodine to be detrimental to Aroclor stability. It was found that this deleterious interaction of iodine and demister could be eliminated by treating the demister with the ferric chloride catalyst in crude Aroclor.

On the basis of that lab work, a third plant test of iodine co-catalyst was conducted. The demister in a plant chlorinator was soaked with crude Aroclor 1142 for thirty minutes. This was accomplished by pumping crude Aroclor from a second chlorinator into the first until crude was detected above the demister. After fifteen minutes the crude level was lowered and raised again for fifteen minutes. A batch was then made with 0.02% iodine, the chlorination mass sampled, and the demister soaked for fifteen minutes again.

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TABLE I

TEST I - TCC RESULTS

AROCLOR 1242		AROCLOR 1248		
CHLORINATOR NO. 2	CHLORINATOR NO. 3	CHLORINATOR NO. 2	CHLORINATOR NO. 3	REMARKS
TCC, ppm		TCC, ppm		
0.19	0.20	0.26	-----	Baseline
0.19	0.20	0.12	0.17	0.05% I_2
0.07	0.19	0.19	0.19	Baseline

TABLE II

TEST II - TCC RESULTS

CHLORINATOR NO. 2 TCC, ppm	CHLORINATOR NO. 3 TCC, ppm	CHLORINATOR NO. 5 TCC, ppm	REMARKS
0.38	0.19	0.37	Baseline
0.37	0.32	0.26	0.05% I_2
0.27	0.17	0.17	0.05% I_2
0.21	0.24	0.25	0.05% I_2
0.28	0.68	0.28	Baseline
0.42	0.41	0.22	Baseline

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This procedure was repeated with 0.02% iodine and then with 0.05% iodine. The next batch contained 0.02% iodine and the demister was not soaked afterwards. It was followed with two normal batch baselines.

TABLE III

TEST III - TCC RESULTS

% I_2	NO. OF SOAKS[(i)]	TCC, ppm	REMARKS
0	0	0.66	Before Soaking Baseline
0	2	0.39	After Soaking Baseline
0.02	1	0.45	
0.02	1	0.29	
0.05	1	0.47	
0.02	0	0.36	
0	0	0.45	Baseline
0	0	0.46	Baseline

*(i) After chlorination

The fourth plant test consisted of removing the demister completely and making two batches with 0.05% iodine and one without. The demister was replaced and a baseline obtained. It is interesting to note that the amount of off gas condensate did not increase when the demister was removed.

TABLE IV

TEST IV - TCC RESULTS

% I_2	TCC, ppm	REMARKS
0.05	0.74	Demister Removed
0.05	0.21	Demister Removed
0	0.35	Demister Removed
0	0.47	Demister in place

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The results of all four plant tests dramatically emphasize the discrepancies between the results of laboratory studies and plant results. Biphenyl samples were taken during each test and chlorinated in the lab apparatus. The results are listed in Table V.

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TABLE VI LABORATORY CHLORINATION RESULTS

TEST	TCC, ppm WITHOUT I_2	TCC, ppm WITH I_2	REMARKS
1	0.07	0.07	Plant Catalyst mix
1	0.12	0.09	Plant Catalyst mix
2	0.23	0.11	Plant Catalyst mix
3	0.14	0.12	Plant Catalyst mix
4	0.11	0.09	Plant Catalyst mix

When the demister was removed, a sample of the black tar-like material that forms $\frac{1}{4}$ to $\frac{1}{2}$ inch coating over the interior of the chlorinator was taken. There was no effect on the improvement due to iodine from 1% of this material added before a laboratory chlorination. It is assumed that differences in the materials of construction between the lab and plant chlorination (glass vs. steel) would improve the plant results if anything.

The effect of the iodine co-catalyst on by-product muriatic acid was considered during the tests. Off gas samples were taken periodically during the first two tests. As expected, it was found that the iodine concentration in the off gas quickly rose to a maximum and then tapered off to zero. In the most severe case, the iodine concentration in the off gas during a chlorination at 195°C with 0.05% iodine was 34 ppm after 10 minutes, 16 ppm after $2\frac{1}{2}$ hours and 0-7 ppm after $3\frac{1}{2}$ hours. This extrapolates to less than 2 ppm I_2 in the muriatic acid if all five batch chlorinations are using iodine and none is lost between Department 246 and the acid product. Free iodine can be visually seen in concentrated muriatic acid at the 1 ppm level. Additional tests will be required to determine if the acid produced is acceptable in the event that iodine co-catalyst use comes to fruition.

Currently it is believed that differences in chlorine quality between the plant and lab are masking the effect of iodine in the plant tests. This is possibly related to the presence of highly chlorinated organic compounds in the Hooker chlorine. The presence or absence of these compounds in Aroclor could explain the variations in the batch TCC results. Future work will include batch chlorinations in lab glassware using various sources of plant chlorine.

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