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PROGRESS REPORT
TECHNICAL SERVICES DEPARTMENT
ANNISTON, ALABAMA PLANT

JOB NO. 009-1104 **REPORT NO.** 2 **DATE** April 27, 1971

TITLE: LOW COLOR AROCLOR 5460

OBJECTIVE: To report results of plant experience producing low color Aroclor 5460, and a summary of work to date and planned action.

PERSONNEL: G. L. Moore, K. G. Hale (V. R. Haupt)

REPORTED BY: K. G. Hale

SUMMARY: Aroclor 5460 with a color less than 1.25 NPA has been produced in three of four plant batches. The fourth plant batch was not finished due to safety problems encountered while processing the batch. A decomposition occurred in the blow tank following the transfer of the batch to the still. Additional lab studies indicated that NaOH concentrations, temperature and hold times are critical. Caustic concentration must be kept below 5% (overall or local) at temperatures above 250°C. to prevent decomposition of A-5060. The presence of air increases the possibility and rate of a decomposition, but the absence of air does not exclude a decomposition caused by high local NaOH concentrations.

Existing plant equipment is not adequate "as is" for completing the NaOH reaction. Severe safety problems exist in using existing plant equipment with only previously proposed changes (NaOH addition equipment).

FUTURE: 1. Issue revised project costs and product costs based on the following:

Target date - 5/28/71

a. Agitate the #4 still.
b. Re-evaluate venting and rupture disc capacity of #4 still.
c. Inert the #4 still with N_2 during reaction.
d. Use soda-lime (ground caustic and lime -~ 50/50). Find source and costs.
e. Soda-lime handling and addition equipment.
f. New bagger head for low color to be used with A-1268 flaking system.

FUTURE: continued

g. Startup A-1268 flaking system installed on CEA 2080.
h. Set up existing storage tank for low color A-5460.
i. Install necessary meter, piping, and instruments for using low color material to make blends.

2. Proceed with design and ordering of equipment following Production approval of revised costs - target - 6/71.

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Background

Foreign competitors produce chlorinated terphenyl products which compete with our Aroclor 5460. These competitive products have lower color than Aroclor 5460 (1 to 1.5 NPA compared to 1.75 to 2.25 NPA).

Marketing requested that an investigation be completed to determine if a low color Aroclor 5460 could be produced. Anniston PR&D investigated several methods of reducing color (Anniston PR&D Reports 446-507-1, 2), and developed a procedure for producing Low Color Aroclor 5460 by distilling over caustic (2.5% by wt.).

During September 1970, an initial plant batch was produced (see TSD Progress Report 009-1104-1 for details). Aroclor 5460 with a color of 1.0 NPA was produced but a problem was encountered when the still burped material out an open vent when the heat-up reached approximately 305°C. No evidence of decomposition was found and the gas evolution was explained by a release of bound water from the caustic when it melted.

Details

Following the first plant batch, a project was issued to add necessary facilities to #4 still for caustic addition and set up an existing storage tank for low color Aroclor 5460. After approval of the project, additional plant tests were scheduled to tie down the scope for additional equipment and further prove the process.

The second and third plant batches of low color production were completed in January 1971. Product colors were excellent as in the initial plant batch - 9/70. However, problems were again encountered despite the following changes in operating methods:

1. After blowing and neutralizing, the batch was transferred to the still and heated to 310°C.

2. At 310°C., caustic addition was started at a rate of approximately 10#/min.

The slow addition of caustic at high temperatures (≥310°C.) was used to avoid high dewatering rates and eliminate entrainment or burping of the batch. The caustic addition during the second batch went smooth with only occasional puffs of vapor (steam?). Distillation went as normal and bottoms were normal. The next batch used the same procedures with only one exception, some off-color Aroclor 5460 was charged to the still during heat-up to make up for a short charge from the chlorinator. After a caustic addition of 500# (1.9%), a release of gas and smoke occurred through the vent where caustic was being added. The caustic addition was cut short and a normal distillation was completed. The product color was again excellent (≤1.0 NPA). Investigations following the release of gas/smoke during caustic addition yielded the first evidence of a decomposition.

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Details - continued

Lab attempts to duplicate the plant experience failed. A wide variation of conditions were tried (see PR&D Mid-Month Report 2/16/71 for details). At the 2.5% NaOH level, a small exotherm was noticed when the NaOH was added at approximately 320°C., but no rapid decomposition was seen. Lab tests with caustic addition at 250°C. indicated a smooth caustic reaction with agitation, vacuum and slow heat-up. All lab data indicated no significant exotherm or decomposition when using the above conditions, therefore a fourth plant test batch was proposed with the following operating conditions:

1. Added lime in blow tank
2. Check for acid in blow tank
3. Add additional lime and NaOH mixed in blow tank @ 250°C.
4. Blow and circulate for mixing (1 hour)
5. Transfer to still
6. Pull vacuum on still
7. Raise temperature slowly
8. Distill as normal when temperature reaches 320°C.

Note: Low NaOH charges (250# or 0.95%) were to be used in initial plant batches, with larger NaOH charges only if required to meet low color spec.

The plant test batch (using above operating conditions) was going in the following manner:

1. Normal A-5060 batch was held in #2 blow tank for approximately 40 hours until circulating pump could be repaired.

2. Acid number was checked to make sure the batch had been neutralized (with lime).

3. Additional NaOH and lime mix was added at 240°C. The addition took approximately 30 minutes.

4. The batch was then circulated and air blown for 1 hour.

5. Transfer to the still went as normal.

6. Vacuum was applied to the still and pulled down to 5-10 mm Hg within 40 minutes (normal).

7. Still heat-up was started.

8. At precisely one hour following the transfer to the still, a loud boom was heard in the blow tank/still area.

9. Heat was cut off the still.

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Details - continued

10. An investigation indicated a decomposition and/or explosion had occurred in the blow tank.

11. Decision was made at this time to stop the low color test.

12. The still batch was drummed out for disposal.

13. One hour after drumout, the last drum of material began smoking and decomposing rapidly. The material began to burn and the flames were snuffed out. Inspection of the material later indicated that a near complete decomposition had taken place and residue was similar to ash.

Additional lab tests to duplicate plant results were completed with the following condlusions (see PR&D Mid-Month Report 4/21/71 for details):

a. NaOH concentrations in excess of 5% will produce decompositions at temperatures above 300°C.

b. Plant problems were caused by local NaOH concentrations higher than 5% which started a self-feeding decomposition.

c. Introduced air increases the possibility and rate of decomposition.

d. Soda lime (ground NaOH and lime mixed) is better than NaOH pellets for controlling the reaction and for mixing properties.

e. NaOH concentrations of approximately 1.0% should produce low color A-5460 in the plant system.

Conclusions:

1. NaOH-Lime process will produce low color A-5460, but the reaction conditions (NaOH concentrations, temperature, hold times, etc.) are critical.

2. Existing plant equipment is not adequate "as is" for completing the reaction.

3. Severe safety problems exist if localized NaOH concentrations are allowed to exceed 5%.

4. Air increases the possibility and rate of a decomposition.

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Recommendations

1. Evaluate feasibility of agitating the #4 still.

   This vessel has an internal submerged pump which will no doubt interfere with agitation. Other potential problems are vacuum service, agitator mounting, and temperatures.

2. Evaluate rupture disc capacity on #4 still.

   The vent lines should also be checked for pluggage.

3. Inert the system with N_2 during the NaOH reaction.

4. Locate a source of soda-lime.

   Any future plant batches should use soda-lime to minimize the possibility of caustic settling out and producing high localized concentrations.

5. Add soda-lime to the still at 320°C. and at a slow rate to control the reaction.

   Studies indicate that the caustic reaction goes to completion at temperatures above 320°C. Slow addition of NaOH-lime mix with agitation and 320°C. should allow control of the reaction and minimize the possibility of getting a buildup or high local NaOH concentration. Future plant batches should be started with NaOH concentration of 0.5-1.0% and work up to requirements to meet specs.

6. Re-evaluate project scope and product costs based on above recommendations.

   Issue revised costs by 5/28/71.

References

1. PR&D Report #1 - 446-507 (1-22-69) AROCLOR 5460 COLOR IMPROVEMENT (G. L. Arnett).

2. PR&D Report #2 - 446-507 (9-30-69) LOW COLOR AROCLOR 5460 - (G. L. Arnett).

3. Tentative Amendment "C" to Aroclor Standard Manufacturing Process (7/14/70 by V. R. Haupt).

4. TSD Progress Report #1 - 009-1104 (9/29/70) LOW COLOR AROCLOR 5460 (G. L. Moore).

5. Revision and Extensions to Tentative Amendment "C" to Aroclor Standard Manufacturing Process. USE NaOH TO PRODUCE LOW COLOR A-5460. (2/16/71, by K. G. Hale).

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References - continued

6. PR&D Mid-Month Reports dated 2/16/71, 4/21/71.

7. Organic Division Technical Department - Special Study 71-9, THERMAL STABILITY OF AROCLOR 5060/NaOH/LIME.

8. Memo B. O. Severson to V. R. Haupt (4/5/71) "QUESTIONS ON LIME-NaOH-A-5460 - JAPANESE EXPERIENCE"

9. AFE 3643 (12/18/70) INSTALL EQUIPMENT TO PRODUCE AND STORE LOW COLOR A-5460.

Ken

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