Monsanto

FROM: K. E. Boucher, W. G. Krummrich Plant

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SUBJECT: DEMONSTRATE THE CONVERSION OF DISTILLED MONOCHLOROBIPHENYL RESIDUE INTO AROCLOR 1242′

REFERENCE:

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In September 5660 gallons of Monochlorobiphenyl for Dow's evaluation was made. Also collected was 4000 gallons of distilled Monochlorobiphenyl residue. This material stored in #1 and #2 blend tanks is nearly equivalent to Aroclor 1121.

OBJECTIVE

Large quantities of residue are produced in the new Monochlorobiphenyl process (Report No. P-1766, Process for Engineering Design for Monochlorobiphenyl, 7/29/74, by D.R. Cova). About 70 pounds of residue is produced for every hundred pounds of Monochlorobiphenyl. To complete the field demonstration of the new Monochlorobiphenyl process, the Mono residue must be successfully converted into Aroclor 1242′. Aroclor 1242′ differs from the normal Aroclor 1242 in that it has a low dielectric constant and a higher refractive index.

A successful demonstration will assure us Aroclor 1242′ can be made in the Aroclor department and at the same time valuable design data may be obtained for engineering.

RESEARCH PROCESS

Monochlorobiphenyl is produced by fractionating Aroclor 1111. The large quantity of still residue is straight takeover distilled. This distillate is chlorinated to Aroclor 1142′, isomerized over $ALCl_3$ (for 5 hours), lime treated (for 2 additional hours), then distilled again, Porocel treated, and stored as Aroclor 1242′.

POTENTIAL PROBLEMS

1. The Mono residue made is expected to be somewhat contaminated with Aroclor 1242. This will cause the 4,4'-DCBP in the Aroclor 1142' produced for the Mono residue to be higher than normal. Further contamination of the Mono residue prior and during the batch chlorination step must be avoided. Otherwise the isomerization step may not be able to lower the 4,4'-DCBP level to less than 2.6%.

2. The aluminum chloride used to catalyze the isomerization of Aroclor 1142' is more volatile than $FeCl_3$ and therefore may carry over from #4 still with the distillate if the lime treatment of 2 hours is not adequate. For this reason aluminum contamination in the resulting Aroclor 1242' and the next normal run of Aroclor 1242 must be checked. In addition, clean out of the filter up-stream of #5 absorber may be desirable following this demonstration.

   #2 still will be used instead of #1 still. In the design scope for Monochlorobiphenyl #1 still is to be used for isomerization. However, the circulating pump in #1 still needs to be replaced, and therefore, this still is not available. #2 still's circulating is greater than in #1 still. Hopefully, the difference in agitation will not effect the results of this demonstration.

DEPARTMENT EQUIPMENT FOR DEMONSTRATION

Unit	Use
#1/#2 Blend Tank	to store Mono residues
#2/#3 Chlorinator	for batch chlorination
#2 Blow tank	a chlorinator, receiver and feed tank to #2 and #4 stills
#2 Still	First for isomerization, then the lime treatment
#4 Still & receiver	to distill Aroclor 1142'
#5 Absorber	for Porocel treatment
#16 Storage	Aroclor 1242' product storage.

OPERATING SEQUENCE

The plant equipment will be used in the following manner:

Residue in #1 Blend tank will be used for this test. #2 blend tank material will be held in reserve. If this first run is successful, residue in #2 blend tank will later be converted into Aroclor 1254.

About 2000 gallons of Crude 1142' will be made by chlorinating three 500 gallon batches using Mono residue. #2 chlorinator will be used from which the resulting chlorinated crude is to be dropped into #2 blow tank and pumped into #2 still.

In #2 still the batch is isomerized at 250°C for 5 hours or more to reduce the 4,4'-DCBP level to less than 2.6% using $ALCl_3$ as a catalyst. The crude will be lime treated for two hours in #2 still and pumped into #2 blow tank. From #2 blow tank the crude Aroclor 1142' will be continuously fed to #4 still. The distillate will be Porocel treated and isolated in #16 storage tank.

EQUIPMENT CLEAN-UP

Contamination of the distilled Mono residue must be held to a minimum. Otherwise the isomerization step cannot be reduced to a 4,4'-DCBP level of less than 2.6%.

A real possibility is contamination in #2 chlorinator from 1142/1154 remaining in the vessel. This will be eliminated by this procedure - using #2 chlorinator, make the last batch of Aroclor 1242 from Biphenyl. Chlorinate the batch to 1121; then transfer to #3 chiroinator before completing the batch chlorination. #2 chlorinator is now clean and ready for chlorinating the Mono residue.

The last two batches of Aroclor 1142 will be used to flush out #2 blow tank and #2 still. Careful attention is needed to be sure all the lime is removed from #2 still. Any lime will stop the isomerzation.

To store the Aroclor 1242', #16 storage tank must be empty and clean.

PREPARATION

A number of specific actions are planned in preparation of this demonstration. Status of each is now given:

1. Provide analytical methods

   Work requests were made to develop analytical methods for 4,4'-DCBP in Aroclor 1142' and aluminum in Aroclor 1242'. Hap Horner's group is expected to meet a 1/1/75 target.

2. Order $ALCl_3$ catalyst

   Fourteen (14) bags each containing 15 pounds of anhydrous $ALCl_3$ were ordered on 11/22/74 from Pearsoll Chemical Corp. Expected delivery is 12/5/74.

3. Test distilled Mono residue

   Research will simulate the process using residue made from #1 blend tank and the $ALCl_3$ catalyst ordered by the plant. EDC 1/1/75.

4. Manual Control of #2 still temperature

   This was tested by Production. Using two burners with 10 lb. gas pressure, the still temperature was held for more then one hour.

5. Transfer Lines

   Project #6147 was forwarded for approval on 11/21/74. Estimate is for $8400 experimental expense. Installation includes (1) a transfer line from #1 blend tank to the biphenyl head tank, and (2) a line from #2 still to #2 blow tank.

AFTER TEST IS COMPLETED

1. If necessary, the test can be repeated using #2 blend tank material. Any Aroclor 1242' made can be disposed of by converting it into Aroclor 1254.

2. Residue from #4 still containing $ALCl_3$ will be dumped into drums. Samples will be taken for DSC analysis and compstability with residue now burned at the incinerator.

AFTER TEST IS COMPLETED (Cont'd)

3. Samples of the Aroclor 1242' will be analyzed for electrical properties plus aluminum content, and then pumped to #7 storage tank. #7 storage material is converted into Aroclor 1254.

4. The next normal 1242 batch through the Porocel column will be checked for aluminum contamination. If necessary, the filter before the #5 absorber column will be cleaned.

5. The newly installed lines will remain. If Monochlorobiphenyl is made on a permanent basis, the new line from #1 blend tank to the biphenyl head tank will be used.

6. If the test is succesafully completed, the remaining Mono residue in #1 and #2 blend tanks will be converted into 1254 with samples of the 1154 after chlorination taken for analysis.

TIMING

Tie-ins for installing the new process lines will probably take 8 hours downtime. Then two batches of 1242 will be made from Biphenyl in the batch chlorinators to clean #2 still. Thus production capacity for 1254 will be reduced. Estimated time to process these two batches in 12 hours. The remaining time estimates are now given starting with the 12800# of Aroclor 1142 in #2 blow tank.

TIME SCHEDULE

Wash #2 still by pumping crude 1142 from #2 blow tank and circulating in #2 still.	10 hours
Inspect #2 still	8 hours
Feed crude 1142 to #4 still and boil down	8 hours
Complete chlorinating 3 batches of Mono residue and transfer into #2 blow tank.	8 hours

TIME SCHEDULE (Cont'd)

Transfer batch to #2 still. Add the $ALCl_3$, heat.	8-12 hours
Heat to 250°C at near atmospheric pressure and hold for at least 5 hours	
Lime treat for at least 2 hours in #2 still	4 hours
Pump through #2 blow tank to #4 still to provide a heel and continuously distill and Porocel heat pump to #16 storage. Batch chlorination of 1016 still residue in #2/#3 chlorinator can now begin.	
Estimated total time Aroclor 1254 production is stopped	72 hours
Drain residue from #2 still	4 hours
TOTAL TIME	76 hours

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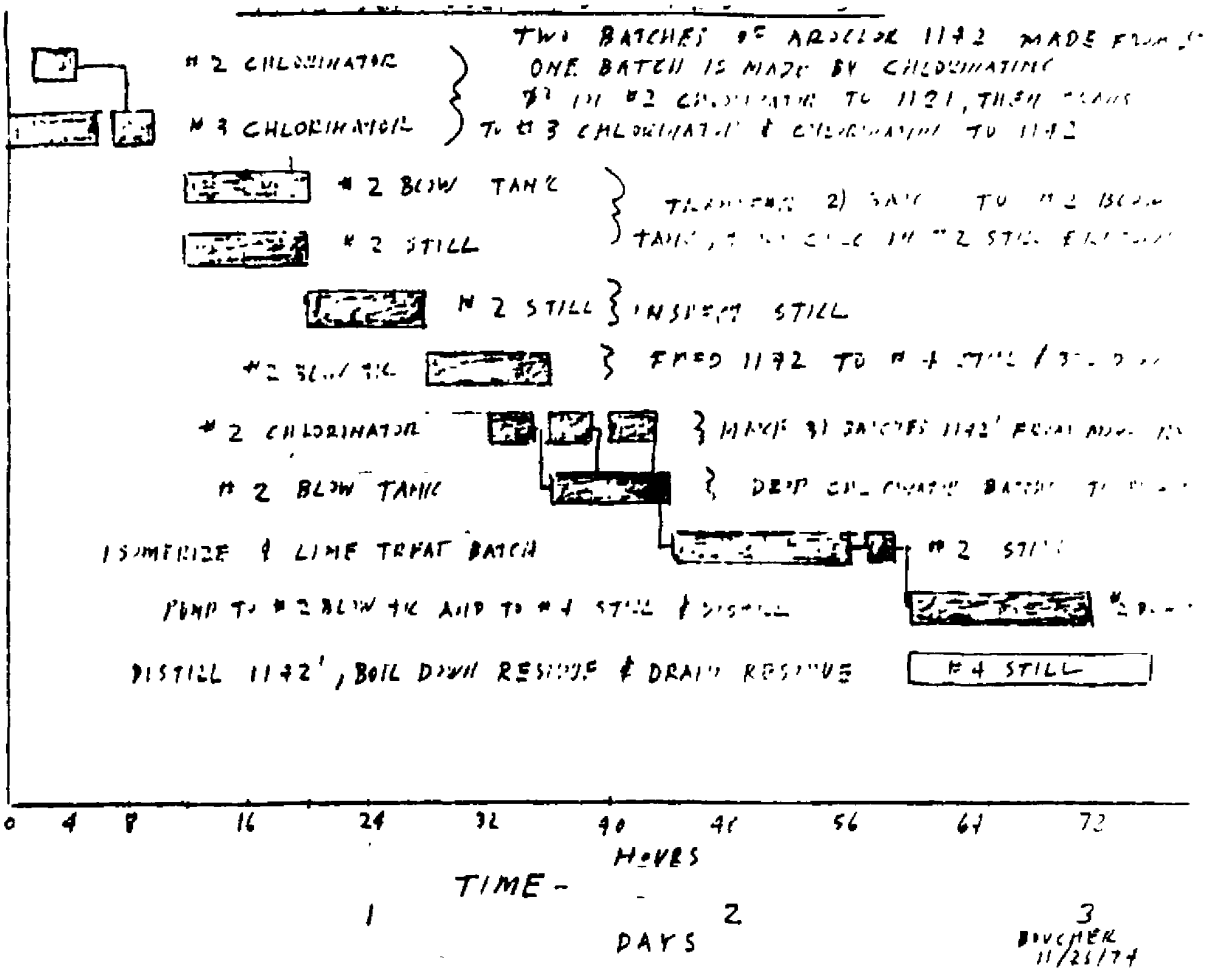

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